



MANUFACTURING OF WEAR RESISTANT CLADS FOR SLURRY/TAILINGS PUMP IMPELLERS VIA LASER CLADDING (LC).

By

KINSMAN MALIKONGWA

Reg. No: 16100282

B.Sc. (Mining Engineering) (Missouri S&T)

**Department of Mechanical, Energy and Industrial Engineering,
Faculty of Engineering,
Botswana University of Science and Technology.
Email: Kinsman.malikongwa@studentmail.biust.ac.bw.
Mobile phone: (+267) 72567691**

**A Dissertation/Thesis Submitted to the Faculty of Engineering in Partial
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of BIUST**

Supervisor: Prof. E. O. Olakanmi
Department of Mechanical, Energy and Industrial Engineering, Faculty of
Engineering, BIUST
E-mail: olakanmie@biust.ac.bw, Tel: (+267) 74276012

Signature: _____ Date: **18-05-2019**

Co-Supervisor: Dr M. Tlotleng
Laser Enabled Manufacturing (LEM) Research Group,
National Laser Center/Council for Scientific and Industrial Research (NLC/CSIR),
Pretoria, South Africa.
E-mail: Mtlotleng@csir.co.za, Tel: +27 12 841 2395

Signature: _____ Date: **18-05-2019**

May 2019

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CERTIFICATION

The undersigned certifies that he/she has read and hereby recommends for acceptance by the Faculty of Engineering & Technology a dissertation/thesis titled: ***Manufacturing of wear resistant clads for slurry/tailings pump impellers via laser cladding (LC)***, in fulfilment of the requirements for the degree of Master of Engineering in (Manufacturing Engineering) of the Botswana International University of Science & Technology (BIUST).



.....
Prof. E. O. Olakanmi

(Supervisor)

Date: 18-05-2019



.....
Dr. M. Tlotleng

(Co-Supervisor)

Date: 18-05-2019

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Dedication

I dedicate this work to my family especially my Grandmother Mavis Mothonyana.

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List of Abbreviations

A - Availability
ANOVA – Analysis of variance
BH – Breakdown hours
CTE - Coefficient of thermal expansion
DoE – Design of experiments
GMAW – Gas metal arc welding
HAZ – Heat Affected Zone
HV – Hardness value
HVOF - High velocity oxy-fuel
IH – Idle hours
LACS – Laser assisted cold spray
LC – Laser Cladding
LED – Laser energy density
MH – Maintenance hours
OM – Optical microscope
S/N – Signal to noise
SAW – Submerged arc welding
SEM – Scanning electron microscope
SMAW – Shielding metal arc welding
SSH – Schedule shift hours
ST6 – Stellite 6
T-800 – Tribaloy T800
U – Utilisation

ABSTRACT

Wear is a huge problem in the mining industry as it depreciates the assets invested. The industry is looking at efficient refurbishment technologies to repair its equipment so as to enhance its service life. This thesis focuses on the development of wear resistant clads via laser cladding (LC) process to mitigate the problem of degradations in slurry tailings' pump impeller made of 304L stainless steel. A critical literature review indicates that Tribaloy T-800 and Stellite 6 powders are candidate materials for modifying 304L stainless steel on account of compatibility in the thermo-physical properties of the clads and the substrate. Hence, consolidation behaviour of fiber-laser deposited Tribaloy T-800 and Stellite 6 clads was analysed for chemical homogeneity, microstructural evolution in addition to geometrical characteristics via process maps. Characterisation techniques such as optical microscopy (OM), scanning electron microscopy/energy-dispersive X-ray spectroscopy (SEM/EDS) and X-ray diffraction (XRD) were employed in the analysis of the regions on the process maps with a view to determining the more suitable material in modifying 304L stainless steel substrate. Five regions of consolidation were identified for each coating with varying parameters for different process regions for each coating accounted for by variation in chemical composition. Tribaloy T-800 clads were taken further for quality optimisation of its wear resistance and microhardness on the premise of their superior consolidation behaviour and mechanical properties relative to that of Stellite 6 clads. Taguchi design of experiments (DoE) was implemented for predicting the coefficient of friction (COF), wear resistance and microhardness of laser clad Tribaloy T-800 clads as feed rate, scan speed and spot diameter were varied. The outcome of this study reveals an optimal combination of parameters for improving the wear resistance of the clads and a good agreement between the predicted and experimental values within acceptable level of significance.

Keywords: Laser Cladding, Stellite 6, Tribaloy T-800, Wear rate; Taguchi design of experiments (DoE).

Chapter One: Introduction

1.0 Chapter Summary: This Chapter introduces laser cladding (LC) technology by elucidating what laser is, types of lasers and how laser is used in cladding. It also discusses the problem of tribo-corrosion in the industries and considers the primary and secondary effects of wear in the industries. Later on, the aim and objectives of this research are presented together with the scope of the study, specific themes to be investigated, expected outcomes, outline of thesis and conclusions.

1.1 Background to the Study:

The name “laser” is an abbreviation of ‘light amplification of stimulated emission of radiation’. Laser is made by optical amplification of light from a stimulated emission of electromagnetic radiation and therefore laser falls under the electromagnetic spectrum (Gu, 2015; Weber, 1999). In general, lasers are made from an optical set up comprising of (1) gain material or medium (2) energy pump and (3) optical cavity. There is quite a number of gain materials that can be used and they classify lasers into three types namely gas (Carbon dioxide-CO₂, Helium-Neon-He-Ne), liquid dye (Xanthene Dye, Oxazine Dye) and solid state (Nd:YAG -neodymium-doped yttrium aluminum garnet) lasers (Higgins 1995; Pogorelsky 2004; Shankarling *et.al.*, 2010); Weber, 1999). As depicted in Figure 1.1, the electrons of the gain material are excited with the energy from the energy pump which can either be electricity or another laser. Upon reaching a certain level of excitement, these electrons will release photons. The optical cavity is a pair of mirrors placed on each side of the medium, one being fully reflective (high reflector) and the other is partially reflective (output coupler). The light from the production of photons bounces back and forth between the mirrors and upon reaching a certain level of intensity it escapes through the output coupler and the process continues (Karman *et.al.*, 1999; Siegman, 2000). The produced light is imparted with better coherence, monochromaticity and uniform polarization through the optical resonator. These characters are far more important for laser applications as they affect the laser beam spot size, wavelength and intensity. These properties are also used to locate them within the electromagnetic spectrum (Figure 1.2) and also to understand their applicability. For instance, it is well known that short wavelength lasers have high absorption in most additive manufacturing processes (Olayanmi *et*

al., 2015). Lasers are being used in different processes like cladding, ablation and hardening that require precise melting, vapourisation and heating respectively.

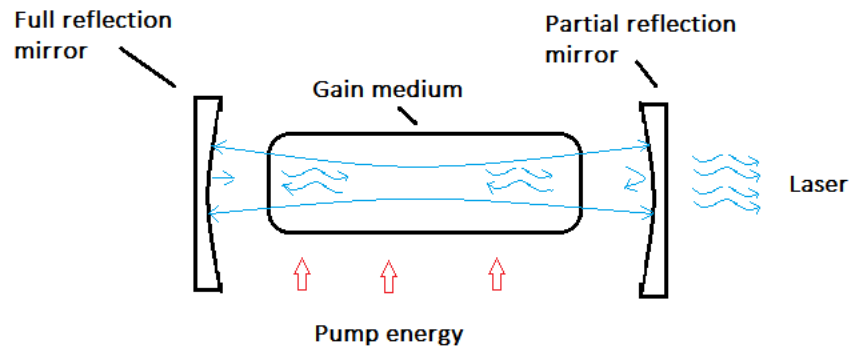


Figure 1.1 Schematic of laser formation process (Source: Weber, 1999).

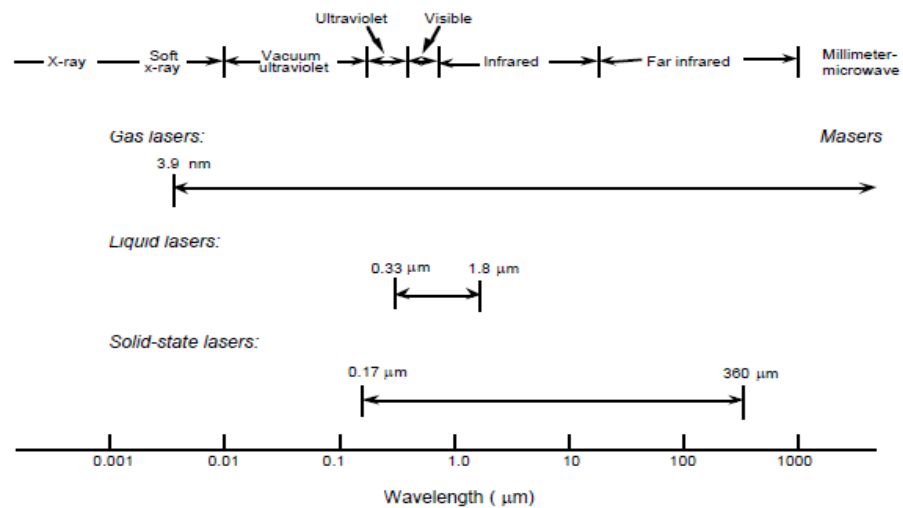


Figure 1.2: Different laser wavelengths. (Source: Weber, 1999).

This study explores the use of lasers in cladding technology for applications in the Botswana mining industry. LC is a repairing and refurbishment technology which is engaged in in aerospace, military, mining, medical, agricultural industries e.t.c. With LC technology, a base metal or substrate is cladded with a metal powder that has superior functional properties. During the melting of cladded powders onto the substrate, minimal dilution between the two metals is allowed (Gaebler *et al.*, 2013). The deposited powder will eventually bond with the surface of the base metal. It is sometimes accompanied with a heat affected zone (HAZ) to an advantage of avoiding

undesirable mixing of the two materials and keeping the individual properties of both metals. Normally, the properties induced can either be improved wear resistance or corrosion resistance. Therefore, it is important to understand LC and the applicability to the mining industry.



Figure 1.3: A well bonded Tribaloy T800 clad on a 304L stainless steel substrate.

Equipment in the mining industry ranges in size and functions from excavators, dump trucks, front-end loaders, earth compacters, drainage pumps, pipes to drill rigs. Even though these equipment are made from high quality materials, they do not perform to their expected service life due to the fact that they operate under uncondusive and wear promoting environmental conditions such as acidic environments as shown on Figure 1.4. Productivity or efficiency of equipment under these circumstances becomes a challenge. Productivity is affected by these environmental conditions specifically looking at wear. On average, productivity occurs through utilization, availability/reliability of the machine on ground, cycle time and bucket capacity for bucket utilising machines or spacing & burden for drilling equipment (Kansake *et al.*, 2015; Mishra *et al.*, 2013). Utilisation and availability of machines are main factors that affect productivity which in turn are also affected by the wear of machine parts. If an equipment is worn out, it will breakdown and will not be available to do work and this means it is also under utilised. Equation (1.1) describes availability (A) and shows that maintenance hours (MH) and breakdown hours (BH) should be minimised to get adequate time for the machine on ground over the scheduled shift

hours (SSH). The same concept goes for utilization (described by equation 1.2) except that it adds on another aspect of idle hours (IH)) of the machine. Productivity of the draglines is shown by equation 1.3 which mentions multiplying the availability, utilization, bucket capacity and total shift hours in a year over cycle time (Rai, 2004). So in this case, when machine components are worn out, the breakdown and maintenance hours increase hence reducing the machine availability on the ground as well as machine utilization.

$$\text{Availability, } A = \frac{SSH - (MH + BH)}{SSH} \quad (\text{Eq. 1.1})$$

$$\text{Utilisation, } U = \frac{SSH - (MH + BH + IH)}{SSH}. \quad (\text{Eq. 1.2})$$

$$\begin{aligned} \text{Productivity} = & \left(\frac{\text{Bucket capacity}}{\text{Cycle time}} \right) * A * U * \text{Swell factor} * \text{Fill factor} * \\ & \text{Operating hours per shift} * \text{No. of shifts per day} * \\ & \text{No. of production days in a year} * 3600 \end{aligned} \quad (\text{Eq. 1.3})$$



Figure 1.4: Oxidation wear on pipes from Grootvlei mine plants (Adopted from Mukhuthu, 2014)

The mining industry just like most industries aims to minimise project costs and this includes minimising maintenance and repair costs. Maintenance costs contribute directly to project costs and they can add up to a significant amount of the operating expenditure. Machine breakdowns mean the need for maintenance and the need to use money to cover the costs incurred. Therefore, there is serious need to address problems of wear. It is now understood that mechanical problems in machines

especially those contributing to breakdown and long maintenance periods affect productivity and project costs. Therefore, tribologists were able to understand that this self-degradation behaviour happening to machine components occurs either through oxidation, wear or corrosion (Kato & Adachi, 2001; Myers, 2002).

1.2 Problem Statement:

Botswana mining companies, especially Debswana mining company, have been hit hard by the problem of tribo-corrosion of which it is very costly on their projects. Machine components are subjected to high wear conditions. As of the year 2017, the water inside the Orapa mining sump was very acidic. The effect is of high costs in a sense that the equipment is exposed to those acidic conditions which oxidises or corrodes almost every equipment from water pumps & pipes, shovel buckets, excavator buckets, drill rigs, crusher liners, mill liners and also not good to the environment. The main challenge arises when getting the production to run throughout annually. The cost of replacing and repairing the machine components becomes too high reducing the net profits of the project. Also, equipment is sent to distant places especially across borders to get the assistance needed as in Botswana there are no equipment re-manufacturing companies. The downtime of this situation bears much cost to the required productivity or profitability. Studies indicated that tribo-corrosion can contribute to up to 4% loss on a country's gross national product (Bayer, 2004). Most of the technologies in place for machine component remanufacturing are not meeting the industry demands or they do not produce satisfactory results.



Figure 1.5. Shrouded centrifugal pump impeller (Addie *et al.*, 2007).

In this research, the tailings slurry/sludge pump impeller which is one of the equipment components subjected to high wear stresses is considered. Tailings are a result of the mineral separation process which enables the attainment of valuable minerals from the mined rock ores (Wills & Nappier-mun., 2006). Tailings become a waste in a form of a paste containing waste rock particles of a certain concentration, chemicals and water. Thickening is the next important process before tailings disposal as the mine tries to recover waste water from process plants as well as retaining an optimum amount of water in the tailings slurry for good material and viscosity properties to promote better pumping conditions. The process is optimised, but there is still degradation of pump impellers. Figure 1.5 shows a sectioned image of a centrifugal pump which is the common pump used in slurry pumping. Impellers can be made from either 300 or 400 series stainless steels (Bloch *et. al.*, 2014).

1.3 General and Specific Objectives of this Study:

This section maps the road followed by this study, showing specific objectives that were followed in order to merge everything into the required information.

1.3.1 General Objective: The aim is to manufacture clads with improved wear and corrosive resistant properties from Stellite 6 and Tribaloy T800 powders to refurbish sludge/slurry pump impellers. The 304L austenitic stainless steel (SS) is used as the base metal in this research which could also be used for manufacturing tailings slurry pump impellers (Bloch *et. al.*, 2014).

1.3.2 Specific Objectives: To achieve the general objective, specific objectives set for this research are as follows: -

- i. Develop LC process maps for Stellite 6 and Tribaloy T800 clads with a view to understanding the effects of variation in process parameters on their consolidation behaviour.
- ii. Comparing the geometrical characteristics, microstructural properties and consolidation behaviour of the two powders with a view to identifying a suitable clad material for the substrate material 304 L SS of which the slurry pump impeller is made of.
- iii. Determine the effects of process parameters on the microhardness and wear properties of the suitable clad material for the substrate material.

- iv. Optimise the quality characteristics (microhardness and wear properties) of the suitable clad material as process parameters are varied via Taguchi design of experiment (DoE).

1.4 Delimitation of the Scope of this Study:

This research designs a wear and corrosion resistant coating which addresses the issue of wear on a mining equipment with focus on slurry/sludge pump impeller. The clads were fabricated using LC technology. Metal powders of Stellite 6 and Tribaloy T800 were selected for this study based on their for wear and corrosion resistance performance as well as the compatibility of their thermo-physical properties with the substrate. LC is used for refurbishment of worn surface and coating of newly acquired equipment so as to improve their performance and life time. LC process parameters were varied to understand their influence on the consolidation behaviour, geometrical characteristics and wear resistant properties of the clads. Different behaviour of the clads were analysed via a range of metallographic tests. Again, tribology studies on mining equipment only focussed on components that are highly worn out and made from metals or their alloys. This selection of components was to investigate where LC technology could be applicable in the mining industry. This research does not carry out economic feasibility analysis of the technology as well as simulation/modelling of the behaviour of the clads.

1.5 Specific Themes of this Thesis Investigated

Varying process parameters and two types of powders were selected to achieve the objectives of this research. The process parameters included feed rate, scanning speed and spot diameter. Process maps of the two metal powders (Stellite 6 and Tribaloy T800) were then constructed based on the consolidation behaviour at varying process parameters. Metallographic analysis was carried out on the clads by using optical microscope (OM), scanning electron microscope (SEM) with a view to understanding the consolidation behaviour of the laser clad clads. Wear tester was engaged to elucidate the resistance to abrasive wear of the clad samples respectively. Then the outcomes of the experiments were optimised using Taguchi Design of experiment (DoE).

1.6 Significance of the study

This research conducts a review on these technologies as well as providing clads with improved mechanical properties and functionality. Again, this research is set to provide knowledge to the Botswana industries regarding the use of laser-based manufacturing (LC) to repair slurry pump impellers and other end of life parts in mining industries. This implies that the Botswana workers involved in mastering the implementation of this technology will benefit in terms of both employment and skills. As for the mining industry, the costs of sending worn out components to other countries for maintenance will be reduced as it is expected that this research will convince them to invest locally on the technology. With all that, industries benefiting from the technology are expected to invest more into it and boost the Botswana Gross Domestic Product (GDP).

1.7 Outline of the Thesis

This thesis consists of five chapters. In Chapter One, the study begins with introducing the research background in which laser is described, its uses in the industry of additive manufacturing and its importance to the laser cladding technology. The chapter also looks at both primary and secondary effects of wear in the mining industry and the different modes of wear. Later, the problem statement of the project was described followed by the aim and specific objectives, delimitation of the scope of this study, specific themes of the thesis investigated as well as the significance of this study. Chapter Two comprises of literature review on forms of degradation from the first stages to final stages of the mining process. Surface modification techniques used in the mining industry are also listed and compared against the proposed laser cladding technique. The materials and experimental methodology adopted for this study are described in the Chapter Three of this thesis. It describes the laser cladding system, the materials selection, experiments conducted, sample preparations and sample analysis methods. It also includes all standards that were employed in carrying out various procedures. Chapter Four involves results analysis and discussion from the first experiment on “effects of laser cladding operational parameters on processing maps of Co-based alloys”. Chapter Five explores the second set of experiment on “coefficient of friction, microhardness and wear behaviour of laser clad Triballoy T800 clads” as well as optimisation of

the process parameters. Chapter Six concludes the findings from this research thesis involving the behaviour of the fabricated clads. Also, future work plans are also included in this chapter.

1.8 Conclusions

In this research study, Chapter One has provided necessary information on the background of this research as well as the problem statement, aim, specific objectives, delimitation scope of work, specific themes of the research as well as the thesis outline. The background in this chapter explains laser, its applicability in additive manufacturing as well as its relevance to laser cladding technology. Still in the background, wear is discussed as the main issue, how it affects the mining industry primarily and secondarily as well as the different modes of found on wear. The problem statement of the project is identified as Botswana mining companies finding it hard to re-manufacture machine components locally and as proposed, laser cladding technology that could provide functional clads on worn out components. It is now clearly stated that the aim was to fabricate wear resistant clads to be used in mining more especially focusing on sludge/slurry pump impellers. The research is narrowed to using Stellite 6 and Tribaloy T800 as cladding materials and 304L SS as the substrate material.

Chapter Two: Literature Review

2.0 Chapter Summary: This chapter presents reviews on typical forms of degradation in centrifugal pump impeller with a view to understanding the nature of wear it encounters in service. To identify an appropriate re-manufacturing technology to restore damaged centrifugal pump impeller to as new, various surface modification technologies were evaluated and co-related to the nature of wear encountered in the equipment. Thereafter, various studies dwelling on the selected re-manufacturing technology were engaged to elucidate how process and materials parameters may influence the remanufactured properties of impeller pump. Findings from this critical literature review were engaged in formulating the objectives of this study listed in section 1.3.

2.1 Typical forms of degradation encountered in centrifugal pump impeller:

There are many operational activities which take place in a mining process towards obtaining the final products such as diamond, iron and copper etc. These operational activities, as depicted in Figures 2.1 and 2.2 could be categorised into mining operations (e.g. digging, loading and haulage) as well as mineral processing activities (e.g. crushing and grinding, conveying/handling/storage, screening and minerals separation and classification). In this section, centrifugal pump impeller will be discussed in regard to the materials they are made of, their operations and the working environment. This equipment is selected for analysis in this section because it is one of the major mining equipment which is hit hard by the degradation phenomenon. Various forms of degradation encountered in this equipment based on the modes of wear are also described.

After beneficiation (the process of separating required minerals from the gangue), the waste which is in a form of mud (slurry) is sent to thickeners where the concentration of solutes is balanced with water recovery systems depending on environmental laws or mining requirements. Slurries can be conveyed as conventional, thickened or paste slurry depending on its density. Other researchers defined slurries based on the concentration of solids, (1) conventional (50-59%), thickened (60-69%) and paste (>70%) (Moolman & Vietti, 2012). A pump is then used to push the slurry to the

tailings dams through a pipeline that is either made from steel, high density polyethylene (HDPE) or made from steel with a HDPE liner. Centrifugal pumps are commonly used but in cases where high density slurries (>70%) are conveyed, positive displacement pumps are preferred. The centrifugal pump is made from numerous components which are:

- Impeller
- Motor
- Pump casing
- Inlet flange
- Outlet flange
- Shaft drive

These are the main components in the centrifugal pump and they function differently. The slurry gets into the pump through the inlet flange and enters the pump casing which encloses the impeller which is a plate made with vanes that rotate and push material towards the beyond the outlet flange. The rotation of the impeller is powered by a motor and the torque transferred through a shaft drive (Cooke, 2006; Ragoth & Natharaj, 2012). The slurry from the pump is then sent to the detoxifier where the chemical content is balanced to acceptable pH values before it can be discarded into the tailings dams (Figure 2.3). The impeller is the most degraded component within the pump.

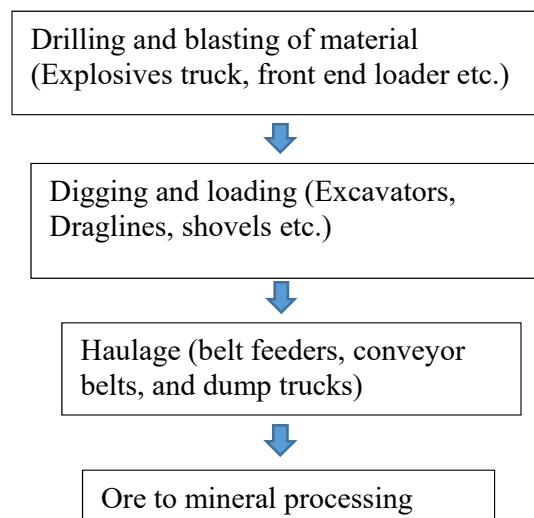


Figure 2.1: Various stages of mining operations.

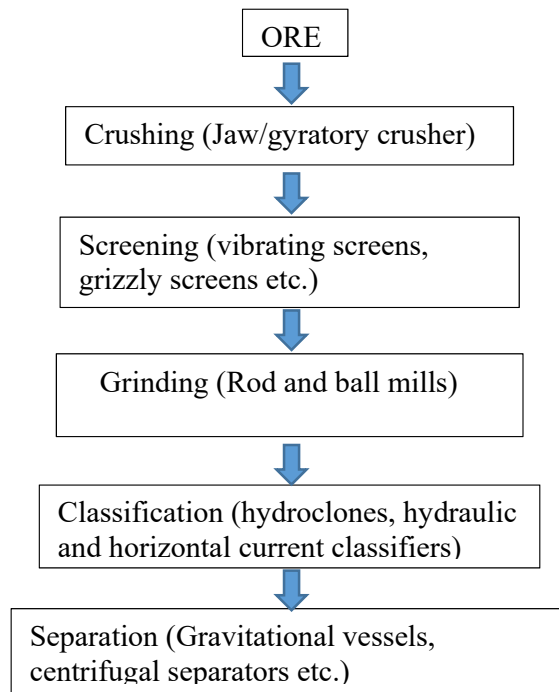


Figure 2.2: A flowchart for mineral processing operations and equipment used.

Impellers are made from stainless steel 300-400 series and grey cast iron (Bloch *et al.*, 2014; Zhang *et al.*, 2013). In a mining equipment, Kato & Adachi (2001) pointed out that wear occurs differently through 6 modes namely (1) abrasive, (2) adhesive, (3) corrosive, (4) fatigue, (5) impact wear and (6) oxidation. Some of these modes of wear are illustrated in Figure 2.4. Adhesive wear (Figure 2.4a) occurs where high bonding strength resists sliding between two surfaces and as a result a deformation more like shearing occurs (Bayer, 2004) whereas when two surfaces are in contact and one micro-cutting through the other the behaviour is known as abrasive wear (Figure 2.4b). In fatigue wear (Figure 2.4c), a number of cycles of sliding or rolling of surfaces on one another is the main cause of wear. More cycles are needed to be able to cause this deformation/wear phenomenon. Corrosive wear (Figure 2.4d) occurs if there is chemical and electrochemical reactions at surface contact thus building a layer on the materials which is then removed by friction (Bayer, 2004; Kato & Adachi, 2001). With impact wear, repetitive percussion occurs on a body. These failure modes have been the root cause for mechanical breakdowns in a wide range of industries and costing large amounts of money. In addition to that, proper

care should be taken to reduce the effects of wear as well as improving the properties of materials used for manufacturing the components so as to prolong their service life.

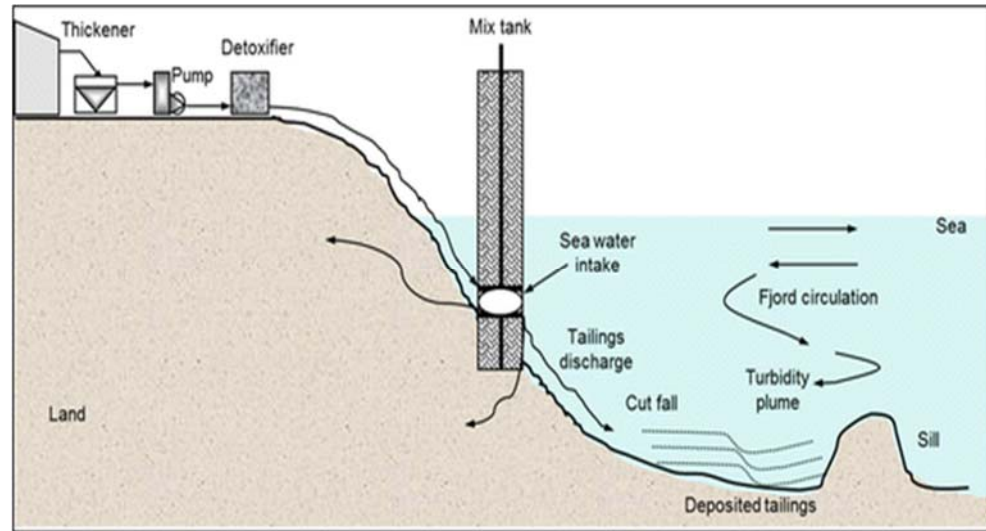


Figure 2.3: Schematic of a slurry pump system (Source: Bascetin *et al.*, 2017)

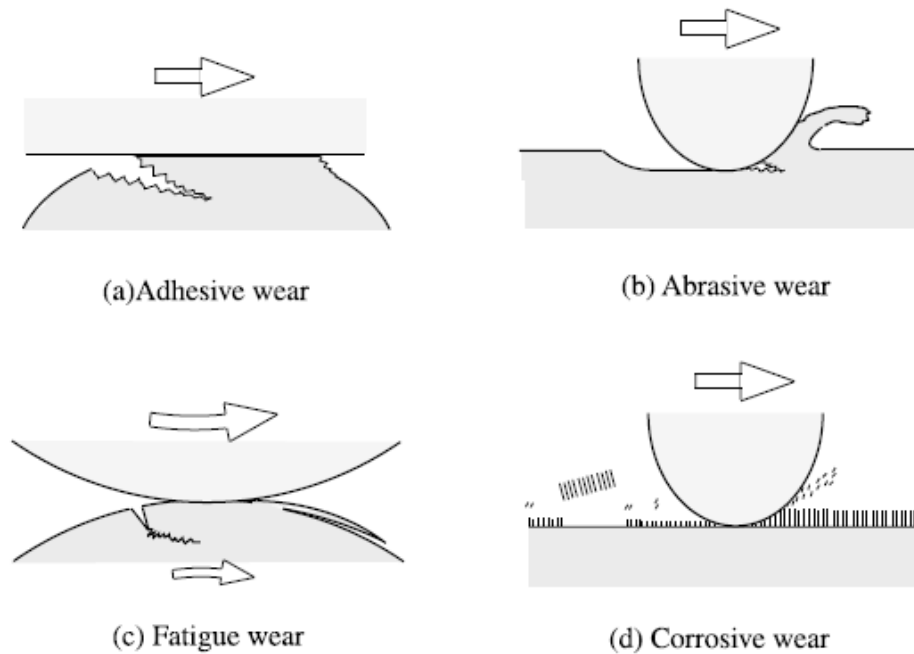


Figure 2.4: Different modes of wear (Source: Kato & Adachi, 2001)

The pump impeller, a subject of interest in this study, encounters high levels of corrosion and erosion degradation due to chemicals attack from processing plants as well as the impact of the particles which damage its surface. The durability of the pump had been related to its operating conditions. According to Sellgren *et al.*, (2005), the wear rate of pump impeller is affected by particle size of the slurry (d_{50}), solids concentrate C_v , pumping speed and total dynamic head of pump (TDH) as shown in Figures 2.5 (a & b). Particle size affects the amount of wear because of the penetrations made by these particles when getting in contact with surfaces. A larger particle size will impose deeper penetrations compared to a smaller one hence an increased amount of wear (Sawyer *et al.*, 2014). As of the pumping speed, the amount of particles passing through a pipe in an hour will vary according to the varying speeds therefore, high pumping speed means more particles passing through and eroding the surface in contact quicker than pumping at lower speeds. Solid concentration contributes to the viscosity of the slurry meaning that if a slurry has high solid concentration, the slurry becomes viscous because of internal frictions of the solids. These solids will increase the amount of friction also occurring in the pump impellers hence eroding them at high rates (Gomez *et al.*, 2015). Having identified corrosion and erosion wear as the main form of degradation encountered in centrifugal impeller pumps as well as the mechanism of its formation, varying types of surface modification techniques which may be engaged in restoring a damaged centrifugal impeller pump to as new will be evaluated in Section 2.2. This is with a view to identifying an appropriate re-manufacturing technology to be explored further for centrifugal impeller pump on the basis of its degradation mechanism it encounters in service.

2.2: Evaluation of various surface modification techniques to restore centrifugal impeller pump to as new.

As mentioned earlier, the goal of this section is to evaluate various surface modification techniques in order to identify the appropriate technology to restore the impeller pump to as new. In selecting various surface modification techniques for evaluation, consideration shall be given to factors such as the provision of functional clads, retaining or improvement of mechanical properties of the component,

sufficient substrate and coating bonding strength and minimum requirements of post treatment activities.

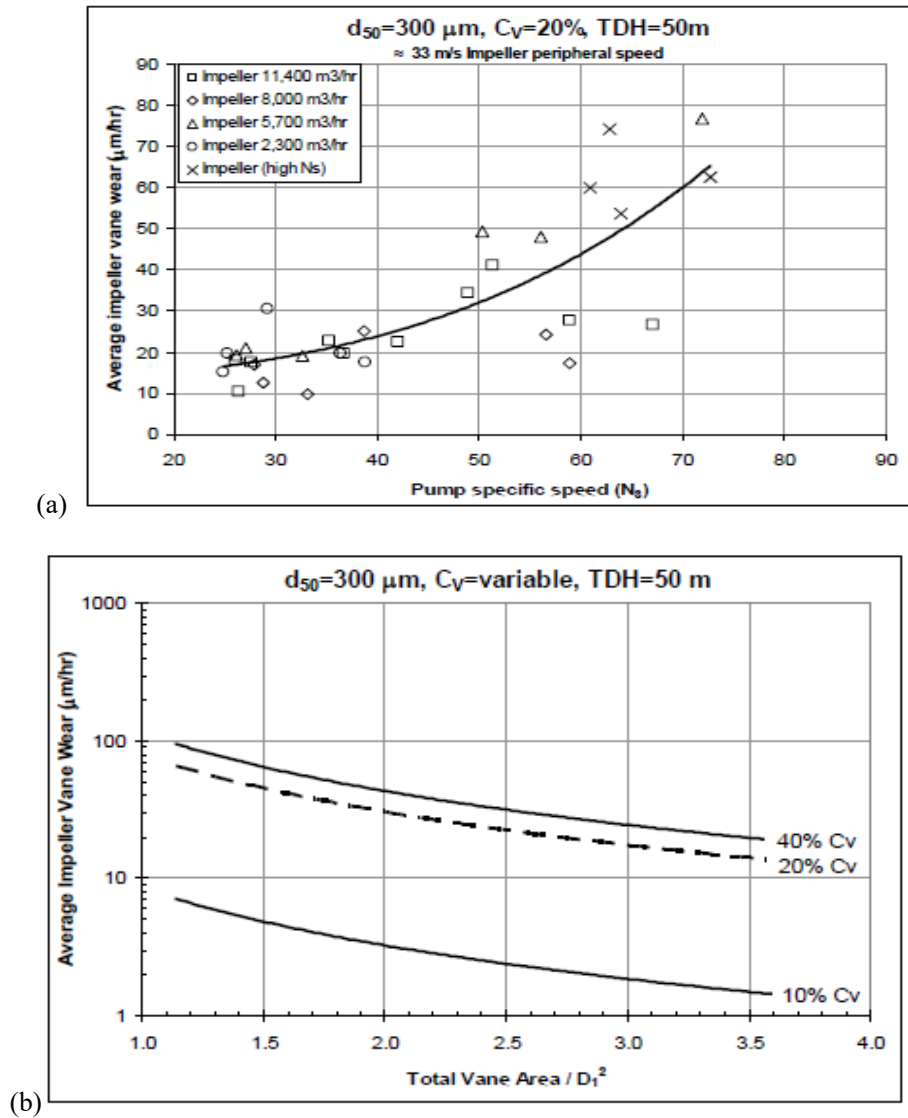


Figure 2.5: Average impeller wear (a) at varying specific pump speeds and (b) at varying solids concentrations. Courtesy of (Sellgren *et al.*, 2005)

Based on literature and common industry practices, there are some surface modifications techniques that are already being used to repair worn out equipment. Such techniques were selected for further discussion and evaluated later on.

Surface modification can be explained as the science of improving the surface geometry or structure of a component and can be achieved through various technologies such as (1) thermal spray, (2) arc welding, (3) polymer coating, (4) laser cladding (LC) and (5) laser assisted cold spray (LACS). These technologies are currently being used in the mining sector. Surface modification techniques are used to serve the following primary purposes namely:

- increase wear, corrosion and oxidation resistance of machine components.
- repair mechanical parts that are already worn out.
- Modify components to suite different functions.

In turn, these primary reasons also contribute the following benefits to equipment owners when they employ surface modification techniques:

- reduced costs of replacing machine components.
- reduced time for breakdowns.
- improved equipment efficiency or machine on ground.
- The time to wait for machine components to be purchased and fitted on the machines is avoided hence productivity in the industry is maintained.

2.2.1 Typical surface modification techniques in the mining sector

2.2. 1. 1 Thermal spraying techniques

Amongst the competing surface modification technologies is thermal spray which involves spraying of heated or melted materials onto surface of substrates to improve its mechanical properties. McDonald (2015) reported that thermal spraying technologies in western Canada were highly used in mining industries with an annual estimate of \$200 million of which \$32 million is used in down-the-hole drilling equipment. In implementing thermal spraying techniques, the feedstock is melted by either by electric arc or combustion flame. Hence, methods of thermal spray can be categorised into: combustion sprays and electric sprays (Espallargas, 2015).

Combustion spray methods use propane, natural gas, oxygen, acetylene, natural gas, propylene and methyl-acetylene to provide the required amount of energy. High velocity oxy-fuel (HVOF) is a variant of combustion spray technique which uses

oxygen and fuel gasses such as propane, propylene, kerosene and hydrogen (Amin et al., 2016). The combustion occurs in a detonation gun (D-gun) which is one of the important components of the system as this is where most of the process mechanism is controlled (Figure 2.6). According to Singh et al., (2012), the process and materials parameters that affect the quality of clads fabricated with HVOF in particular are (1) fuel-oxygen gas ratio, (2) pressure of the detonation wave, (3) gas temperature, (4) powder particle size and (5) spray stand-off distance (Singh *et. al.*, 2012).

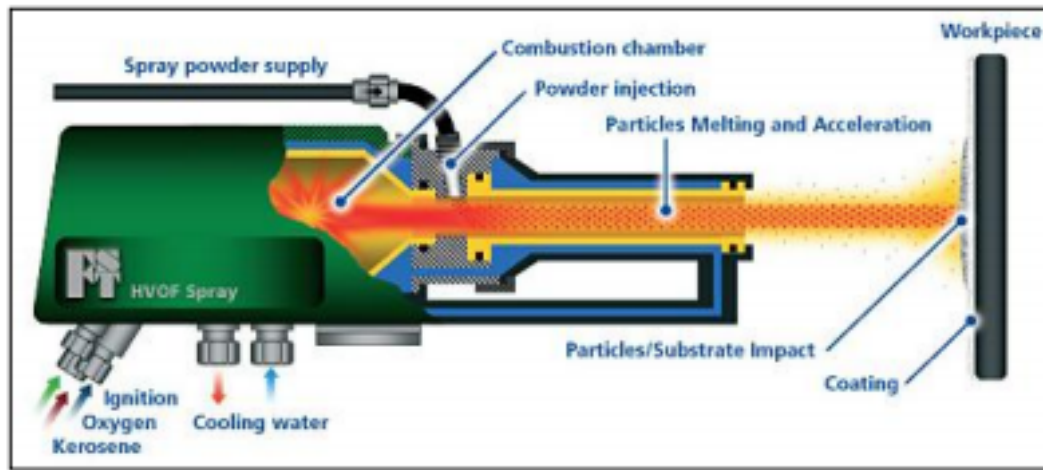


Figure 2.6: HVOF schematic diagram (Source: Amin *et al.*, 2016).

HVOF works with any materials ranging from metals, alloys, ceramics to polymers. This makes it applicable to a wide range of industries including mining. In mining it is used for coating mineral processing pump components by inducing wear and corrosion resistant clads (Kumarasamy *et al.*, 2016). One of the advantages of HVOF clads is that, good functional properties (such as corrosion, temperature degradation resistance etc.) can be attained at a variety of thickness ranging from micrometers to millimeters. However, clads designed from this technique are subjected to porosity and also the process requires mechanical masking in order to coat complex designs which requires time (Mahbub, 2005).

In liquid stabilized plasma spraying (electric sprays), plasma is created by a copper anode and a graphite anode causing evaporation of water within the inner vortex of torch (Figure 2.7). The water flows continuously in the liquid jacket acting as a

coolant to the process. The material is then introduced by a nozzle as a wire, powder or suspension into the plasma jet where it is melted and propelled towards the substrate (Hrabovsky, 2009). Since the particles are molten, they hit the surface then become flattened and solidified upon cooling. This technique can be used to improve surface corrosion, wear and thermal properties.

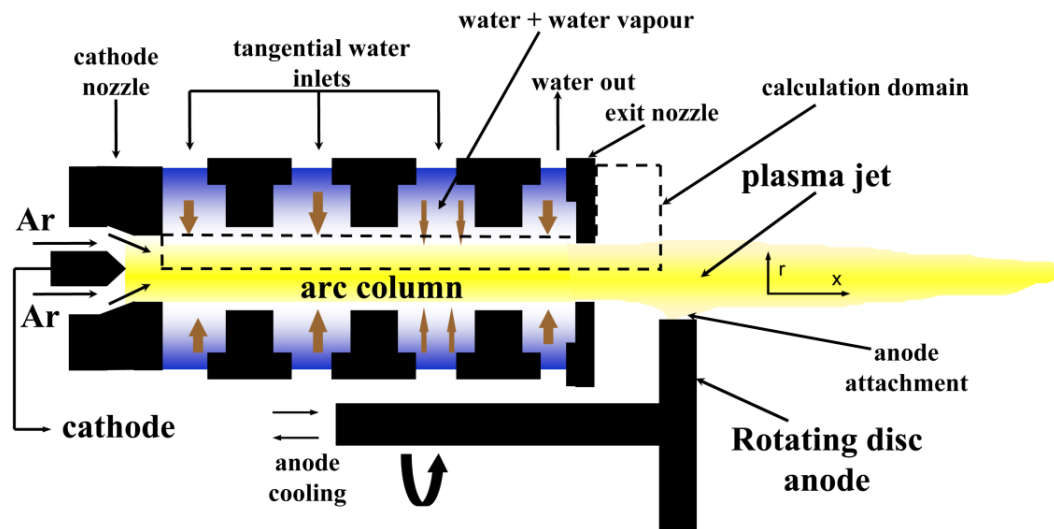


Figure 2.7: Liquid stabilized plasma spraying technique (Source: Hrabovsky, 2009).

The plasma flow rate in this technique cannot be adjusted individually, however, it is controlled by a balance of heat flow in the arc column. Other process parameters include arc current, arc power and mass flow rate. This technique is flexible in terms of the material that can be processed with it such as ceramics, composites, metals and alloys. Also the thickness range can be varied from a minimum of 50 μm to a few millimeters (Vardelle *et al.*, 2015). It has found its applications within industries of mining, aerospace, biomedical, aeronautics and electronics (Jr. Tucker, 1993). Nevertheless, plasma spray clads face challenges of porosity and low bonding strength. The bond between the coating and the substrate depends on the wettability of the jetted particles as they hit the surface.

Thermal spray techniques are considered to be advantageous in a sense that they can provide a wide range of coating thickness from 0.01 μm to 6.5 mm (Davis, 2004). With such a variety of coating thicknesses, thermal spray is applicable to many

industries, also the technology works well with a variety of materials such as metals, ceramics and polymers. Furthermore, the technology can be used to rebuild abrasive worn out components making it suitable to repair worn out pump impellers. Operation of the equipment is mostly automated so a trained technician and understanding of the materials needed will be the special skills required in the process. Despite the merits of thermal spray techniques, it is however disadvantaged by the issue of moderate bonding strength and coating defects (porosity and cracks). Porosity is a major concern as it affects the durability of clads and with residual stresses formed during the cooling processes, cracks can easily form.

2.2.1.2 Arc welding technologies

The variants of this technology are gas metal arc welding (GMAW), shielded metal arc welding (SMAW) and the most common in hard-facing is submerged arc welding (SAW). Arc welding utilises an arc formed from an electrode and work piece to melt and deposit metal powder/ wire fed into the system. A flux is often used to add alloying elements as well as generate shielding gas (Weman, 2011). Quality and quantity of the end product is controlled through welding speed, arc voltage, arc current, polarity, filler wire angle, number of welding wires, type of flux (acid, neutral or basic), size and shape of welding wire. Arc welding technologies are used in the mining industry to repair components of heavy machinery such as wear resistant overlay plates in bulldozer buckets, shovel teeth, dipper bucket liners (Barnes *et. al.*, 2015). Arc welding technologies (AW) are very advantageous because have a high deposition rate, deep penetration and works well with providing thick clads. However, they cannot be used to make thin clads (<1.5 mm) and this limits the applicability of the technologies. Like most of welding technologies, challenges of excessive distortion, heat affected zone (HAZ), dilution, porosity and cracks are found within (Figure 2.8). A very limiting factor with AW becomes the input materials they can work with, they are limited to metals only which means they cannot be applicable to non-metal work pieces. The quality of the weld is even lower, homogeneity is something that cannot be easily achieved.

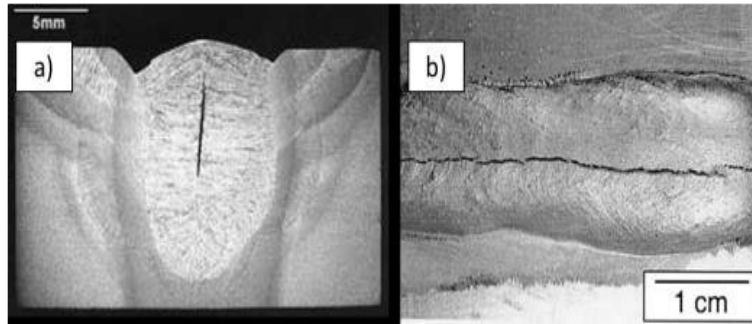


Figure 2.8: Submerged arc welding images of cracked (a) cross section and (b) surface (Source: Aucott, 2017).

Okechukwu *et al.*, 2017 compared the service life of components as well as the level of dilution imparted by different techniques of arc welding. The dilution ratio was calculated from the height of the dilution divided by the total height of the area of dilution and weld height. Table 2.1 reveals that SAW technology imparted the highest dilution ratio whereas oxy-acetylene gas welding (gas welding method) had the lowest. Most of these welding methods require special skills to handle the operation and they are used in mining industries to hard-face components like the excavator bucket teeth. Technical and cognitive skills are the most required with more challenges faced by the technical or operation side due to the complexity of worn out substrate geometries. Arc welding technologies are hand operated requiring too much concentration in order to get good weld alignments and uniformity. This makes the technologies less suitable for rebuilding end of life parts.

Table 2.1: Dilution factors of welding processes. (Source: Okechukwu *et al.*, 2017).

S/N	Welding process	Dilution factors (%)
1	Oxy-acetylene gas welding	0-5
2	Tungsten inert gas welding	5-15
3	Shielded metal arc welding	20-45
4	Flux cored wire arc welding	20-45
5	Submerged arc welding	20-50

2.2.1.3 Polymer coating

Another surface modification technique is the polymer coating method. The technique utilises a heated monomer that is deposited on a substrate and then treated with electron beam, ultraviolet (UV) radiation or infra-red radiation (Yanqin *et al.*, 2018). In the end, the substrate is covered by a polymer appportioning the required properties. The quality of the coating is affected by various material and process variables such as monomer chemistry, coating technique and treating technology (Kausar, 2018). The monomer as shown in Figure 2.9 has to be vaporised and conveyed through a channel till it is deposited on the substrate. Therefore, there is need for the monomer to be able to maintain a uniform vapour pressure before reaching the substrate in order to have a uniform thickness. Also, it has to be less reactive as this will prevent it to react with oxygen thereby reducing defects. Various polymers are used commonly in the polymer coating process such as polyethylene, polyester, polyaniline, nylon and acrylic. The polymer is selected based on different properties provided which can be wear resistance, electrical conductivity, corrosion resistance and other mechanical properties. Sputtering, vacuum thermal and spray coating re some of the coating techniques used in the process and these result in different coating thicknesses that may be suitable for varying applications (Bishop, 2015).

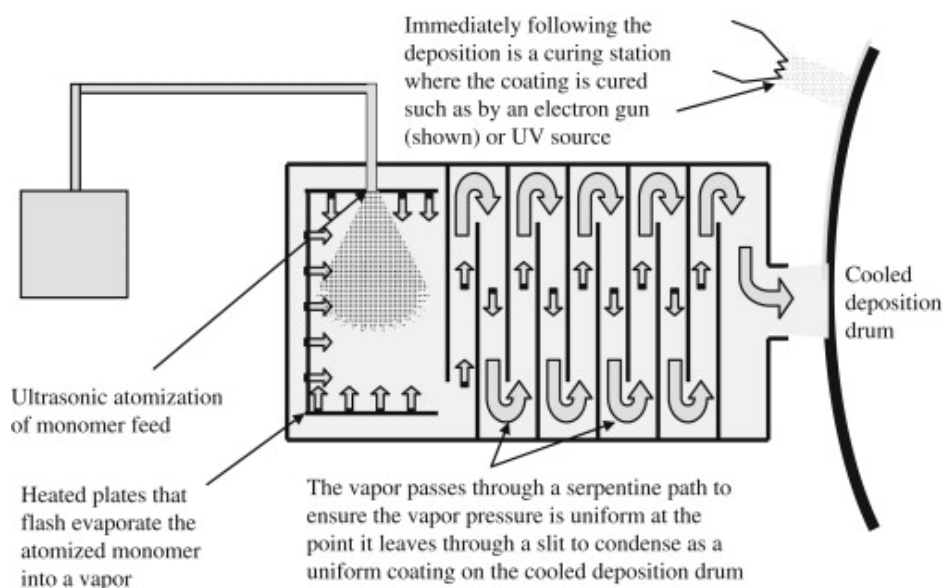


Figure 2.9. Polymer coating process. (Source: Bishop, 2015).

The polymer coating process has seen applications in the fields of biomedical, automotive, electronics and packaging. The technology is able to build clads from nano-scale to a few millimeters with properties required. However, the biggest challenge with polymer coating technologies is fabricating uniform coating thickness and also being able to adjust properties of interest (Kausar, 2018). Also, it has not been explored to address issues of wear in the heavy duty equipment for mining industries but has been used for corrosion protection in equipment such as pipes and tank interiors. The polymer coating technology is essential for coating of components encountering abrasive degradation, however, it cannot be suitable for rebuilding degraded metal components as bonding can be an issue for heavy operations due to materials of different coefficient of thermal expansion. Also, the operation requires polymer clads as well as special understanding of both the technology and the materials.

2.2.1.4: Laser Cladding (LC)

A clad is a bond of two different materials and usually carried out in manufacturing to coat the surface of a substrate with another material with the required mechanical properties. Laser cladding (LC) utilises a laser beam to melt a very thin layer of the substrate/base metal together with the metal powder deposited through a nozzle on the base metal thereby forming a new layer on top of the base. This surface engineering technology has been used in many industries like aerospace, military, mining, oil and gas, agriculture etc. Existing literature indicates that LC is extensively employed in the mining industry for fabricating shearers picks for mining (Torims, 2013). Also, Che *et al.*, (2014) successfully repaired worn out shearers picks used in the coal mining by improving their hardness as well as increasing its service life by 5 times (Figure 2.10). A wide range of materials can be used with LC, from metals, metal alloys to ceramics to deposit clads of varying thicknesses ranging from 5 μm to 2 mm.



Figure 2.10: Worn shearer pick (a) and laser cladding repaired shearer pick (b). (Source: Che *et al.*, 2014).

Variability of coat thickness means LC can be used for different applications. For instance, in mining applications a drill bit operates in a high wear environment made up of rocks of different hardness, therefore a much thicker coating is relevant compared to coating a sports bicycle frame where a lighter weight is essential. LC clads show continuity of mechanical properties or the coating becomes homogenous except on parts where the clad tracks end. Cladding at appropriate process parameters can ensure uniformity of the mechanical properties of the clad which provides a finished product with a homogenous quality. Amongst the properties that ensure the durability of a coating is the bonding strength. The bond strength between the substrate/base metal and the coating should be very high with little inter-metallics involved. The heat affected zone (HAZ) should be very low, it is controlled through the laser power density which is the combination of laser power, scanning speed and diameter of the laser spot. A higher power density can induce more HAZ, so the power density has to be determined at appropriate level which allow sufficient bonding with minimal dilution. However, there is thermal gradient between the base metal and coating, therefore residual stresses develop inside the coating due to rapid cooling rates (Fu *et al.* 2016). Both compressive and tensile residual stresses develop in the coating but when rapid cooling occurs, the tensile residual stresses become dominant over compressive residual stresses and deteriorate the quality characteristics of the coating (Wang *et. al*, 2015; Fu *et al*, 2016). Laser cladding technology can be used to recover abrasive worn out components which makes it worthy for repair of pump impellers. Furthermore, the technology is automated and can be programmed to perform on complex geometries making the technology more

valuable. Skills required are based on the technical operation of the automation and also the cognitive understanding of the technology.

2.2.1.5 Laser Assisted Cold spray (LACS)

Laser assisted cold spray is a modified technique that utilises kinetic energy of gases and laser power to induce fusion of solid particles. The technique uses high speed gases (e.g. nitrogen, argon, helium) to accelerate powdered particles onto a substrate as shown in Figure 2.11. Bonding occurs when the accelerated (at >500 m/s) particles softened by laser energy are impinged onto the substrate surface and undergo plastic deformation because of the impact that is over the threshold limit of the particles' velocity (Irissou *et al.*, 2008; Olakanmi & Doyoyo, 2014). Main processing parameters for the technology are laser power, gas pressure, temperature, powder's density, particle velocity, powder's particle size/distribution and substrate hardness (Cavaliere *et al.*, 2014). LACS can work with a variety of materials such as metals, polymers, ceramics and composites. Therefore, it can be used for a wide range of applications in medical, military, agriculture and mining etc. Applications of LACS also involve aesthetics design on metal for architecture, coating of wind turbines, aircrafts parts, automobiles parts and construction equipment (Villafuerte, 2010). Application to mining involve coating of slurry pump impellers, valves and casings (Mcdonald, 2015). The technology utilises a de Laval nozzle to accelerate particles unto mechanical deformation, hence, it eliminates issues of oxidation, residual stresses and chemical reactions.

However, brittle material cannot be used or sprayed without an addition of ductile binders (Ghelichi *et al.*, 2009). It is also exorbitant to use LACS since high gas flows and consumptions are needed as some gases like helium are expensive. LACS uses supersonic jet compressed gas with gas flow velocities between 500 and 1000 m/s (Grujicic *et al.*, 2003), hence, clads produced by this process are also subjected to high compressive stresses. High compressive residual stresses are essential mechanical properties as they avoid initial crack propagations and improve fatigue life. Despite that, the main challenge is that with CS, high strength and hard metals cannot be processed and also the coating ductility is reduced due to the deformation

of powder particles on impact (Yin *et al.*, 2018). This features gives it limitation in rebuilding of worn out components in need of high strength and hardness properties such as those required by the pump impeller. Even so, the operation of the technology is much easier as the nozzle can be moved freely by hand which can be operated without high level of skills.

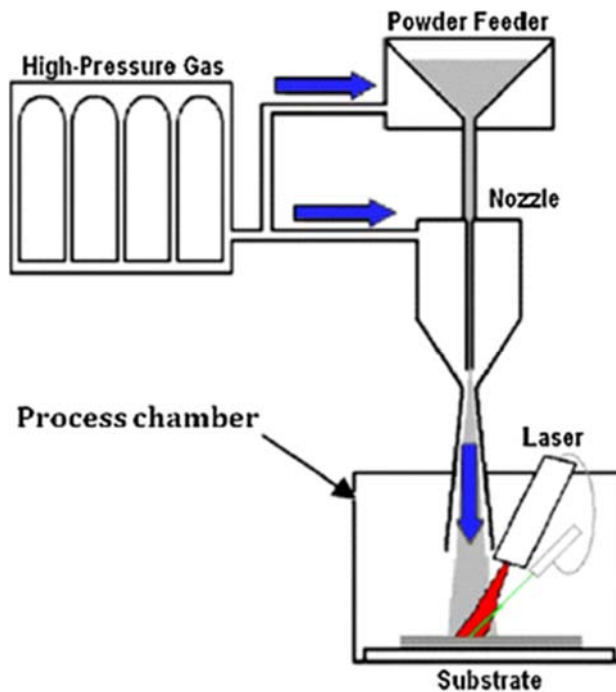


Figure 2.11: Schematic for Cold spray (Lupoi *et al.*, 2011).

2.2.2 Evaluation of surface modification techniques and their co-relation to the mode of wear in impeller pump.

In the review of all surface engineering technologies that are applied in the mining industry, it can be seen that some technologies still need to be improved upon so as to address the issues of wear in the industry. For instance, most of the arc welding technologies induce high localized heat input on the substrate which leads to increased HAZ. Most of the mining equipment especially heavy machinery like loaders which are made from quenched and tempered steels are susceptible to hydrogen induced cracking from weld melt pools, hence, they become brittle during processing (Barnes *et al.*, 2015). Welds which are susceptible to brittleness are not good for application in mining as they will fail due to fatigue. 304L stainless steel,

the substrate of which the centrifugal pump impeller is made of, is found to be affected by hydrogen cracking when repaired with arc welding technologies (Kivisäkk, 2010). Other welding technologies such as SAW have very high heat input that induces a great amount dilution which eventually reduce mechanical properties. However, welding technologies are much cheaper in cost compared to other technologies.

Thermal spraying technologies are applicable to the mining industry, they can retain mechanical properties better than the welding techniques. However, substrate to coating bonding is very weak which means it could not achieve the target of re-manufacturing heavy mining machinery. There is too much porosity with thermal spray with that regard, post treatment is required such as shot peening, re-melting etc. Also, machining is required in order to maintain the required coat thickness. Laser cladding has proven to have better heat input control over other methods and has great substrate-coating bonding. The HAZ can be minimised but at the same time the mechanical properties being retained. Furthermore, the technologies were evaluated based on whether they can be used to restore worn out pump impeller to as new.

Table 2.2: Summary of evaluation of various surface engineering technologies.

Surface Modification Technique	Heat affected zone (HAZ)	Bonding strength	Retaining of Mechanical properties	Porosity level	Capital Investment	Post treatment activities	Ease of operation
Laser Cladding	Low	Very high	Very high	Low	Very high	Machining	Moderate
Laser assisted cold spray	Low	Very high	Very high	Moderate	Very high	surface re-melting, machining	High
Polymer coating	None	Moderate	High	Low	Low	Post heating, machining	Moderate
Arc Welding	High	High	Low	High	Low	Post heating, machining, Machining, Shot peening, Hot Isostatic press	Low
Thermal Spray	Low	Low	Very high	Very high	Moderate		Moderate

*Note that the rating goes from Very high, High, Moderate, low to Very Low.

It was discovered that only thermal spray and laser cladding) are applicable in remanufacturing of worn out pump impeller while other technologies were found to be unsuitable either due to the materials they work with or the need for special technical skills. Of thermal spray and laser cladding techniques, LC remains formidable over its counterpart due to its good features of high bonding strength and low porosity. Table 2.2 above presents evaluation of different surface technology. On the basis of analysis in Table 2.2, LC is now selected as the most suitable re-manufacturing technology for restoring the centrifugal impeller pump to as new.

2.3 Laser cladding: Its nature of laser-materials interaction, processing and materials parameters and their influence on the functionality of the clads.

This section discusses the nature of laser-material interactions as obtained in LC process. Thereafter, the effects of LC process and parameters on the functional performance of the clads are presented. The primary goal of these activities is to identify research areas worth exploring in engaging LC as a remanufacturing technology for centrifugal impeller pump. The functionality of laser clads is dependent on the processing parameters as well as the characteristics of the materials being processed as indicated by Figure 2.12.

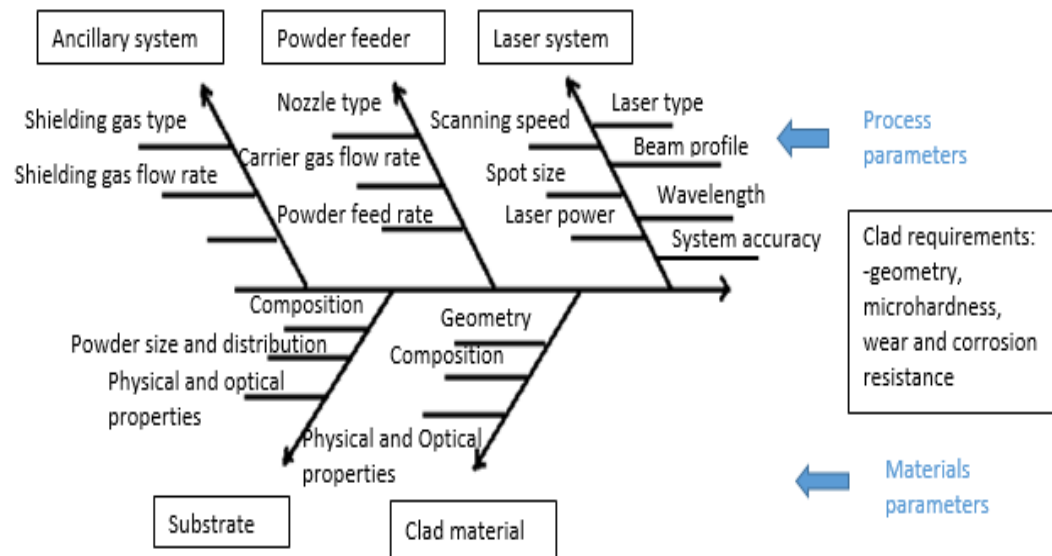


Figure 2.12. Ishikawa diagram for laser cladding process.

Several process controls can be varied in order to achieve a certain quality within clads. Laser parameters controls include laser power, laser scanning speed, angle of deposition, laser beam spot size, laser wavelength, beam profile, beam quality factor, nozzle type, shielding gas and carrier gas. Laser materials processing involves the materials being deposited as well as that of the substrate. Deposited materials affect the qualities of the clads based on feed type (wire or powder), feed rate, chemical composition, particle size and shape (powders) and wire feed diameter. The substrate on which the material is being deposited on will also affect the outcome of the process due to its chemical composition. Figure 2.12 illustrates various process and materials parameters that influence the functionality of laser fabricated clads via Ishikawa diagram. All these factors are set to contribute to the required coating that will be used for re-manufacturing of worn out pump impellers. The clads are deposited to have a better wear and corrosive resistance in order to perform their function in the industry. In the next section, the nature of laser-materials interactions as related to LC process is presented. Some of these laser cladding variables are further discussed in detail below.

2.3.1 The nature of laser-materials interactions and variants of laser cladding process.

Laser cladding process has numerous variants that can be used in different applications and industries. These variants are listed as (1) powder based and (2) wire fed cladding. In the process of powder based cladding process, materials may be introduced as powder fed via the nozzle (Figure 2.13b) or powder pre-placed on a substrate (Figure 2.13a).

In powder-based LC variant, materials are introduced in powder form and sufficient melting is required on both the powders and the base, to allow for sufficient bonding. The common technique of powder fed cladding involves both the material (delivered through a nozzle) and the laser being loaded concentrically on the substrate. The powder is either coaxially loaded with the laser or the powder being deposited from the side with the laser beam at the center. In the process, a controlled amount of laser power and powder feed is required to carry out this operation at a certain stand-off distance. A shielding gas (inert gas) is used to protect the melt pool from oxidation.

With this technology, the clads become more uniform with reduced cracks and porosity.

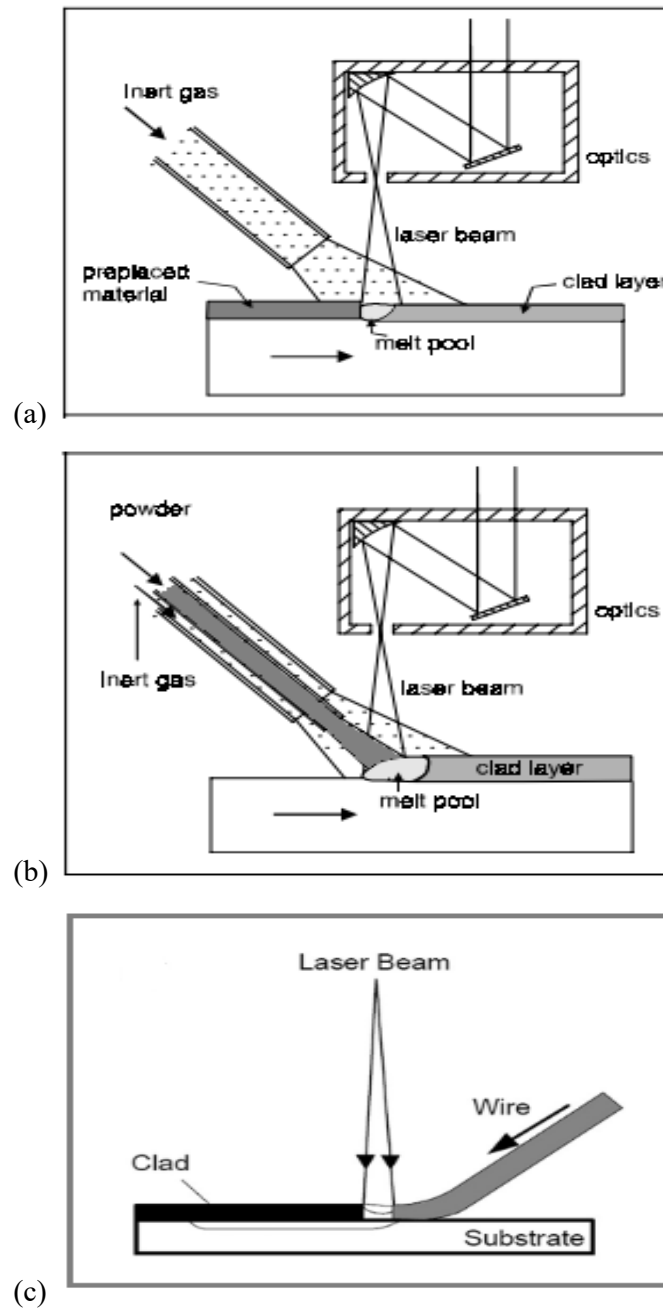


Figure 2.13. Schematic diagrams of (a) preplaced powder feed, (b) powder nozzle feed and (c) wire feed systems. (Source: Liu *et. al.*, 2017; Schneider, 1998).

Pre-placed powder which is common in selective laser melting (SLM) process can also be used in LC as well. Here, the powder is spread on the substrate prior to laser beam scanning. This process has issues relating to proper particle fusion and clad-substrate bonding as the powder may be too much hence shielding the heat from reaching the base or the heat not being sufficient to fuse particles together. In another variant of LC process, the feedstock is introduced into the system as a wire into a melt pool (Figure 2.13c). The wire fed LC process unlike the powder fed system, saves material lost during the process as it is confined as a wire till it reaches the melt pool (Figure 2.13c). However, the quality of the clad tracks from wire feeding are of lower quality compared to powder feeding due to issues of drop transfer. Variables that come with feeding include feed type (wire or powder), nozzle type, feed rate, chemical composition, particle size and shape (powders) and wire feed diameter. Features such as feed type and nozzle type are chosen based on the application of the LC process. Some studies had been conducted based on the suitability of using powder feeding instead of wire feeds or *vice versa*. In a study to clad Inconel 625 with both wire and powder feed, it was found out that better results were achieved at low power and low feed rates when using powder feeding whereas for wire feed a higher power and higher feed rate has to be used (Laschkow *et al.*, 2017). Borges *et al.*, (2010) also found that powder feeding gave high quality overlays with a better controllable dilution compared to wire feeding (Borges *et al.*, 2010). Therefore, on the basis of comparative analysis (Table 2.3) of variants of LC process carried out in this section, it is hereby decided in this research to adopt the powder feed variant for the LC process.

Typical nozzles used in powder feeding systems include (1) continuous coaxial nozzle, (2) 3 way coaxial nozzle system and (3) lateral injection nozzle. These nozzles are shown in Figures 2.14. Lateral nozzles are unidirectional which is simpler and more affordable. The constraints of this nozzle is that it is not flexible and cannot be used in complex geometries. 3-way nozzle systems on the other hand can be used in fabricating complex geometries as they have 3 powder lines which support one another in tilting movements.

Table 2.3: Summary of evaluation of various materials feeding technologies.

Cladding results	Preplaced powders	Wire feed	Powder feed
Particle fusion	Low	Very high	High
Porosity	High	Moderate	Low
Cracks	Moderate	High	Low
Clad-substrate Bonding	Very low	Very high	Very high
HAZ	Very low	High	Low

*Note that the rating goes from Very high, High, Moderate, low to Very Low.

The continuous coaxial nozzle system allows for distribution of powders around all peripheries of the nozzle. The continuous coaxial nozzle is found to be highly efficient in productivity and can be used in areas of complex geometry. It is highly used but very expensive (Arrizubieta *et. al.*, 2017; Zekovic *et. al.*, 2007). In this research, after careful inspection of literature, it is decided upon to use the continuous coaxial nozzle due to its high efficiency.

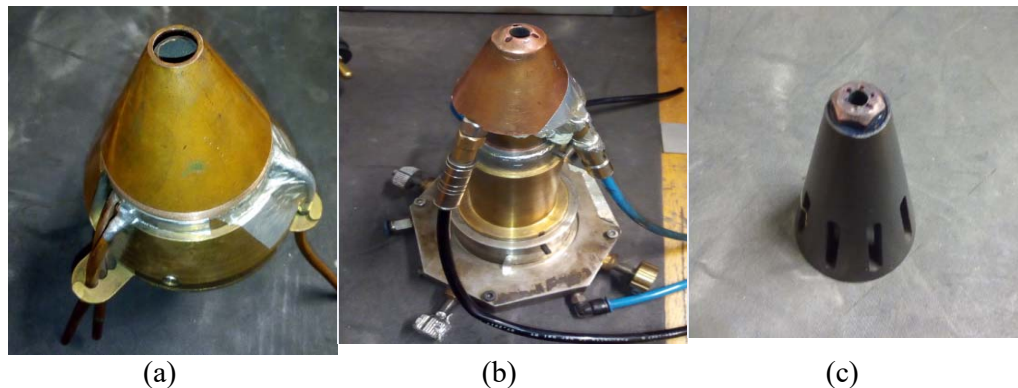


Figure 2.14: Nozzle types for laser cladding at the Council for Scientific and Industrial Research center (CSIR), Pretoria, South Africa (a) Coaxial nozzle (b) 3 Way nozzle (c) Lateral injection nozzle.

Laser-material interactions processes are also affected by the characteristics of the substrates involved. The substrate for cladding can either be metals, metal alloys and composites based on the functionality or the material of interest. The chemical composition of substrates affect (1) the applicability for that material, (2) the quality of the clad and bond and (3) the productivity of the cladding process. Some

composites have high crack sensitivity to heat processes meaning that the use of cladding is not recommended while other materials will provide a high laser absorptivity surface which will result in high deposition rates. High laser reflectivity is the reason for less absorptivity. Metals such as aluminium, iron and molybdenum have high reflectance which means they will result in low laser absorptivity (Naeem, 2013). The chemical composition can also alter the characteristics of the clad due to diffusion of elements between the substrate and the coating (Kusmoko *et. al.*, 2014). In this study, the substrate (304L austenitic stainless steel) was selected based on its applicability. The pump impeller is said to be made from both 300 and 400 series stainless steels (Bloch *et. al.*, 2014). The impeller can be used in areas with high corrosive media. 304L stainless steel has high reflectivity of 98% on a polished surface which reduces with increasing surface temperature. However, the energy transfer efficiency was found to be high from an Nd:YAG laser heated 304L with an estimation of only 10% energy lost as heat to the surroundings (Tadamalle *et. al.*, 2014). A low energy loss will assist in accumulation of heat energy in enhancing substrate melting. The 304L steel has low resistance to corrosion and erosion degradation, therefore, there is need for surface modification (Loto, 2017). Selection of cladding materials is based on corrosion and wear resistance properties and it is found that Cr and Mo content in alloys promotes high corrosion resistance by reducing the current flux that is set to pass through a corrosion scale (Ko *et al.*, 2017). According to literature, the two Cobalt based alloys Tribaloy T800 and Stellite 6 have been used widely in areas of aviation and marine and have high corrosion and wear resistance with either Cr or Mo contents (Barekat *et. al.*, 2016; Navas *et. al.*, 2006). ST6 and Tribaloy T800 have a melting range of 2345-2541 and 2350-2465 degrees Celsius respectively which is not far from melting range of 304L stainless steel at 1399-1454 degrees Celsius. Thermal coefficient of expansion are found to be 12.6 and 14.2 $\mu\text{m}/\text{m},^{\circ}\text{C}$ for a temperature range (20-500 $^{\circ}\text{C}$) which means they can work well with 304 L steel having a coefficient of thermal expansion (CTE) of 18.4 $\mu\text{m}/\text{m},^{\circ}\text{C}$ for a temperature range (0-538 $^{\circ}\text{C}$) (Kusmoko, 2016). Furthermore, the metallic binders within the substrate and the two metal alloys such as Ni and Cr are present within both the substrate and coating powders and they increase the resistance to spallation in a case where there is too much variation of the CTE (Li, 1980).

Laser beam wavelength is also of importance in laser-based manufacturing processes. Most of the industrial lasers for cladding processes recently range in a nano-scale of which is an important aspect in terms of laser material interaction and production. Short wavelength lasers have high laser absorption in comparison to those with longer wavelengths. It was found that a fibre laser (wavelength $\sim 1.06\mu m$) had twice absorptivity than the CO₂ laser (wavelength $\sim 10.6\mu m$) thus having a higher cladding efficiency (Wang *et. al.*, 2016). The laser power has some of the features that are entailed within it, giving its character. These features include laser beam quality factor (M^2) and intensity. They can be used in selection of lasers of better quality for quality assurance purposes. The beam quality factor is calculated from the two equations:

$$BPP = \omega_0 \times \theta_0/2 \quad (2.1)$$

$$M^2 = \frac{BPP}{\lambda/\pi} \quad (2.2)$$

Where BPP is the beam parameter product (BPP), θ_0 is the far field divergence angle, ω_0 is the waist beam radius and λ is the laser beam wavelength. The BPP is the used to rate the quality of the laser. High BPP value will give a lower beam quality and this normally can be depicted graphically (Figure 2.15).

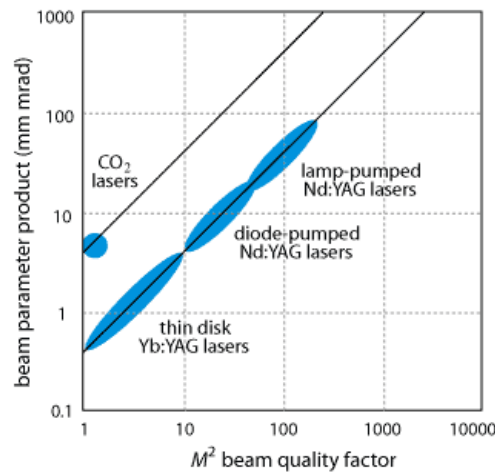


Figure 2.15: Beam quality factor and BPP of different lasers (Source: Paschotta, 2008).

A better beam quality is a result of a better beam coherence and thus leading to a controllable laser power. These also in turn affect other parameters such as focus

distance, spot size and shape. Spot size and shape are very important aspects in cladding processes as they contribute to the laser energy density. The coherence of the laser beam has to be of high quality to allow for a controllable laser beam in the laser cladding process. In summary, it is evident from this section that powder-feed variant of LC process has gained increased popularity as it imparts clads with low porosity, cracks and HAZ in comparison to preplaced powder or wire feeding systems. In addition, the importance of selecting appropriate nozzle systems has been highlighted with the continuous nozzle system identified as being suitable on the premise of its efficiency in terms of productivity as well as in fabricating clads of complex geometry. The roles of material properties (chemical composition of the deposited materials and the substrate) in determining the final properties of the cladding were also discussed. These properties were understood to influence the laser absorptivity, bonding strength, thermal coefficient of expansion and final clad's quality characteristics. The laser beam properties were also discussed and it was established that, the influential properties within the laser beam affecting the outcome of the clad were wavelength, beam quality factor and intensity.

2.3.2 Effects of process parameters on the quality characteristics of laser deposited clads

In this section, the influence of LC process parameters on the processability and quality characteristics of clads made in Stellite 6 and Tribaloy (T-800) clads are discussed. This approach is premised on the compatibility of the thermo-physical properties of deposition materials with 304L stainless steel substrate (pump impeller material) as stated in Section 2.3. Where appropriate literature on the choice materials are not available, studies dwelling on other corrosion- and wear-resistant materials would be employed in elucidating LC processability and its effects on the quality of clads. Firstly, the effects of laser energy density (LED) on the processability and quality characteristics of the selected materials will be considered. LED combines the effects of laser power (P), scanning speed (V) and beam diameter (d). Other parameters to be considered include powder feed rate, chemical composition, flow rates of shielding and carrier gas and powder size and distribution. These parameters are considered critical in manipulating the properties of the LC deposited clads as many findings have indicated the significant influence of these parameters in

controlling the quality characteristics of the clads (Huang *et al.*, 2011; Olakanmi *et al.*, 2019). The processability of the selected materials entails exploration of the nature of process maps which elucidates the consolidation behaviour of the selected materials as LC process and materials parameters are varied. Processability examines desirable regions with less defects such as cracks, lack of continuity of the clad and discolorations. Quality characteristics to be considered include the clad geometry, microstructural evolution, wear- and corrosion-resistance properties. These characteristics are quite relevant to the operation of the impeller pump when conveying slurry materials in the mining environment.

2.3.2.1 Effects of laser energy density (LED) on the processability and quality characteristics of laser clad Stellite 6 and Tribaloy (T-800) clads.

A proper study of operational parameters influencing the consolidation behaviour of laser deposited materials can be explored through the design of experimental processing maps. Process maps are visual illustrations which are used in the scientific developments studies of the LC process based on the operational conditions being explored (Beuth *et al.*, 2013). A search through the available literature yielded no study dwelling on the effects of LC process and materials parameters on the processability of Stellite 6 and Tribaloy (T-800) alloy powders. Hence, studies dwelling on the LC processability of 316L stainless steel powders are employed to understand how process parameters influence the processability of clads. To gain insight into how LED influences the processability of clads, individual roles of its constituents (laser power and scanning speed) will be discussed. Thereafter, the consolidating influence of the LED will be then presented.

Figure 2.16 depicts significant alteration noticed in the processability of 316L stainless steel powders when varying laser power and scanning speed. Observing Figure 2.16 at a fixed feed rate of 400 mm/min and varying laser power, it is clear that the width of the clad tracks is narrow at the lowest laser power input of 2000 W while the tracks widen with every power increment resulting in widest clad track at the highest power input of 4500 W. Drezet *et al.*, (2004) and Goodarzi *et al.*, (2015) reported that if a higher power is used, there is more melting occurring and as a result,

the melt pool will flow outwards due to the Marangoni convection currents which causes spreading of the melt pool (Figure 2.17).

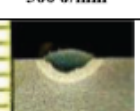
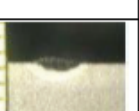
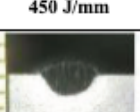
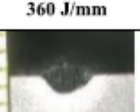
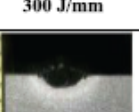
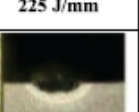
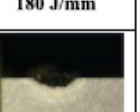
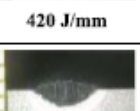
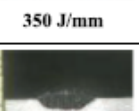
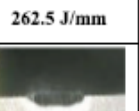
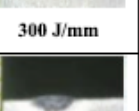
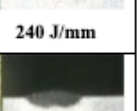
		Cladding Speed				
		400 mm/min	500 mm/min	600 mm/min	800 mm/min	1000 mm/min
Power	2000 W	 300 J/mm	 240 J/mm	 200 J/mm	 150 J/mm	 120 J/mm
	2500 W	 375 J/mm	 300 J/mm	 250 J/mm	 187.5 J/mm	 150 J/mm
	3000 W	 450 J/mm	 360 J/mm	 300 J/mm	 225 J/mm	 180 J/mm
	3500 W	 525 J/mm	 420 J/mm	 350 J/mm	 262.5 J/mm	 210 J/mm
	4000 W	 600 J/mm	 480 J/mm	 400 J/mm	 300 J/mm	 240 J/mm
	4500 W	 675 J/mm	 540 J/mm	 450 J/mm	 337.5 J/mm	 270 J/mm

Figure 2.16. Shows 316L clad cross sections from cladding at a feed rate of 20 g/min (Source: Goodarzi *et al.*, 2015).

It is also evident from Figure 2.16 that the amount of dilution and the shape of the “burn in” are also both influenced by the laser power introduced in the process. For instance, the depth of dilution increased as the laser power was increased from the initial amount of 2000 W to 4000 W but upon reaching a power of 4500 Watts, the dilution depth does not become deeper to follow the trend but rather, it is much wider in comparison to other dilution zones.

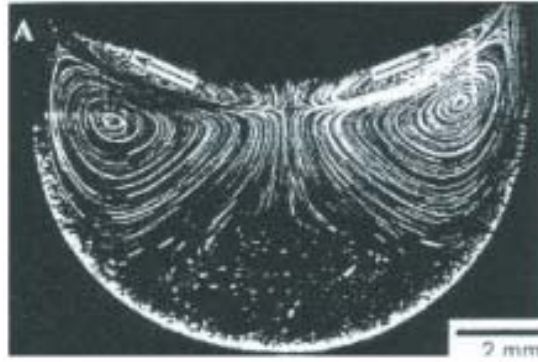


Figure 2.17: Eddies of Marangoni convection forces (Source: Drezet *et al.*, 2004).

According to Goodarzi and co-investigators (2015), Marangoni forces are also reported to be responsible for re-distributing heat within the melt pool and to the substrate, hence, having an impact on the dilution ratio and “burn-in” shapes. Very low laser powers can also introduce micro-porosity due to lack of inter-particulate and substrate-coating bonding (Figure 2.18a). It is better that appropriate laser powers are determined for the process with a view to attaining pore- and crack-free clads (Figure 2.18b). Substrate plate deformations and clad cracks are also some of the effects that can occur due to high laser power processing due to residual stresses upon rapid cooling.

Figure 2.16 also shows a clear difference in the clad geometry due to variation in scanning speed at constant laser power (2000 W). At lowest speed of 400 mm/min, the clads are widest and thickest in cross section. These are the outcomes of a laser beam focused in a single area for a long time before passing onto another thus forcing the material it interacts with to excessively melt. A larger amount of material is melted and in the process there is a building up and flow of the material hence thick clads formed. The flow of material comes about as a result of the difference in the tension in situ of the liquid phase. The thermal gradient between the liquid close to the surface and the one at the center of the clad causes a thermocapillary effect by which the high-tension liquid at the surface attracts the low tension one from the clad center. The effect is known as Marangoni convection currents which causes the variations of clad width and height (Limmaneevichitr *et al.*, 2000). Drezet *et al.* (2004) modelled Marangoni currents to be acting from the center of the clad to the surface and outwards for laser heat treatment resulting in deep and wide weld pools.

Contrary, Gu *et al.* (2015) reported Marangoni currents acting from the surface converging inward into the center of the clad for selective laser melting of Aluminum alloy powders and TiC nano-composites. This variations in the Marangoni forces needs to be studied for the LC process of ST6 and T800 alloys. When the scanning speed is increased, clads become narrower and thinner showing that less material is being melted by the laser-material interaction. Similar findings were reported by Barekat *et al.* (2016) in which upon varying the feed rate and scanning speed, the clad geometry varied. Relating the effect of scanning speed and Marangoni forces, it may be understood that an increased scanning speed means less formation of melt pools in comparison to slow scan rates therefore, the little melt pool formed will have high viscosity which can resist some of the Marangoni forces hence low distribution of the pool and thin tracks. Barekat and co-investigators (2016) also established that the bonding between the coating and the substrate was affected by the scanning speed with effective substrate-coating bonding attained at lower scanning speed of 4 mm/sec while most clads were detached at higher scanning speed of 7mm/s consequent upon lesser amount of heat being dissipated to the substrate due to inefficient laser-material interaction. Laser spot diameter has been found to influence the laser energy input. It is depicted that an increase in laser beam size results in lower energy input or density as the power will be distributed on a wider area. Few literature has studied the effects of the beam diameter on clad characteristics. Spot diameter was found to have a positive correlation with dilution and clad height meaning that, as the beam diameter increases, the dilution and clad height increase (Parekh *et al.*, 2016). In other instances where the feed rate and other variables are constant, beam diameter was found to have influence on the clad width as it determines the size of the melt pool (Chryssolouris *et al.*, 2002).

As noted earlier, LED combines the influence of laser power (P), scanning speed (V) and beam diameter (d) into either of equation 2.3 (LED per unit area) or 2.4 (LED per unit length).

$$\text{Laser energy density (LED)} = P/V * d \quad (\text{J/mm}^2) \quad (2.3)$$

$$\text{Laser energy density (LED)} = P/V \quad (\text{J/mm}) \quad (2.4)$$

High LED can be achieved by combining lower scan speed or lower beam diameter with higher laser power which result in excessive melting. A lower LED can also be achieved by increasing the scan speed or beam diameter with this resulting in a reduced amount of melt pool. Cladding involves thermal processes, therefore, chemical reactions occur in the materials involved. Hence, there is a need for shielding the materials in process against oxidation by using inert gasses. Oxidation in metal clads results in discolourations and may also make them susceptible to cracking. Oxidation can inhibit some of the mechanical properties of materials. For instance, if a material oxidises it becomes susceptible to a shorter stress-rupture life making it weak (Liu *et al.*, 2015). Figure 2.16 confirms that alteration in laser power and scanning speed resulted in the dissipation of varying degree of LED (J/mm) which imparted substantial difference in the geometry of clad tracks. For examples, if more heat is dissipated during the LC process, more heat is then re-distributed to the substrate through the convection currents and more dilution occurs from increased melting of the substrate. The difference occurring with “burn-in” shapes as laser power is varied could be attributed to difference in LED dissipated. The difference in LED input is one of the factors controlling the Marangoni forces. A low LED input results in high viscosity of the melting pool, hence a lower Marangoni intensity (Antony & Arivazhagan, 2015).

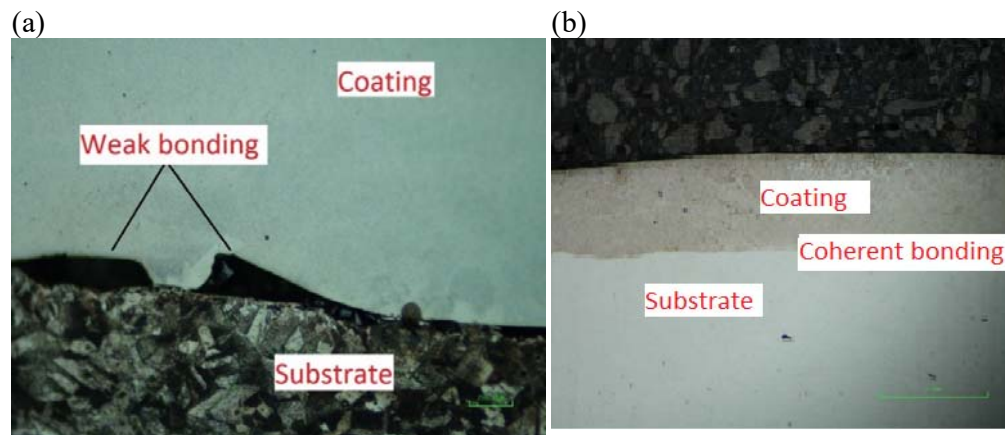


Figure 2.18: Typical clads showing (a) weak bonding between the coating and the substrate and (b) proper fusion between the substrate and the coating.

A search through the literature reveals that no study had analysed how LC process parameters alter the microstructure of Tribaloy 800 clads. Hence, it was decided that

studies which describe the nature of microstructure in these clads at constant LC parameters be employed so as to gain insight into the microstructural evolution from the substrate-coating interface to the surface of the clads. Analysis of the microstructure of LC deposited Tribaloy 800 clads at optimised parameters by Navas *et al.*, (2006) revealed the existence of a planar crystallization zone close to the interface. Also, cellular and dendrite crystallization were identified from above the interface to the surface of the laser track while the overlap zone between tracks was characterised coarse microstructure and the formation of a lamellar eutectic phase (Figure 2.19). Navas and co-investigators (2006) accounted the microstructural evolution reported for Tribaloy 800 as follows: (i) the existence of a planar crystallization at the substrate-coating interface is attributable to the substrate acting as a heat sink at the interface which resulted in high (G/V) ratio obtained from high temperature gradient (G) and low solidification velocity (V); (ii) the existence of a cellular and dendrite crystallization structure (Figure 2.19b) observed just above the interface as well as a fine dendritic microstructure (Figure 2.19c) in the central area of the laser track could be explained by continuous decreasing of G and the progressive increasing of V ; (iii) the double scan of the laser resulted in heating above the eutectic temperature thereby dissolving part of the dendrite formed and subsequent cooling of a coarse microstructure (Figure 2.19d) resulting in a eutectoid reaction. Table 2.4 indicates that the dendritic zone, characterised with hard and corrosion resistant intermetallic laves phase in the microstructure of Tribaloy 800 is richer in molybdenum (Mo) and silicon (Si) while the inter-dendritic zone has higher proportion of solid solution of fcc-cobalt (Co), iron (Fe) and chromium (Cr). According to Navas *et al.*, (2006), the formation of dendritic zone is induced by the high level of Mo and Si which were added in quantities exceeding their solubility limit. They also pointed out that low carbon content in Tribaloy 800 does not promote the precipitation of M_7C_3 carbides. X-ray diffraction analysis revealed the existence of three phases namely: (i) matrix fcc-Co based solid solution (ii) laves phase (Co_3Mo_2Si) and (iii) eutectic lamellar structure (Co_2MoCr) in the coating and revealed their crystalline structure (Figure 2.20). Table 2.5 highlights the importance obtaining a homogenous composition throughout the thickness of the LC fabricated coating through appropriate selection of LC process parameters. This implies that appropriate LC parameters allow the degree of dilution to be manipulated without inducing undesirable changes in the alloy's initial chemical composition.

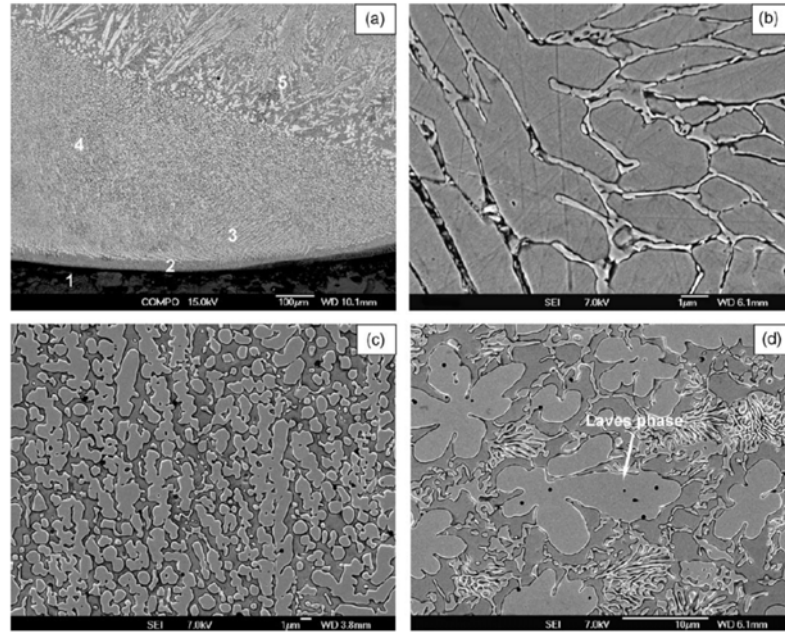


Figure 2.19: SEM morphologies of the laser clad layer: (a) back scattered electron image showing: (1) substrate; (2) planar crystallization region; (3) cellular growth zone; (4) fine dendritic microstructure; (5) overlap zone between tracks. Secondary electron images of: (b) a detail of the cellular structure near the planar crystallization; (c) the dendritic microstructure of the track center zone; and (d) the morphology of the overlapping zone (Source; Navas *et al.*, 2006).

Table 2.4: Chemical composition (wt.%) of the different phases obtained in LC fabricated Tribaloy 800 clads by EDS analysis (Source: Navas *et al.*, 2006).

	Co (wt%)	Cr (wt%)	Mo (wt%)	Fe (wt%)	Ni (wt%)	Si (wt%)
Dendrite	37.09	11.98	41.61	3.76	5.47	0.09
Interdendritic zone	56.78	19.74	13.32	7.61	1.85	0.82
Lamellar structure	48.6	20.47	22.07	5.84	2.68	0.34

Table 2.5: Chemical composition (wt.%) of T-800 laser coating obtained by EDS and GDL analysis (Source: Navas *et al.*, 2006).

	Co (wt%)	Cr (wt%)	Mo (wt%)	Fe (wt%)	Ni (wt%)	Si (wt%)
EDS surface	44.16	16.01	27.07	5.71	3.78	1.06
EDS central area	42.23	15.21	28.80	6.81	3.75	1.17
EDS interface	44.06	13.67	30.05	5.43	3.70	1.09
GDL surface	42.50	17.90	27.30	6.60	3.40	1.10

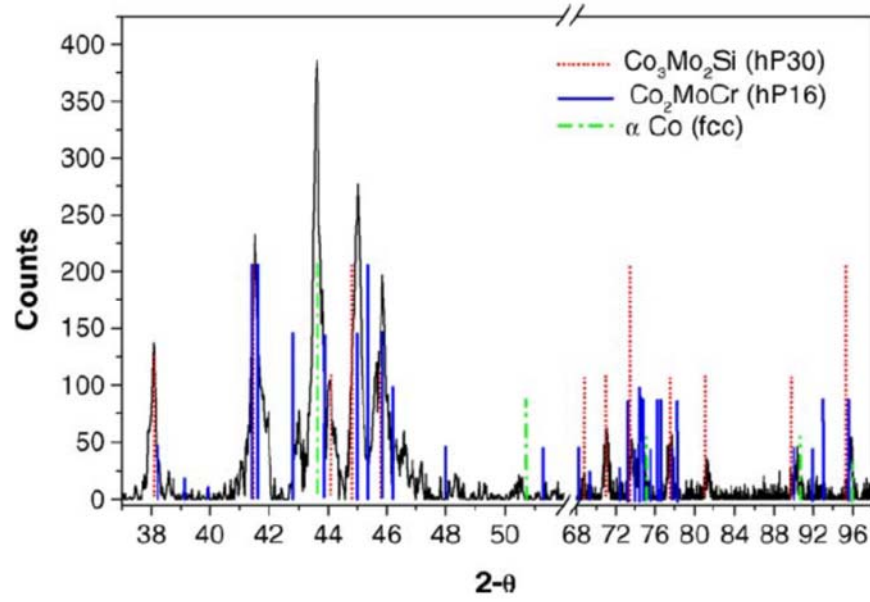
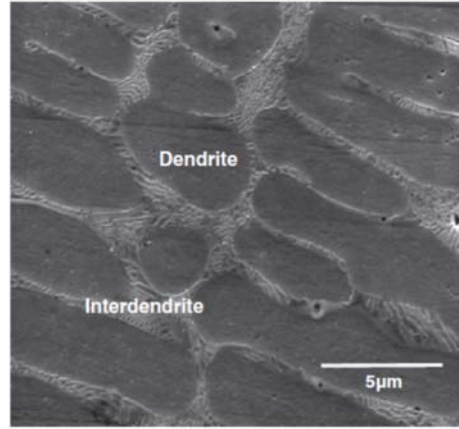


Figure 2.20: XRD pattern of the layer of LC fabricated Tribaloy 800 coating (Source: Navas *et al.*, 2006).

Singh and co-investigators (2014) investigated how alteration in LED (from 32 to 52 J/mm²) influences microstructure, microhardness, solid particle erosion (SPE) and cavitation erosion in 3.5% NaCl solution of LC fabricated Stellite 6 deposited on stainless steel 13Cr–4Ni. They pointed out that LC deposited Stellite 6 clads formed fine dendritic microstructure irrespective of LED adopted for processing. This is accounted for by rapidly solidifying microstructure which allows a small volume of molten metal to be cooled through a sink of substrate resulting into large cooling rates. Furthermore, the microstructure (Figure 2.21) of laser cladded Stellite 6 coating has hypoeutectic structure containing Co-rich dendrites and Cr-rich inter-dendrites. The two regions in the microstructure are compositionally different at all LEDs studies as evident in Table 2.6 which shows that the dendrites consist of a super-saturated solid solution of Co–Cr with specific orientations and inter-dendrite (white phase) along grain boundary.



(a)

Figure 2.21: Typical microstructure of LC Stellite 6 coating showing dendrite and inter-dendrite regions (Source: Singh *et al.*, 2014).

Table 2.6: Chemical composition of dendrite and inter-dendrite regions in LC deposited Stellite 6 coating (Source: Singh *et al.*, 2014).

Elements	Laser energy density			
	32 J/mm ²		52 J/mm ²	
	Dendrite (Matrix)	Interdendrites (Grain boundary)	Dendrite (Matrix)	Interdendrites (Grain boundary)
C	0.46	1.19	0.44	1.97
Cr	23.70	43.62	23.91	59.28
Fe	3.20	2.90	4.10	2.24
Co	66.79	44.1	62.24	23.67
Ni	3.41	1.93	3.23	1.67
W	3.44	6.27	3.67	7.40
O	1.52	2.39	1.68	2.95

Table 2.7: Bulk composition of clad surface at various laser energy densities (Source: Singh *et al.*, 2014).

Elements	Composition at laser energy density			
	32 J/mm ²	37 J/mm ²	46 J/mm ²	52 J/mm ²
C	1.41±0.19	0.97±0.28	1.05±0.3	1.7±0.54
Cr	29.02±0.2	28.1±0.07	26.67±0.39	27.67±0.17
Fe	2.74±0.4	4.2±0.23	11.54±0.87	11.64±2.79
Co	58.9±0.18	58.45±1.16	51.96±1.16	50.11±2.41
Ni	2.02±0.29	2.55±0.55	3.65±0.35	4.02±0.83
W	4.93±0.09	5.01±0.67	3.88±0.24	3.83±0.46

Table 2.8: Corrosion parameters obtained for untreated and laser clad stainless steels (Source: Singh *et al.*, 2014).

Specimen untreated and laser clad	E_{corr} (mV)	I_{corr} (A/cm ²)	E_{pk} (mV)
Untreated 13Cr-4Ni	-383	4.53×10^{-6}	-41
Untreated AISI 304	-112	29.3×10^{-9}	244
32 J/mm ²	-130	4.2×10^{-9}	55
37 J/mm ²	-149	12.2×10^{-9}	379.8
46 J/mm ²	-250	206×10^{-9}	320
52 J/mm ²	-294	185×10^{-9}	16.6

Increased LED resulted in increment in Cr content in the inter-dendritic zones accompanied by reduction in Co content while no alteration in chemical composition was reported in the dendrite region irrespective of LED adopted. The work of Singh and his team provide insight on how elemental diffusion across various regions of coating's microstructure could induce microstructural evolution as laser process parameters are varied. Findings from this study also revealed that corrosion resistance (Table 2.8), microhardness (Figure 2.22a), SPE (Figure 2.22b) are highest at the lowest LED. They attributed the most improved resistance to SPE and cavitation obtained at the lowest LED to the dimensionless parameter which relates to kinetic energy and elastic recovery respectively. Navas *et al.*, (2006) evaluated the microhardness and room temperature sliding wear resistance of LC deposited Tribaloy 800 clads. The coating was characterised with microhardness close to 850 HV_{0.3} and a wear coefficient (k) between one and two orders of magnitude lower than the substrate (Figure 2.23). The analysis of the clad worn surfaces suggested a transition from an adhesive-oxidational mechanism to a more severe plastic deformation and crack formation wear process as applied load increases.

Analysis of the cited literature in this section reveals that, so far, no study had related the wear- and corrosion-resistance as well as microstructural evolution of Tribaloy 800 to LC parameters. As noted earlier, both Stellite 6 and Tribaloy 800 clads are candidate deposition materials for 304L stainless steel on the basis of their thermo-physical properties. Hence, no systematic study which investigates their processability, microstructure and mechanical performance via a processing map had been carried out.

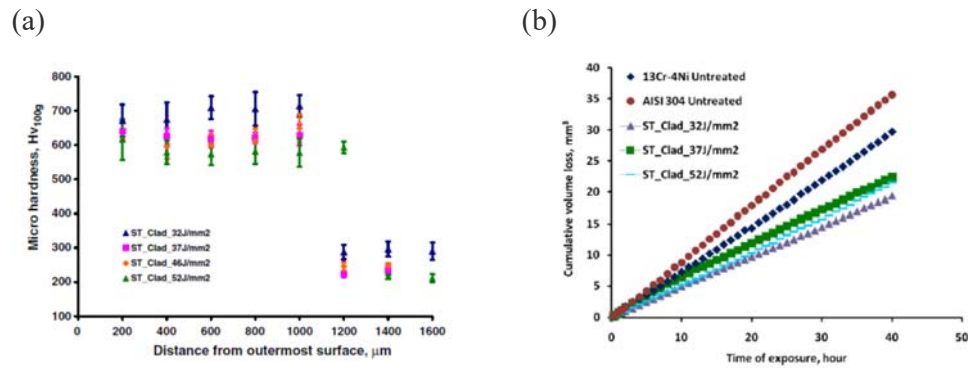


Figure 2.22: (a) Hardness profile of clad formed at different energy densities (b) Cumulative volume loss after erosion at different energy densities compared with untreated substrate (Source: Singh *et al.*, 2014).

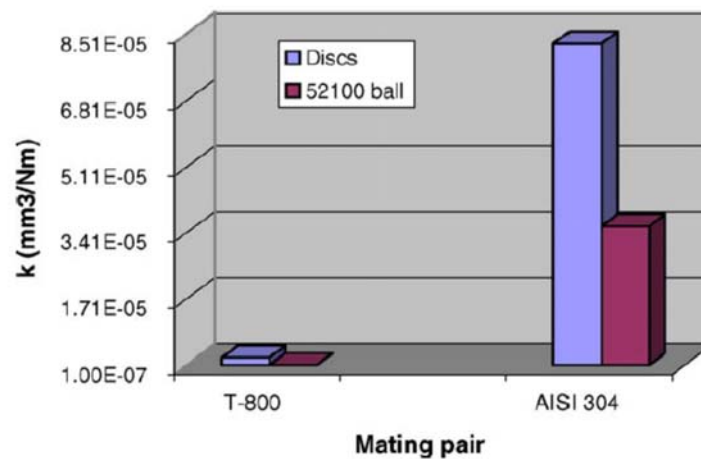


Figure 2.23: Wear coefficient of the confronted tribological pairs for a contact frequency of 3.98 Hz and a normal load of 3N (Source: Navas *et al.*, 2006).

Process maps serve the purpose of identifying a range of acceptable processing conditions for meeting the desired surface coating requirements and therefore, results obtained from LC experiments without investigating process maps are seemingly unfounded and lacking scientific basis. To determine the most suitable deposition material for 304L stainless steel, it is important that a systematic study which employs processing maps dwelling on the same range of LC parameters are adopted as a basis for comparison of the processability and microstructural properties of Stellite 6 and Tribaloy 800. In addition, current literature investigating laser processability of materials have only employed physical geometry and surface morphology of the clads in identifying process regions for different materials

without correlating their findings to detailed microstructural and chemical analysis of the deposits across the regions of the processing maps.

2.3.2.2 Effects of Feed rate on the processability and quality characteristics of laser cladded Stellite 6 and Tribaloy (T-800) clads.

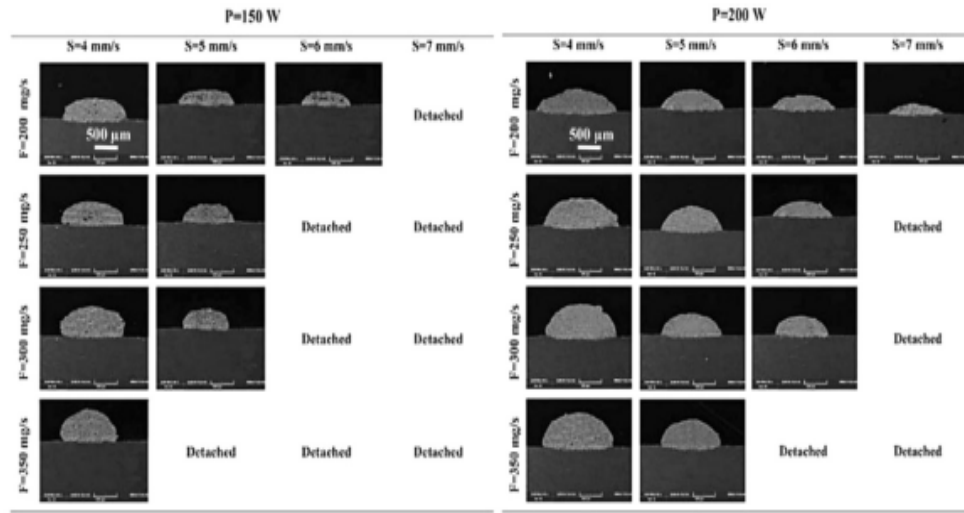


Figure 2.24: Track cross sections at different processing parameters. (Source: (Barekat *et al.*, 2016).

The amount of material fed into the laser cladding system also plays critical role in controlling the quality characteristics of clads. Figure 2.24 shows the effects of feed rate on the cross sections of the clad tracks. By holding constant the scanning speed and laser power at 4 mm/s and 150 W respectively as shown on Figure 2.24, it is observed that a feed rate of 350 mg/s provides clads with greater thickness compared to those with lower feed rates of 300, 250 and 200 mg/s (Barekat *et al.*, 2016). This is simply due to the amount of material melted, if more material is introduced into the process then more material will be melted depending on the energy density. Thermocapillary forces will act within the pool causing the low-tension liquid to be drawn to the surface of the melt pool thus spreading the material outwards which results in thick and wide clad tracks. Excess material tends to shield heat from reaching the substrate, hence, the clad becomes detached from the base as shown in Figure 2.25. Goodarzi *et al.*, (2015) also proved that the amount of substrate being melted reduced with increasing powder feed rates. (Figure 2.25). In order to have material and heat flow within the melted the material, therefore, excess material

results in formation of a little melt pool or a pool that is high in viscosity. Viscosity is found to be able to disrupt Marangoni forces therefore poor flow and heat transfer (Gu *et al.*, 2015).

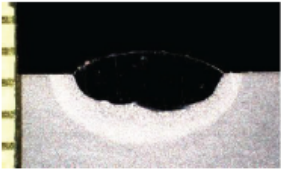
Powder feeding 20 g/min	Powder feeding 30 g/min	Powder feeding 40 g/min
		
Power : 4500 W	Power : 4500 W	Power : 4500 W
Cladding speed: 400 mm/min	Cladding speed: 400 mm/min	Cladding speed: 400 mm/min
Substrate melted area: 4.35 mm ²	Substrate melted area: 3.17 mm ²	Substrate melted area: 2.41 mm ²

Figure 2.25: Effects of varying powder feed rates on the substrate melted area.
Source: (Goodarzi *et al.*, 2015).

The feed rate is believed to have strong influence over the solidification phenomenon and the resultant microstructural evolution of laser deposited clads (Mahamood and Akinlabi, 2017). Figure 2.26 shows the microstructures at different feed rates. At a lower feed rate of 1.44 g/min, it can be seen that there is a formation of a thick lath of a Widmanstätten grain structure of Ti6Al4V due to a feed which induces excessive molten pool due to the laser energy density more than sufficient to melt the available powdered materials.

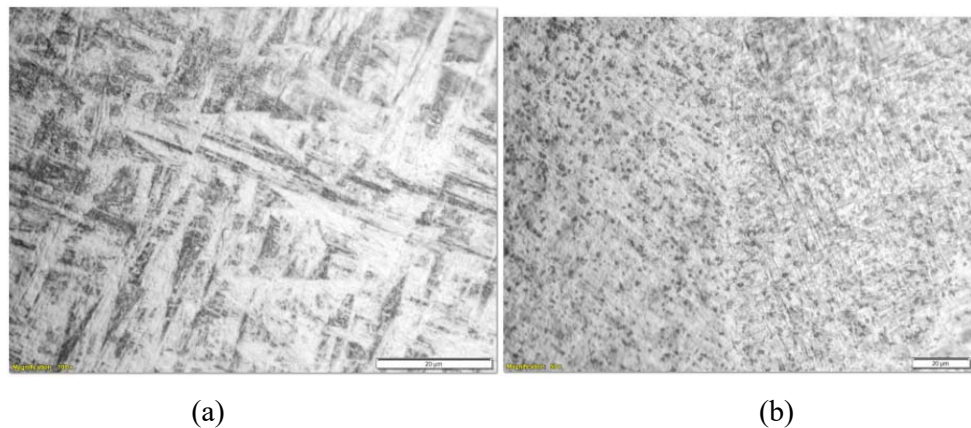


Figure 2.26 (a) Micrograph of clad at powder flow rate of 1.44 g/min and scanning speed of 0.05 m/s (b) micrograph at powder flow rate of 5.76 g/min and scanning speed of 0.05 m/s (Mahamood & Akinlabi, 2017).

Consequently, this melt pool solidifies after a long period allowing grains to grow in size (Figure 2.26a) in comparison to those which are smaller from high feed rates. Higher powder flow will result in a reduced amount of melt pool hence quick solidification occurs and small sized grains in the structure (Figure 2.26b).

Even though, few studies have related the powder feed rate to microhardness, wear and corrosion resistance, it has been found that the feed rate is a significant parameter in the optimisation of clads. Huang *et. al.*, (2011) studied the effects of feed rate on microhardness and it was established that there was a positive correlation between the two (Figure 2.27). Literature availability on the relationship between the powder feed rate against wear and corrosion resistance is quite scarce. Hence, further study exploring how the feed rate alters the chemical composition, microstructure, wear and corrosion resistance of ST6 and T800 clads would be desirable.

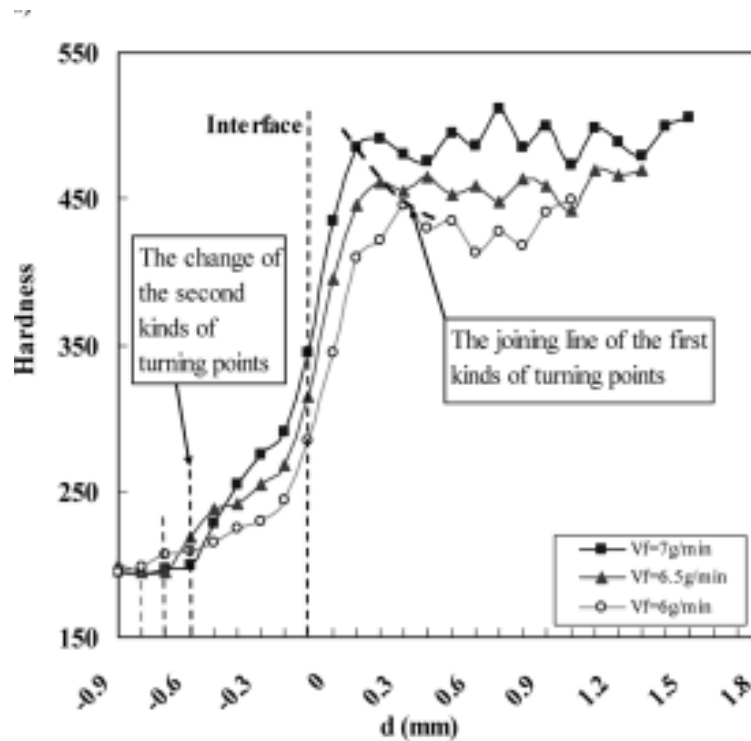


Figure 2.27 showing the correlation between feed rate and microhardness (Source: Huang *et. al.*, 2011).

2.3.2.3 Effects of powder particle size and distribution on the processability and quality characteristics of laser cladded Stellite 6 and Tribaloy (T-800) clads.

One of the important material parameters which influence the quality characteristics of laser fabricated clads is the powder particle size and distribution. For instance, the microstructure of a clad is believed to be affected by the particle size and distribution as pointed out by Chong *et al.*, (2018). In a study to investigate the particle size effect of in-situ (Ti-V)C reinforced Fe-based laser cladded layers, it was found that finer particles had a high laser absorption efficiency compared to coarser particles (Chong *et al.* , 2018). This was seen when more heat input was achieved for S3 (23-38 μm) sample rather than in S1 (180-380 μm) sample. Also, the clads were much uniform for S3 sample in comparison with the other sample for the same energy input (Figure 2.28).

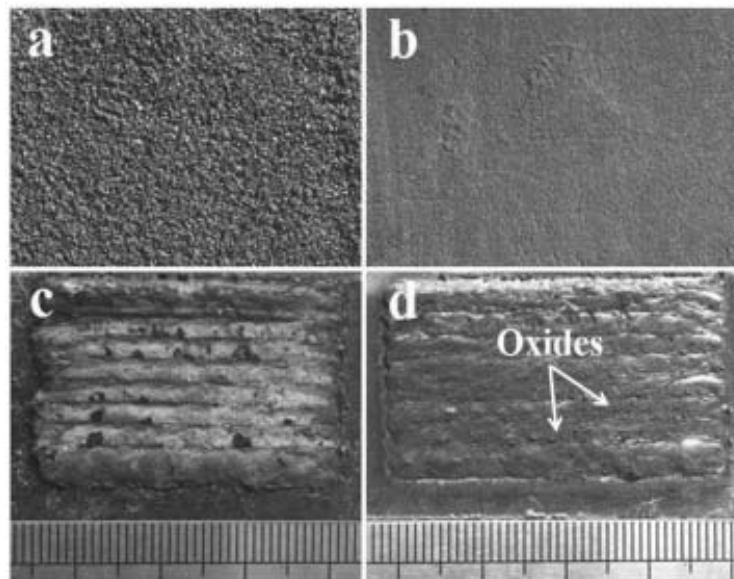


Figure 2.28. Indicating (a) S1 coarse powder, (b) S3 fine powder, (c) S1 clad tracks and (d) S3 clad tracks. Source: (Chong *et al.*, 2018).

Chong and co-investigators (2018) also reported that finer powders are subjected to more intense oxidation compared to their coarse counterparts. Therefore, it was seen that the grain size becomes larger in clads made from finer particles as shown in Figure 2.29. This may be due of excessive melting of the fine particles which allowed

the formation of bigger grains from the slow solidification rate. This outcome was also confirmed by Heigel *et al.*, (2016) who reported that finer particles melted easily.

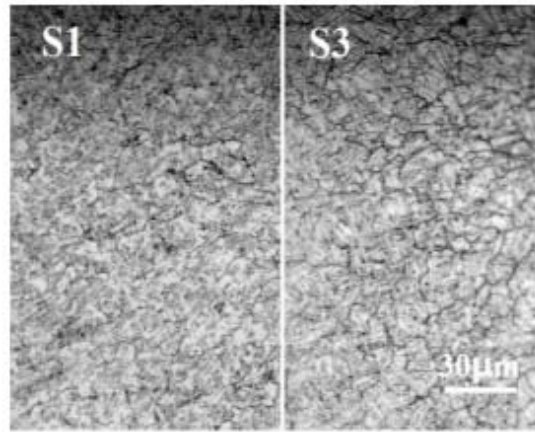


Figure 2.29. The microstructures of S1 (coarser particle feed) and S3 (finer particle feed) respectively. Source: (Chong *et al.*, 2018).

Particle size and distribution does not only affect the microstructure but also other functional parameters which are related to the microstructure such as hardness and corrosion resistance. However, literature relating powder particle size with other mechanical properties of laser fabricated clads is limited. Chong *et al.*, (2018), related the particle size with both microhardness and corrosion resistance and established that the microhardness varied as listed; 180-380 μm (~700 HV), 75-150 μm (~850 HV) and 23-38 μm (~550 HV). In terms of corrosion resistance (Figure 2.30), the finest particle distribution (23-38 μm) showed a high corrosion resistance followed by the most coarse particle distribution (180-380 μm) and the size distribution 75-150 μm being the least corrosive resistant (Chong *et al.*, 2018). Meanwhile, it is recommended that powder's particle distribution falling within the range of 40-150 μm be employed in developing wear-and corrosion-resistant coating for mining industry as it provides a compromise in optimising both wear-and corrosion-resistance required for pump impeller.

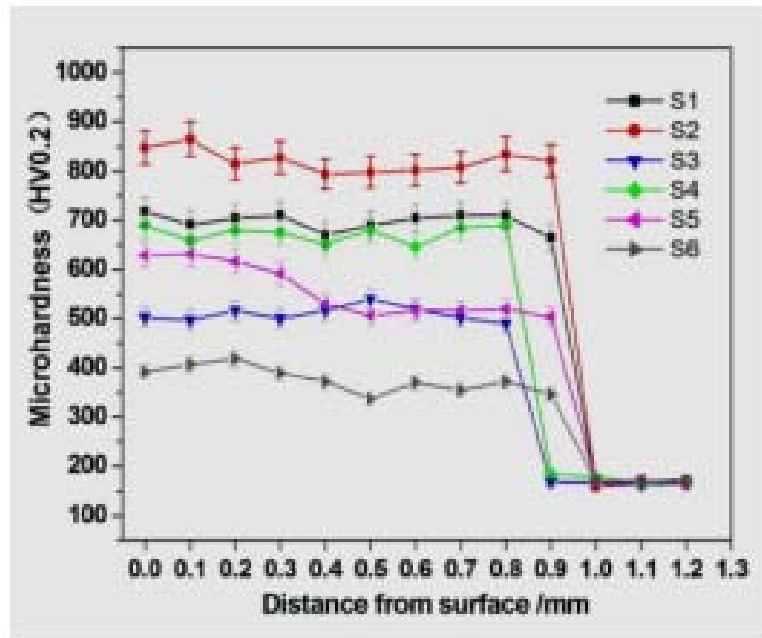


Figure 2.30. Showing the relationship between microhardness and powder size distribution of S1 (180-380 μm), S2 (75-150 μm), S3 (23-38 μm) and the remaining samples with mixed sizes (Chong *et. al.*, 2018).

2.4 Conclusions

This chapter introduced the operating conditions under which the slurry pump impeller is employed in the mining industry as well as the nature of degradation it encounters in service. The importance of surface modification techniques in restoring the slurry pump impeller to as new was discussed. Thereafter, an evaluation of different techniques used for re-manufacturing mining equipment was carried out. Surface modification techniques considered included: (1) thermal spray, (2) arc welding, (3) laser cladding (LC) (4) laser-assisted cold spray (LACS) technologies and (5) polymer coating. After a careful evaluation, LC was adopted as the most efficient refurbishment technique for addressing the issue of equipment degradation in the mining industry. LC was considered most advantageous over other surface coating technologies in terms of low HAZ, low porosity, high bonding strength, high retaining of mechanical properties and thickness flexibility. Process and material parameters associated with LC technology were also discussed as well as the effects of laser-material interaction on the functionality of the clads. Major material

parameters associated with LC were found to be the cladding powders, substrate as well as their thermo-physical properties and chemical properties. It was established that the cladding process gives better results when the powder fed system and materials (both substrate and clad) with low laser reflectivity are used. In terms of processing, it could be seen that the laser wavelength, feed rate, scanning speed and laser power were highly influential on the quality characteristics of the coating. Furthermore, a short laser beam wavelength ($\sim 1.06 \mu\text{m}$) was found to be more efficient in comparison to a long wavelength ($\sim 10.6 \mu\text{m}$) due to its high absorption by material surfaces whereas the feed rate, scanning speed and laser power were found to be directly affecting the clad geometry, microstructure, microhardness, wear and corrosive resistance.

Chapter Three: Experimental approach

3.0 Chapter Summary: This chapter describes the approaches and methods that were used in this research towards achieving the set objectives. The project is an experimental design approach in which experimental equipment, methods, materials, process parameters engaged are hereby described. The sample preparation techniques and equipment are also elucidated together with various characterisation methods. This chapter further describes the Taguchi method of design of experiments (DoE) which was used in the optimisation and mathematical modelling of the cladding process for quality control purposes. The details on determining the estimated process parameter and the predicted response ranges are also described.

3.1 Materials

The experiment focused on metal alloy powder deposition on a substrate of 304L stainless steel which is used and applicable to the mining industry. The substrate is used in areas of high tribo-corrosion including the design of slurry pump impellers. Two metal alloy powders were explored to determine their processability and suitability as cladding materials for the identified substrate. Additionally, the two metal alloys were explored to determine which powder would provide improved resistance to wear and corrosion demands of the mining industry. The powders or cladding materials used were Stellite-6 (ST6) and Tribaloy T-800.

3.1.1 Substrate

Clads were deposited on an AISI 304L SS substrate. This is an austenitic stainless steel with good mechanical properties of low temperature ductility and toughness as well as good corrosion resistance arising from chromium content based on its chemical composition. The substrate is primarily iron-based in composition while it contains 20% chromium (Hsiao *et al.*, 2008). 304L SS also has good weldability and formability properties however, it has low yield strength in the annealed state (Baldenebro-Lopez *et al.*, 2015; Salleh *et al.*, 2011). The chemical composition of the substrate is as shown in Table 3.1.

Table 3.1: Chemical content of AISI 304L stainless steel (Joseph *et al.*, 2016).

AISI	Cr	Ni	C	Si	Mn	P	S
304L	18-20	8-12	<0.03	1	2	0.045	0.03

In technological application, 304L SS is used in different industries such as aerospace, medical, nuclear, oil and gas industries. In the mining industry it is used in manufacturing tailings slurry's pump impellers for handling waste from mineral processing plants (Bloch *et al.*, 2014; Salleh *et al.*, 2011). Due to its low fatigue strength and susceptibility to pitting corrosion, stainless steel is now clad with different metal alloys to improve its low wear resistance properties.

3.1.2 Feedstock

3.1.2.1 Stellite 6

In this research, Stellite 6 was studied for its cladding behaviour and applicability as a coating to address issues of wear. This metal alloy is cobalt-based and was supplied by Kennametal SA (Pty). The ST6 powder was analysed through an SEM which indicated that it had a particle size distribution of +45-90 μm and spherical morphology as shown in Figure 3.1.

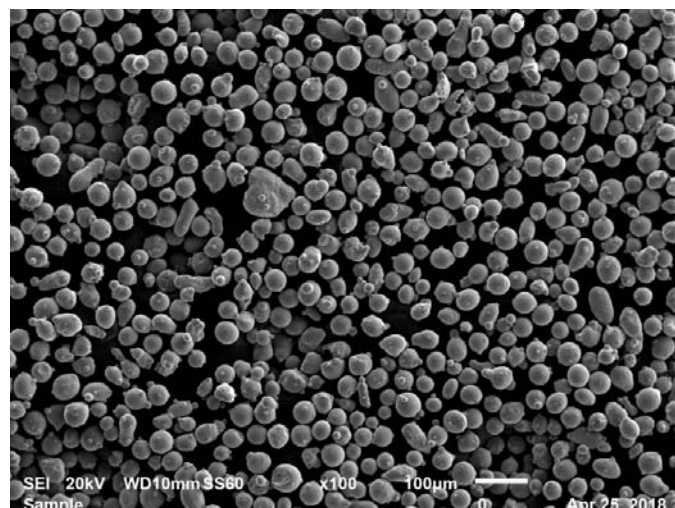


Figure 3.1: Micrograph of Stellite 6 powder showing the spherical morphology of its particles.

Table 3.2 shows the chemical composition of Stellite 6 powder as per EDS analysis. Tungsten content induces hardness in Stellite 6 clads which makes the metal much effective when addressing issues relating to wear (*Yu et al.*, 2009). Due to its chromium (Cr) content, Stellite 6 (ST6) has been used in areas of aerospace, marine, power generation, oil and gas industries as imparts high temperature oxidation- and corrosion-resistance (*Yu et al.*, 2009). With these properties, it is now important to study the metal alloy for further applications in the mining industry where there is high level of wear and corrosion. Stellite 6 is a high temperature alloy which can be processed at temperatures of 1285-1395 °C because of its Tungsten (W) content, a metal with the highest melting point (*Hasan et al.*, 2017; *Kusmoko*, 2016). The temperature range for melting ST6 is slightly different from the one for 304L (1399-1454°C) which means they can be processed together and also they both contain Cr which assists in bonding the two metals through the binder elements.

3.1.2.2 Tribaloy T-800

Tribaloy T800 powder used in this laser cladding research had **particle size distribution ranging from +45-90 µm** as supplied by Kennametal SA (Pty). These metal powder particles have a spherical morphology that allows for an improved flowability. A confirmatory analysis was carried to confirm the morphology using the SEM and the micrographs are as shown in Figure 3.2.

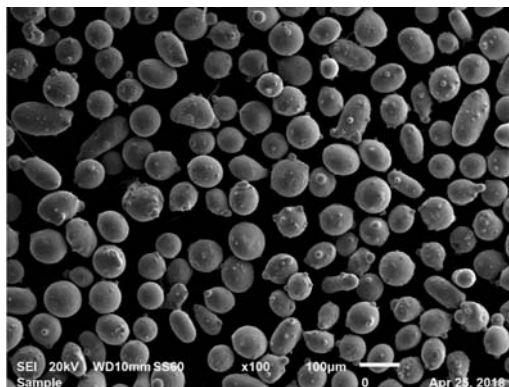


Figure 3.2: Spherical morphology on Tribaloy T800 powder particles.

Tribaloy T-800 is a cobalt-based alloy that is extensively used in thermal spraying deposition and belongs to the Co-Mo-Cr-Si known as the Tribaloy family. Molybdenum and silicon enhance good hardness and corrosion resistance properties which contribute much to its applicability in surface modification of aircraft engine parts subjected to wear and corrosion (Rulli, 2006). Therefore, it is of interest to investigate the alloy for applicability to the mining industry where severe levels of tribo-corrosion can be met. The melting range for T-800 is 1289-1352 °C which is also closer to that of 304 L stainless steel making the metal alloy compatible to be processed together with its substrate (Kusmoko, 2016). Also Ni and Cr will assist in binding the two metals since they are present in both metals. The second row of Table 3.2 presents the chemical composition of this alloy.

Table 3.2: chemical composition for coating metal powders from EDS analysis.

Materials	Co	C	Si	Cr	Ni	W	Fe	Mo
Stellite 6	61.2	1.2	2	28.6	-	4	3	-
Tribaloy T-800	46.7	0.8	3.5	18	1.5	-	1	28.5

3.2 Experimental equipment

Various equipment were used throughout this study. The details of the equipment used are presented in in this section while a summary of the equipment and software used in this research is presented in Appendix A.

3.2.1 Laser cladding system

Single track clads of ST6 and T800 were deposited with an Eskom IPG fiber laser system having maximum power of 4 kW and 1073 nm wavelength. The laser type and wavelength proves to be significant as other researchers have found that, solid-state lasers with short wavelengths of about 1.06 µm are more productive compared to long wavelength laser systems (Wang *et al.*, 2016). Furthermore, it had been established that laser absorptivity by the substrate is affected by laser wavelength as a short wavelength is more absorbable by the substrate compared to a laser with a greater wavelength (Wang *et al.*, 2016). Fibre lasers and CO₂ lasers have wavelengths of 1.06 µm and 10.6 µm respectively. In this regard, a fibre laser absorptivity is more than that of CO₂ laser, hence, more energy efficiency lies within fibre lasers. Which

is why in this study, fibre lasers were considered. The Council of Scientific and Industrial Research (CSIR) Eskom IPG fiber laser system used in this study consists of a 5-axis articulated robotic arm to which the laser cladding is attached. Articulated robots are very advantageous compared to other industrial robots like linear robots, cylindrical robots, parallel and spherical robots (Figure 3.4). In robotics and advanced manufacturing, an important aspect is degree of freedom (DOF) on a robot system. It allows the robotic arm to rotate about different axis hence giving it structural flexibility (Faravar, 2014). The DOF in a robotic arm is made from Cartesian coordinate system (X-Y-Z) and to increase that flexibility, the number of motors should be increased to different directions on the joints.

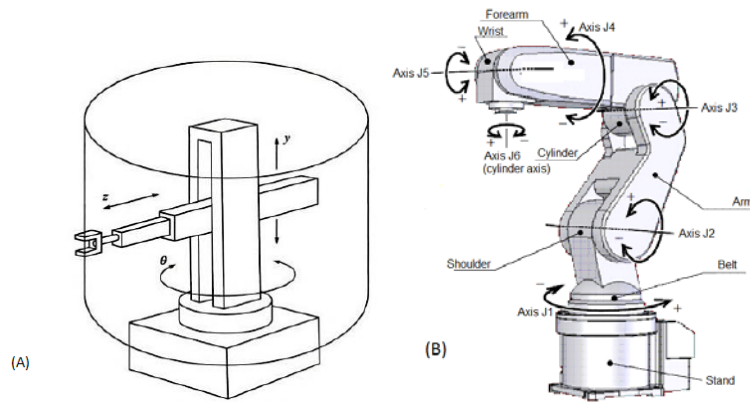


Figure 3.4: (A) Cylindrical robot, (B) 6-axis articulated robotic arm Cartesian coordinate system. Adopted from: (Faravar, 2014) and (<http://kyb.fei.tuke.sk/lab/en/modely/mit.php>) respectively.

A multi-hopper system was used in the laser system to supply material powders to the nozzle. Argon gas was used as both a carrier and shielding gas. The laser machine was operated from a distance using a remote controller by a qualified CSIR technician. A schematic diagram of the experimental set up is shown in Figure 3.4.

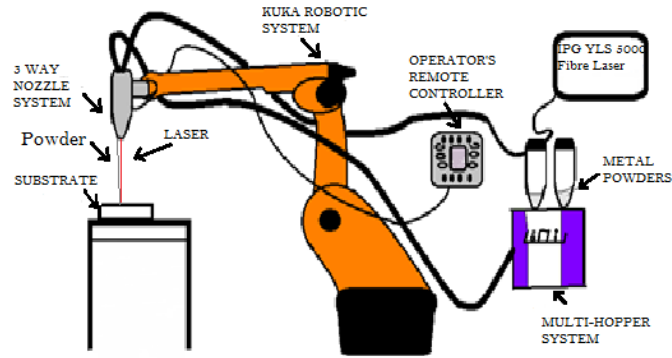


Figure 3.5: Laser cladding experimental set up.

3.3 Powder flow rate study

The Siemens GTV PF 2/2 multi-hopper system measures powder feed rates in rev/min, hence, it had to be qualified by measuring the mass of powder deposited at a given time (g/min) and gas flow rate. Figures 3.6 was obtained from the analysis of the powder flow rate data reported for each powder.

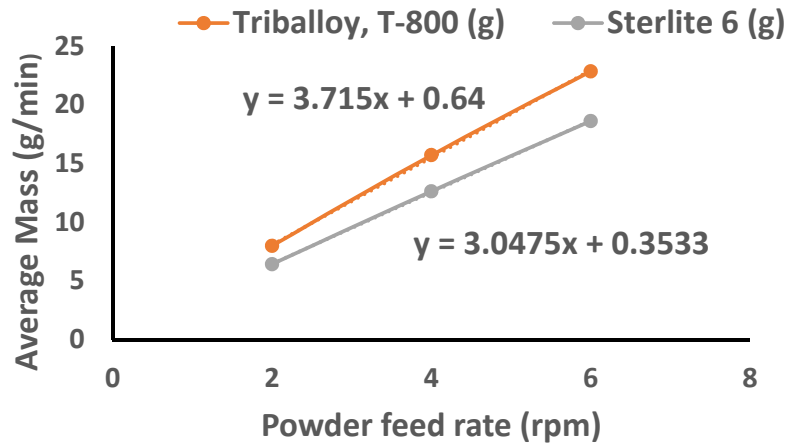


Figure 3.6: Flowability chart of both metal alloy powders.

Figures 3.6 shows that the mass deposited per unit time for each powder is directly proportional to the powder flow rate in revolution per minute (rpm). Straight line equation of the form ($y = mx + c$) obtained from the graphs were employed to calculate appropriate powder feed rate (rev/min) that correspond to the desired mass

of powder deposited in g/min. For instance, if the process requires a feed rate 10 g/min of Tribaloy T-800, the multi-hopper system will be adjusted to 2.5 rev/min via equation (3.1).

Calculations of feed rate from equations;

$$\text{Mass of Tribaloy per minute, } Y = 3.715X + 0.64 \quad (3.1)$$

Where **X**- Feed rate in rev/minute and **Y**- Feed rate in g/min

To determine 10 g/min in terms of rev/min, calculations are made as follows;

$$10 \text{ g/min} = 3.715X + 0.64$$

$$X = (10-0.64)/ 3.715$$

$$\underline{X = 2.5 \text{ rev/min}}$$

3.4 Deposition of clad samples

Preliminary experiments (first stage) of this study was aimed at determining the process maps for the two metal alloys with a view to understanding the adjustable process parameters and also to minimize the range to be adjusted so that time or consumables are preserved. In the second stage of this study, the alloy powder with improved processability relative to the other was taken further for processing by optimising its surface roughness and wear resistance as LC process parameters were varied. A summary of the experiments conducted is also shown under appendix B.

3.4.1 Laser cladding process maps of Stellite 6 and Tribaloy T-800 deposits

The powders were deposited as received with the parameters shown in Table 3.3. These values were varied based on an extensive LC literature search and the preliminary experiments. The clads were deposited on AISI 304L SS substrates that were machined to dimensions of 100 x 50 x 10 mm. Acetone was used to clean the substrate in order to remove any contamination before deposition. Single tracks with single layer were then deposited at varying parameters as already discussed (Appendix C and D).

Table 3.3: Cladding parameters for co-based alloys.

Metal powder	Laser power (Watts)	Scanning speed (mm/sec)	Stand-off distance (mm)	Powder feeding rate (g/min)	Spot size (mm)
Stellite 6/ Tribaloy (T-800)	1800	5-25	12	5-20	4

3.4.2 Optimisation of Tribaloy T800 clads using Taguchi Design of Experiments (DoE)

Laser cladding process was carried out as a follow up from previous preliminary experiments that were narrowed down to a certain range of input parameters. The cladding process was carried out using the Eskom IPG fiber laser system with maximum power of 4 kW and 1073 nm wavelength. The cladding system was operated with a 5 axis Kuka articulated robotic arm. The parameters were carried out through a L₉ orthogonal array as shown in Table 3.5. Three process parameters were varied as indicated (Table 3.4) and the laser power, carrier and shielding gas flow rates remained constant at 1800 kW, 2.0 l/min and 8.0 l/min respectively. The shielding gas stabilises the atmospheric pressure and inhibits oxidation of the samples as a result of the thermal induced process. Single track single layer clads were deposited and repeated once on a 304L SS substrate (Appendix I).

Table 3.4: Process variables for optimisation.

Process variables	Levels of parameters		
	1	2	3
Feed rate (g/min)	5	7.5	10
Scanning speed (mm/sec)	5	10	15
Spot diameter (mm)	2	3	4

Taguchi (DoE) assists in minimising the amount of trials for the experiment by only varying the variables considering the available combinations. The outcome is then matched with the process parameters. S/N ratios of lower is better are used based on the clad characteristics wear rate and coefficient of friction whereas "the larger the

better” was used in conjunction with microhardness. The application for S/N ratios are explained furthermore under section 3.7.

Table 3.5: L₉ Taguchi orthogonal array for Tribaloy T800 cladding process parameters.

Run	Feed rate (g/min)	Scan speed (m/min)	Spot Diameter (mm)
1	5	5	2
2	5	10	3
3	5	15	4
4	7.5	5	4
5	7.5	10	2
6	7.5	15	3
7	10	5	3
8	10	10	4
9	10	15	2

3.5 Sample preparations

Sample preparations were carried out to allow for analysis of microstructure, dilution, heat-affected zones (HAZ), clad geometry, cracks, porosity and microhardness tests. Metallographic sample preparation techniques consisted of the following steps:

3.5.1 Sample cutting

Samples were cut from clad plates using a Struers Labotom-15 metal cutting machine (Figure 3.7) so that they can be easily mounted. The metal cutting machine is installed with a water-cooling system that allows for samples to be cut with minimal heat generation zones, hence, avoiding defects on samples. The cutting disk that was used is the Struers 50A35 disk made from resin bonded aluminium oxide.

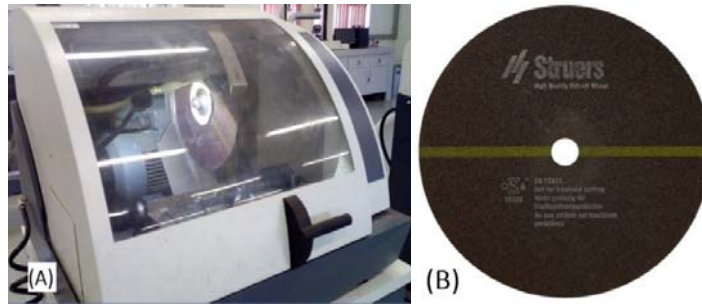


Figure 3.7. (a) Struers cutting machine and (b) Struers cutting disc used for sample preparation.

3.5.2 Mounting

Hot press mounting was carried out by using a polyfast bakelite with carbon filler which is electrically conductive. The conductive bakelite assists in absorbing excess charges from building up on the sample during SEM imaging and overheat it (Kinder, 2003). Struers CitoPress-5 mounting machine located at BIUST's Material Testing Laboratory (Figure 3.8) was used with the following parameter settings:

- Temperature- 180⁰C
- Pressure- 325 Bars
- Mounting time-3.5 minutes
- Cooling time-1.5 minutes



Figure 3.8. Struers Hot mounting press machine.

3.5.3 Grinding and polishing

A MRC M-1B grinding and polishing machine (Figure 3.9) was used to grind the mounted sample surfaces with water and silicon carbide (SiC) grinding paper of grit sizes P220, P320 and P500 (Table 3.6). Thereafter, the samples were polished to mirror finish with MD Largo disc, MD Dac disc and MD chem cloth used in that order with diamond suspensions of MD Largo 9 μ m, MD Dac 3 μ m and Akasel fumed silica 0.2 μ m alkaline respectively (Table 3.6). Grinding and polishing were done manually at speeds 150 and 200 rpm respectively.

Table 3.6: Grinding and polishing consumables.

Grinding and Polishing Consumables	Struers SiC grinding paper: P220, P320, P500
	Struers MD-Largo disc, MD-Dac disc, MD-chem polishing cloth
	Struers Water-based diamond suspension MD-Dac 3 μ m, MD-Largo 9 μ m, Akasel Fumed silica 0.2 μ m alkaline



Figure 3.9: Grinding and polishing machine at BIUST's Material Testing Laboratory.

3.5.4 Etching.

In order to reveal the microstructure of the clad samples, different etchants were used. Etching was carried out on the clads using different techniques and chemical reagents. Stellite 6 sample was etched by immersing it the samples for 5 seconds in a solution of 13 g FeCl_3 + 2ml Nitric acid (65%) + 200ml hydrochloric acid (32%). Electrolytic etching was carried out for Tribaloy T800 clads using a 1.5V Frederiksen Direct current (DC) power supply and stainless steel electrodes in a solution of 10 ml Nitric acid (65%) + 10 ml Hydrogen peroxide (50%) + 100 ml Oxalic acid (10%). The Tribaloy samples and the cathode were immersed in the solution without contact while the anode was used in contact with the metal samples for only 2 seconds. The equipment used for electro-etching is shown in Figure 3.10.



Figure 3.10: Apparatus for Electro-etching.

3.6 Sample Characterisation

3.6.1 Microstructural analysis

An optical microscope (OM) was at first used to view microstructure properties of cracks, inhomogeneity, discolourations, clad geometry, macro-pores, grain structures etc. Furthermore, a JSM-7100F Scanning electron microscope (SEM) (Figure 3.11) installed with an Energy Dispersive X-Ray Spectroscopy (EDS), was used to analyse the microstructure, chemical composition, micro-cracks, micro-pores, dendrite arm spacing and image mapping of microstructures. Additionally, the SEM was used to analyse the morphology of metal powders as well as measure the particle sizes.



Figure 3.11: Scanning Electron Microscope (SEM) at the Geology lab, BIUST.

When utilizing SEM, the electron beam hits the sample surface at diameters of 20-100 nm to focus intensively than using an optical microscope. The surface of the sample has to be electrically conductive, hence, the reflected electrons are used to draw an image of the specimen.

3.6.2 Vicker's Microhardness test

Microhardness test was conducted using an Innovatest 4000 series tester which utilizes a pyramid diamond indenter. The machine is installed with an optical microscope of 40X maximum magnification. The pyramid diamond indenter has an angle of 136° between opposite faces. The Vicker's test was done at a force of 500 gf and a dwell time of 10 seconds was maintained. The position of the indentations within the clad can be varied according to the interest. In this research, the indentations were lined diagonally across the clad to the substrate as indicated in Figure 3.12.

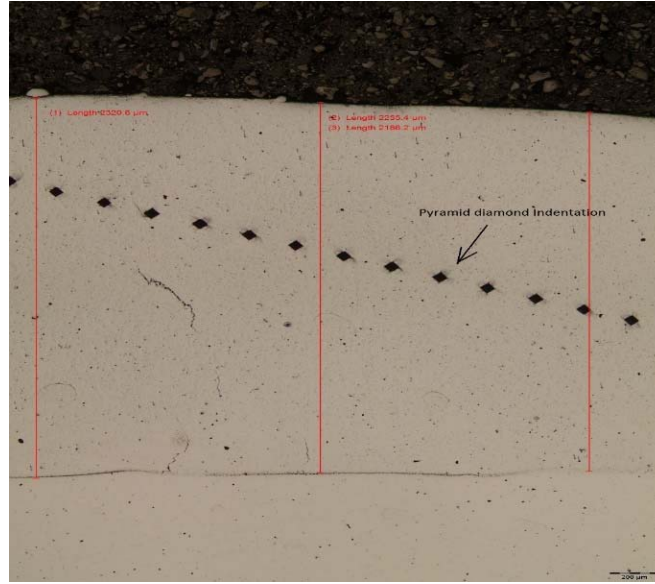


Figure 3.12: Vickers test indentations on a coating.

When conducting the test, force was applied and then removed to measure the average diagonal lengths. A formula was used to calculate the response in terms of HV as follows:

$$HV = 2000L \sin(a/2)/d^2 = 1854.4L/d^2 \quad (3.2)$$

Where;

L is test force in gf

d is the average diagonal length in μm

a is the face angle (136°)

3.6.3 Wear test

The test carried out was a reciprocating ball on disc test using an Rtec tribometer (Figure 3.15) installed with computer based Rtec MFT17 software. The wear testing equipment has a maximum normal load of 150 N. Samples were grinded and polished along the transverse direction prior to the test as shown in Figure 3.13. Room temperature and dry conditions were considered for the test approach which was

conducted using a Silicon Nitride spherical indenter of 6.35 mm in diameter. Normal load of 120 N was applied at a reciprocating sliding distance of 2 mm and a sliding speed of 3 mm/seconds. The set up ran for 10 minutes on each sample.

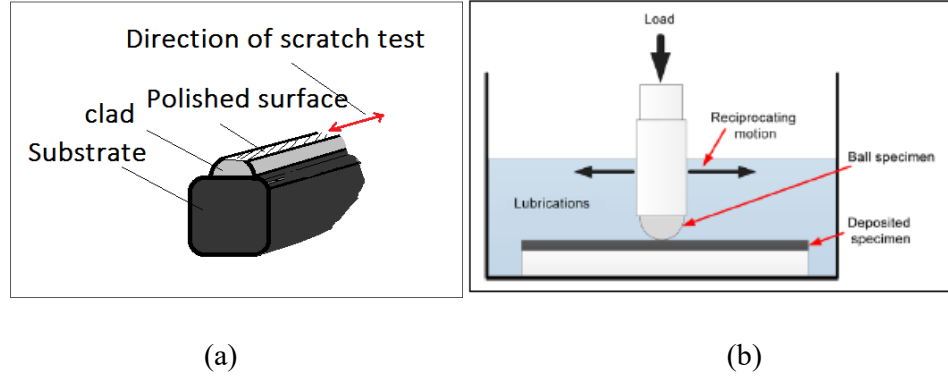


Figure 3.13. (a) Schematic of sample preparation and (b) reciprocating ball on disc set up (Source: Bajwa *et. al.*, 2016).

The tribometer locates the position of the indenter and measures its movement along the x and z direction. Also the normal force applied in terms of these directions was given as F_x and F_y . Coefficient of friction (COF) was also determined as the test outputs at 15 second intervals on a Microsoft excel spreadsheet. The method of wear analysis used, follows the ASTM G99 standard to calculate the wear volume similar to Shebani *et al.*, (2015) using equations 3.3 and 3.4.

$$V_W = \frac{a\pi h^2}{3b} [3R - h] \quad (\text{mm}^3) \quad (3.3)$$

$$h = R - \sqrt{R^2 - b^2} \quad (\mu\text{m}) \quad (3.4)$$

Where V_w is the wear volume, R is the radius of the pin, a and b are the wear track half length and width respectively. Measurements were conducted as shown on Figure 3.14.

Archard's model formula was then used to obtain the wear rate using equation 3.5:

$$W_R = \frac{V_W}{N \times l_s} \quad (3.5)$$

Where W_R is the specific wear rate, N and l_s being the normal load applied by the tester and the reciprocating sliding distance respectively.

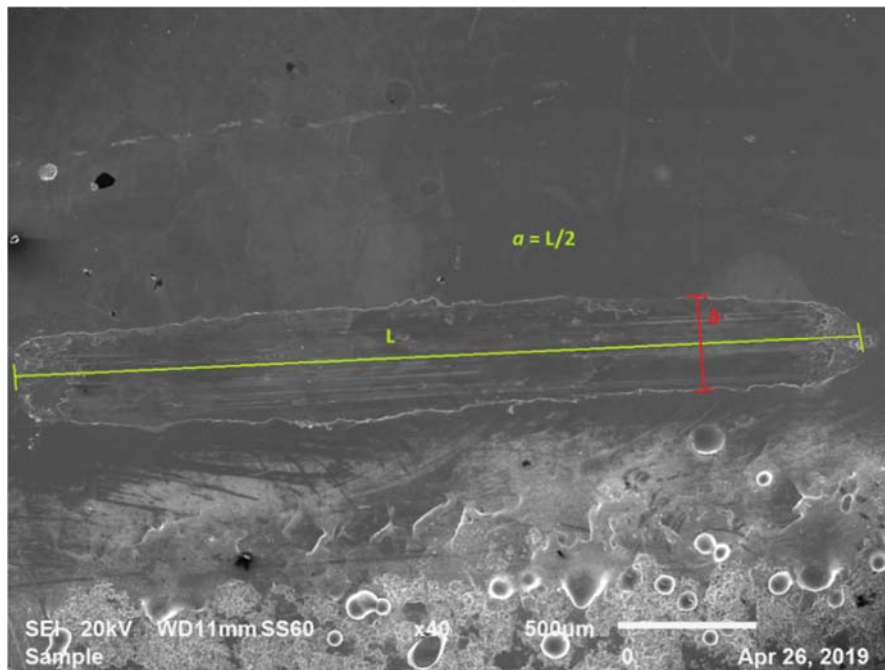


Figure 3.14. Measurements as conducted on wear scars for analysis.



Figure 3.15. Ball-on-disc scratch test (BIUST).

3.6.3 X-ray diffraction (XRD)

X-ray diffraction technique (Figure 3.16) was used for phase identification of both starting powders and the clads so as to identify the phases therein present in various samples. Findings from the XRD analysis were linked to the response of the powders under varying processing conditions with a view to understanding their wear resistance performance. A Bruker D8 advanced powder diffractometer installed with a Cu tube X-ray and a LynxEye XE energy-dispersive strip detector was utilised for pattern identification. An X-ray radiation at a wavelength of 1.54056 Å was used as well as 2θ at a range of 20-110 degrees. The XRD pattern of the starting powders (Figure 3.17) confirms the presence of compounds with the same chemical composition found by EDS analysis (Table 3.2). Figure 3.17 reveals that T800 metal powder comprised of $\text{Co}_3\text{Mo}_2\text{Si}$, CoMoSi , Co_7Mo_6 , Co (Cr) and Co phases which constitutes the elemental composition from EDS. Stellite 6 was also found to have Co, CrO, $(\text{Cr, Fe})_7\text{C}_3$ and W_2C phases. In T800 coatings, $\text{Co}_3\text{Mo}_2\text{Si}$ is responsible for the formation laves structures which provide the required properties of the alloy such as wear and corrosion resistance. Similarly, ST6 coatings are made from dendritic

structures that require the Cr content for their formation and this is achieved by the $(\text{Cr, Fe})_7\text{C}_3$ which provides the corrosion and hardness properties.



Figure 3.16. X-ray diffraction unit.

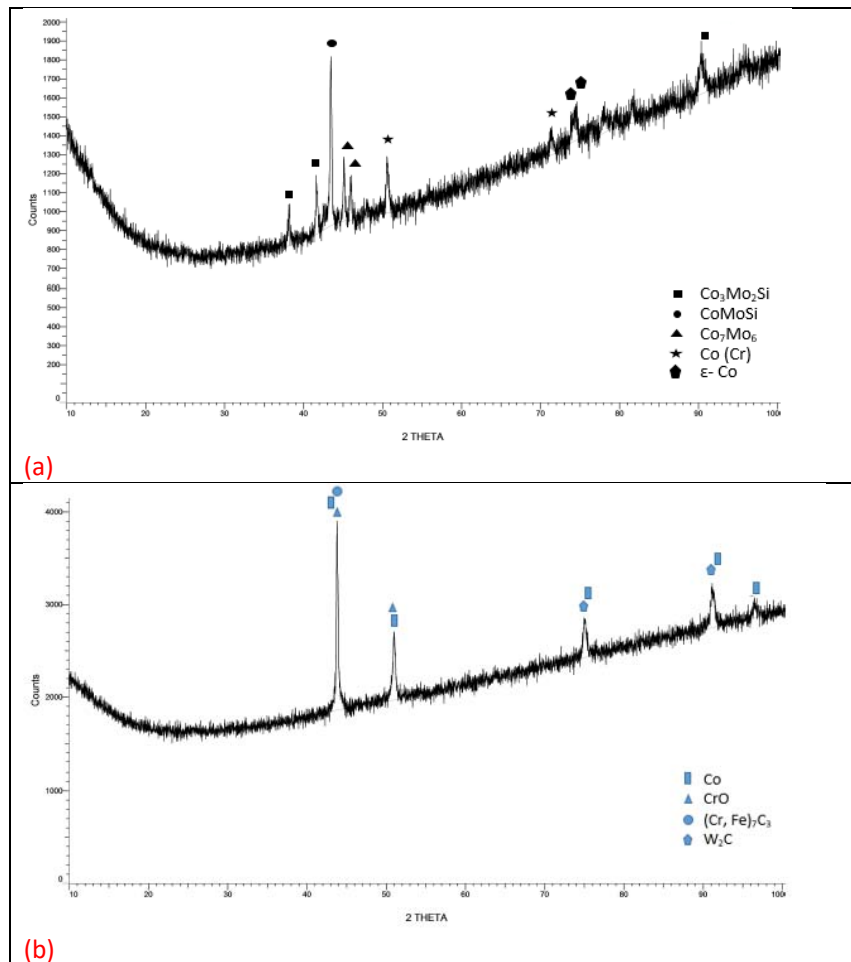


Figure 3.17. X-ray diffraction pattern for (a) T800 and (b) ST6.

3.7 Process optimisation and modelling

Since in this experiment parameters were varied, Taguchi design of experiment technique was used from the Minitab 18 software in order to optimise the process parameter to obtain the desired clad characteristics. The characteristics were conducted with ‘the *smaller-the-better*’ for COF and wear rate while ‘the *larger-the-better*’ was considered for microhardness. The DoE optimisation tool based on the required output follows equations 3.6 and 3.7.

Larger the better- when a response variable is to be maximised.

$$S/N_{l-b} = -10 \log_{10} \sum \left[\frac{1}{x_i^2} \right] \quad (3.6)$$

Smaller the better- when a response variable is to be minimised.

$$S/N_{s-b} = -10 \log_{10} \sum [x_i^2] \quad (3.7)$$

Where X_i is the measured response and n is the number of repetition of the experiment at the same parameters. The ANOVA statistical analysis was then carried out in order to understand the significance of the impact of the individual process variables on the final clad characteristics. F values for the ANOVA were obtained by calculating the standard deviation of the factors within and between factors. The standard deviation is used to calculate the adjusted residual error and sum of squares for the. Mean squares were then calculated by dividing the sum of squares by the degree of freedom (Df). The Df is the count of factors minus one. The F-value was determined by Factor mean square divide by residual error mean square. The residual error mean square is the level of noise and an F-ratio more than one means the factors have an effect more than the level of noise. Since it cannot be understood the error of the noise or at what level above the noise the effects could be considered practical, the p value is then used. The p value is obtained from a DF versus F test table. It gives statistical value which can be determined if it falls within the probability range of the noise or not. If the p value is less than 5% it is then not in the range of the noise and the effects are considered to be practical.

ANOVA also assisted in providing mathematical model equations to optimise the fabrication of T-800 clads using LC. Multiple linear regression model was used in

providing the mathematical equations to be used in optimising the required clad output and matching it with the laser cladding control factors. The mathematical models were obtained from a table of ANOVA and regression coefficients using the formula:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_1 + \dots + \beta_k X_k + \varepsilon \quad (3.8)$$

Where Y is the response variable, ε is a random error and β_k is the population regression coefficients.

In concurrence with Mathur, (2012), estimation of optimum parameters was carried out to determine the final recommended coating. This was achieved by using the Minitab 18 Taguchi optimisation tool which utilises the confidence level (α), F-ratio at the confidence level at $1-\alpha$ ($F_{\alpha 1, f_e}$), degree of freedom (Df) and error of variance (V_e) to calculate the confidence interval for the confirmatory experiments and population of the characteristics with equation 3.9, 3.10 and 3.12.

$$CI_{CE} = \sqrt{F_{\alpha 1, f_e} V_e \left[\frac{1}{n_{eff}} + \frac{1}{R} \right]} \quad (3.9)$$

$$CI_{POP} = \sqrt{\frac{F_{\alpha 1, f_e} V_e}{n_{eff}}} \quad (3.10)$$

$$n_{eff} = \frac{N}{1 + \text{Total degree of freedom associated with estimate of optimum values}} \quad (3.11)$$

$$E_R = F_O + S_O + D_O \quad (3.12)$$

Where E_R is the estimated response, F_O is the response at the optimum feed rate, S_O is the response at optimum scan speed and D_O is the response at optimum diameter. These were used to find the optimum ranges of the characteristics through equations below:

$$\text{Response (COF, WR or H)} - CI_{CE} < E_R < \text{optimal response} + CI_{CE} \quad (3.13)$$

$$\text{Response (COF, WR or H)} - CI_{POP} < E_R < \text{optimal response} + CI_{POP} \quad (3.14)$$

The optimum characteristics was then obtained and the confirmatory sample was tested to observe if the responses fall within the response range.

3.8 Conclusion

This chapter begins by introducing the feedstock and the substrate involved in the cladding process. It explains their thermo-physical properties, chemical properties and their suitability to the cladding process. In addition, the cladding parameters for the first experiment involving the two powders (ST6 and T800) were discussed followed by a second experiment which involves optimisation of T800 clads. Furthermore, the clad characterisation techniques and equipment are then explained together with the optimisation technique Taguchi design of experiment (DoE). Minitab 18 software was used to provide S/N ratios by considering “smaller-the-better” option for the coefficient of friction and specific wear rate while “larger-the-better” was used for microhardness. The ANOVA tests were also carried to determine the significance of each factor against the factors of feed rate, scan speed and laser spot diameter. Mathematical models for optimising the factors and responses were obtained via multi linear regression analysis from the software. Lastly, the estimation of optimum parameters was also carried out for the Tribaloy clads to find the optimum range of the characteristics. These optimum ranges were used to observe if the confirmatory optimum sample will have characteristics within the range.

Chapter Four: Effects of laser cladding parameters on the processing maps of co-based alloys

4.0 Chapter summary: This chapter studies the consolidation behaviour of two metal alloys of Stellite 6 and Tribaloy T-800. These metal alloys were deposited (Appendix C and D). Process maps for the alloys were obtained at the end of the study together with microstructural evolution across the clad. Furthermore, a comparison was made based on the microstructure, chemical composition and the microhardness.

4.1. Process maps of laser-cladded Tribaloy T800

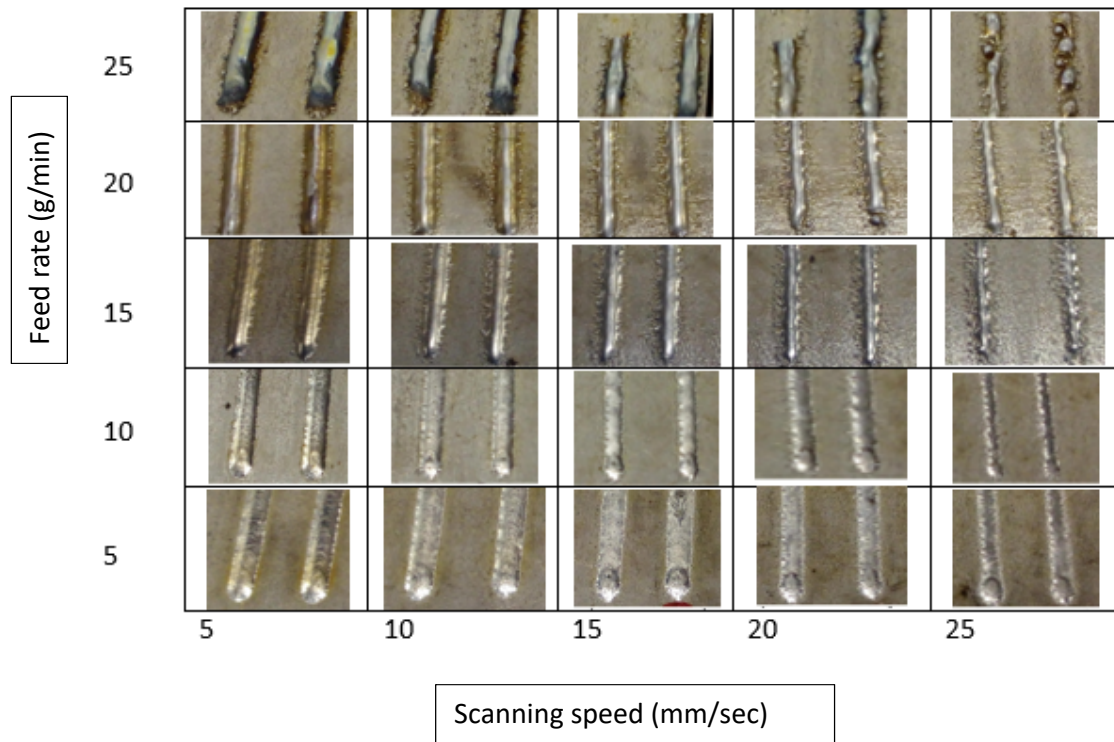
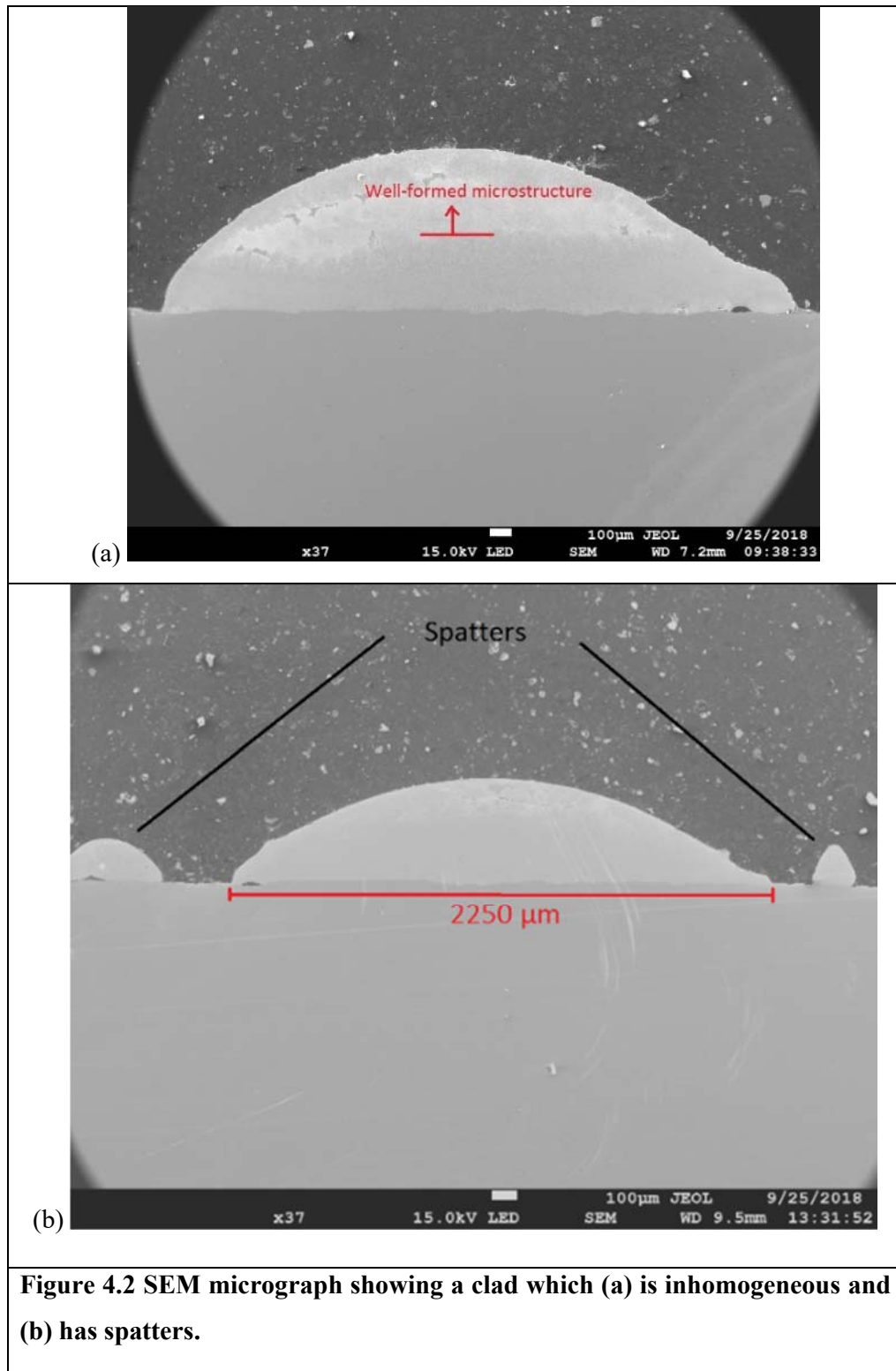


Figure 4.1. As-deposited single tracks of Tribaloy T800 clads developed with varying feed rate and scan speed.

In this section, the consolidation behaviour of Tribaloy (T-800) clads deposited at varying feed rates and scanning speeds is discussed. Figure 4.1 shows the as-deposited images of single track clads of Tribaloy (T-800). A summary of the data

set collected for T800 is also shown in Appendix E. The images were used for constructing the process map for the powder using nodal arrays (experimental points). Scanning speeds were varied at the interval of 5 mm/s from 5-50 mm/s while feed rate varied at 5 g/min rate from 5 - 25 g/min. These process maps were categorised into regions depending on their track geometry, clad bead formation (continuous or discontinuous) and colouration. Clad track geometry is also described as either wide or narrow, thick or thin based on measured values of the width and the height of the surface coating, respectively. A clad track is considered to be wide when its width is equal to or above 2500 μm and narrow when its width is less than 2500 μm . This limit is not associated with spattering as shown in Figure 4.2. The track height is considered thick when it is equal or more than 200 μm and thin when it is less than 200 μm . The threshold values of clad width and height were selected based on the need to promote adequate re-melting during inter-track and inter-layer deposition of the clads by laser beam without the formation of inter-layer or inter-track porosity or other microstructural defects which degrade the quality characteristics of the clads.



The bead formation in this study was also classified as either continuous or discontinuous. Based on the work of Hurtado (2016), discontinuity in this research takes into account macroscopic pores and cracks as well as any other non-uniformity that can be seen within the clads such as lack of inter-particulate bonding and breakage in track. Colours were also part of visible characteristics that can be used to qualify the surface clads. In this piece of work, the base colour for Tribaloy when there is no oxidation during LC is described as being silvery. Varying colours and intensities were also seen in the fabricated surface clads, therefore these were taken as being caused by oxidation. Pangsrivini (2016) studied the oxidation behaviour of laser clad Ti-6Al-4V clads, and found out that the intensity of discolouration was an indication of contamination, the low colour intensity of silver and light grey for the two metals (Tribaloy and Stellite 6 respectively) was also considered as the base colours for non-contaminated clads.

4.1.1 Regions across the process map of Tribaloy (T-800)

After a careful inspection of the T-800 deposits, five regions of consolidation behaviour on the process map of Tribaloy (T-800) were found and presented in Figure 4.3 as (1) Wide, thick, continuous and silvery clads, (2) Narrow, thin, continuous and silvery clads, (3) Narrow, thick, discontinuous and discolored clads, (4) Narrow, thick, discontinuous and/or discolored clads and (5) Wide, thick, discontinuous and/or discolored clads.

4.1.1.1: Region 1 (Wide, thick, continuous and shiny grey clad)

Region 1 was found to exist when feed rate was varied between 5-15 g/min and the laser beam was scanned within the range of 5-15 mm/s. These parameters resulted in clads that were wider ($> 2500 \mu\text{m}$) and thicker ($> 200 \mu\text{m}$) in comparison to the other 4 regions. Lower scanning speed induces high energy density inputs which led to the concentration of high laser heat energy in a single spot for a longer dwell time. Therefore, the degree of powder consolidation in this region was very high as there was also more melting and clads were thicker and wider. This is due to the negative thermocapillary gradient within the melt pool which allows outward flow of

Marangoni convection from the center to the edge of the melt pool. Studies show that the occurrence of a temperature gradient between the centre and edge of the melt pool results in variation of the surface tension between the centre and the edges _of the melt pool during LC. Consequently, this initiates Marangoni thermocapillary forces for fluid flow which influences the flow of melt pool from a region of increasing temperature (low surface tension) to a region of low temperature (high surface tension).

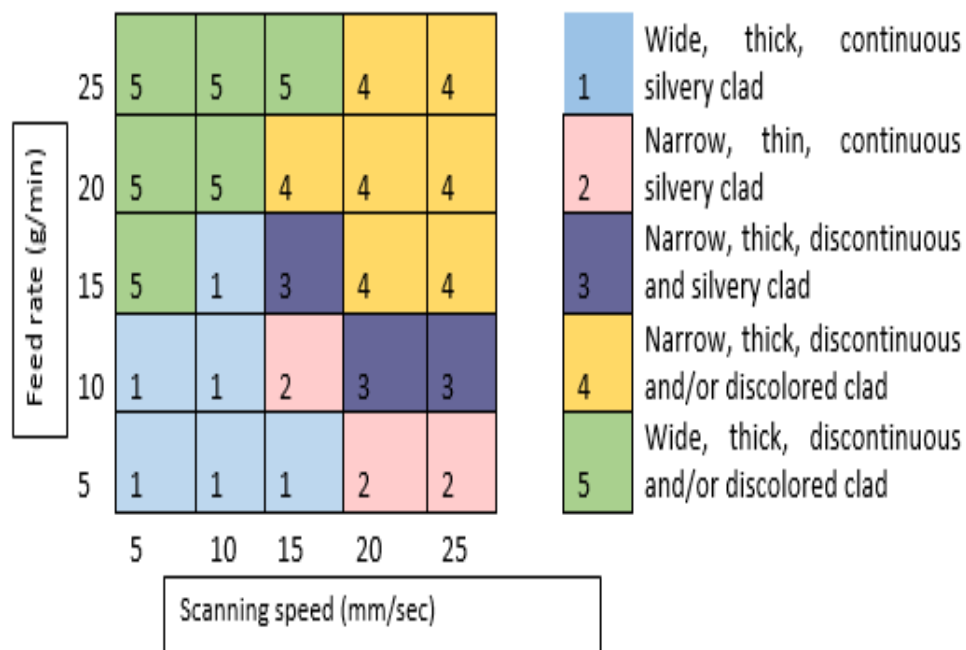


Figure 4.3. Process map for Tribaloy T800.

The radial outward flow of the melt pool results in the formation of wider and thicker track (Pangsrivinij, 2016). Also, when there is more energy density there will be excess melting, the flow of the melt pool will in turn result in widening of tracks upon solidification(de La Batut *et al.*, 2017). Similar findings reported by Gedda (2004) showed that clads were very wide and thick at lower scan speeds of 1.7 mm/s to 15 mm/s in an observation of melt-solid interactions in laser cladding and of pre-placed Stellite 21 powders. The clad in this region is also continuous, a clear evidence of sufficient melting occurring due to high energy density thus allowing for inter-

particulate bonding as well as inter-layer and inter-track re-melting. The silvery colouration of the clads, in region 1, is the base colour for Tribaloy T800 clads. This region proves that the laser-material-shielding gas flow interaction is sufficient to avoid contamination/ oxidation of the clads.

4.1.1.2: Region 2 (Narrow, thin, continuous and silvery clad)

Region 2 is as a result of feed rates that were varied between 5-10 g/min while the laser beam scan speed was varied at a speed range of 20 to 25 mm/s. The clads from these set of parameters were found to be narrow in width (2372 to 2400 μm) and thin in thickness (127 to 144 μm). Low powder feed rates at higher laser scanning speeds have proven to be responsible for thin clads. Similar findings by Barekat *et al.* (2016) on laser cladding of Co–Cr–Mo alloy deposited on γ -TiAl substrate established that clad height depends on feed rate and that thin clads were achieved when feed rates were lower and increased with an increasing in feed rate resulting in thicker clads. The laser beam in region 2 was scanned at high speeds of (≥ 20 mm/s). This did not permit for a longer laser-material interaction time to induce sufficient melting as compared to region 1. In terms of temperature gradient between the center and the edge of the melt pool, the Marangoni thermocapillary forces for fluid flow initiates the flow of melt pool from a region of lower temperature (high surface tension) to a region of high temperature (low surface tension). This results in the formation of clads with reduced width and thickness (Drezet *et al.*, 2004). Although, there was no excess melting, the laser energy density was enough to support proper fusion of powder particles. This is explained by the uniformity/continuity of the clad. No discolouration was observed in region 2 clads which meant that the shielding gas was enough to avoid discolourations or oxidation.

4.1.1.3: Region 3 (Narrow, thick, discontinuous and silvery clad)

The region of narrow, thin, discontinuous and no discolouration clad occurred when employing a scanning speed variation between 15 and 25 mm/s and material feed rate of 10-15 g/min. The width of the clads was found to be lower than 2250 μm . Heat input is sufficient to impart thicknesses above 200 μm . Clads in this region were

found to be discontinuous. The form of discontinuity in the clads is due to lack of sufficient inter-particulate bonding at clad boundaries. Upon observing the surface of the clad tracks, it is seen that they are well melted and fused at the top center rather than at the clad side boundaries as shown on Figure 4.4.

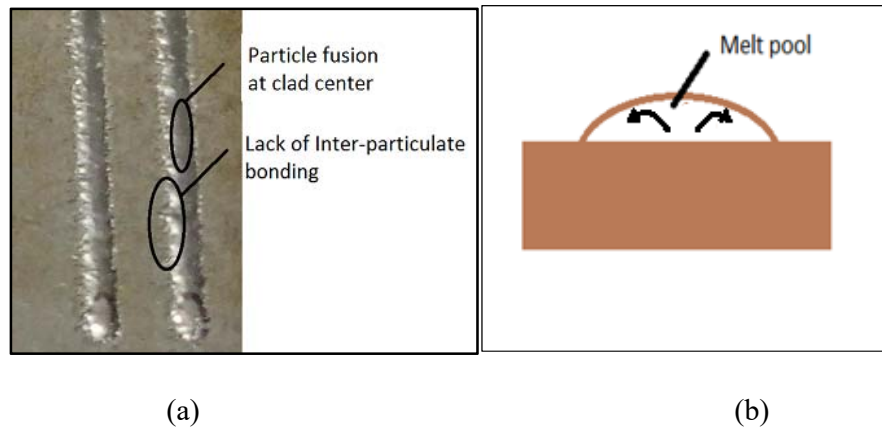


Figure 4.4. (a) Image showing track discontinuity from region 3 and (b) Marangoni convection current on clad melt pool.

Drezet *et al.*, (2004) modelled Marangoni convection in laser heat treatment and found that the Marangoni convection currents are expected to flow outwards from the center to the edge of the melt pool based on high temperature variations occurring within the melt pool with low variations in liquid tensions. Nonetheless, a limited amount of melt pool was formed in the clads of these regions and was located at the top center of the clad, hence, had low thermocapillary activity. This occurred due to a high feed rate which reached boundaries that are outside the laser spot diameter and would not allow for the generation of sufficient melt pool. In this case, the high surface tension is located at the center of the melt pool thereby attracting any low tension liquid to flow to the center and a greater height is built, hence, thick clads. Region 3 clads showed no discolouration which may be understood as a sign of no contamination.

4.1.1.4: Region 4 (Narrow, thick, discontinuous and/or discolored clad)

Region 4 was found to exist when the laser beam speed varied from 15 to 25 mm/s and the feed rate was varied between 15 and 25 g/min. The region is classified by clads that have a track height range of 398-953 μm falling within the category of “thick”. The clads were also categorised as narrow as the width ranged from 1853-2446 μm . As more feed is introduced into the system, upon interaction with the laser beam, more material is melted resulting in a height build-up of the tracks. Even though a greater thickness was achieved by feeding more material into the system, the laser-material interaction did not permit for sufficient melting of the powders and also because of the high scan speed (≥ 10 mm/s). The Marangoni effect in this case could have been interrupted because of lack of sufficient melt pool because the generation of the molten metal is located at the top center of the clad where the laser beam can only reach resulting in capillary instability. This effect will result in the variations in liquid tensions as well as temperature gradients to induce molten flow towards the center of the pool where there is high surface tension and greater clad heights being achieved. An increase of feed into the laser-material system also means insufficient melting as limited energy density heats a larger amount of powder thus resulting in discontinuous clads. Discontinuity varied from lack of inter-particulate bonding of powders on clad boundaries to balling of tracks at feed rates varying from 20 to 25 g/min. Region 4 clad tracks also show a sign of contamination as there were discolourations. The intensity of discolouration increases from brown to dark grey from a feed of 20 g/min to 25 g/min respectively which is a clear evidence of reduction of shielding gas efficiency due to increasing material feed.

4.1.1.5: Region 5 (Wide, thick, discontinuous and/or discolored clad)

The region of wide, thick, discontinuous or discolored clad was found when feed rates were varied at high values of 15-25 g/min in conjunction with a laser beam scanning at lower speeds of 5-15 mm/s. A lower scanning speed allows for an increased dwell time of the laser beam on a single spot and therefore resulting in development of melt pool which will flow and will cool to form wider clads. The width of region 5 clads ranges from 2610- 3235 μm which means they are the second

widest after region 1 clads. The clads are also thick from a range of 525-1458 μm . Due to more feed introduced by the system and also the powders being allowed to interact more with the laser beam, there is an increased track height. The laser-material interaction supports the formation of discontinuous tracks as there is too much feed more than the energy density could melt. The clads easily peel off demonstrating that the feed shields the energy density from heating the substrate. Lack of heat reaching the substrate results in weak or no clad-substrate bonding which is shown by clads that easily come off in region 5. This also means there was very low thermocapillary activity occurring at the lowest part of the coating mainly because interruptions occurred due to lack of melt pool or a viscous liquid that cannot be effected by weak Marangoni forces hence low heat flow. Less viscosity was achieved at the top of the clad within the reach of the laser beam and where more material flow from the Marangoni forces was achieved to give thick clads. The clads are also dark grey showing a high intensity of discolouration and contamination. The shielding gas is less efficient for region 5 feed rate.

4.1.2 Microstructural evolution across the regions on the process map of laser-cladded Tribaloy T800 clads

The clad micrographs in Figure 4.5 show the microstructural variations distinctively indicating that there is a significant effect achieved from varying the two variables of powder feed rate and scanning speed. These variations of process parameters have shown some level of importance in the microstructural evolution of these surface clads and the co-relation needs to be understood. Therefore, the SEM micrographs were taken at (1) 50 μm below the clad surface, (2) half-clad height and (3) the fusion zone. These microstructures were now linked with the process map regions they are associated with as shown by Figure 4.5 (a-o). It can be seen from the micrographs that, the clads are characterized by either a eutectic matrix of cobalt solid solution, cellular crystallization, eutectic laves phase, primary laves phase or fine dendritic structure (Figure 4.6). The mechanical properties of Tribaloy metal alloys are dependent on a higher % volume fraction of laves phase as they are said to provide much resistance to abrasive wear and corrosion (Nsoesie *et al.*, 2013). To retain the

properties required, it is better to focus on regions with a greater % vol of primary laves phase.

In region 1, the near surface microstructure (Figure 4.5a.) is characterized by a mixture of fine and large dendritic structure immersed in a cobalt solid solution. This microstructure differs from that of the clad middle (Figure 4.5f.) consisting mainly of coarse dendrites and eutectic laves phase. Above the fusion zone (Figure 4.5k) the structure is made up of a cellular dendritic structure. Cellular structures are achieved by a high thermal gradient and low solidification rate (Navas, Cadenas, et al., 2006). At the clad-substrate boundary, there is high temperature gradient (G) as the base metal acts as a heat absorber and there is also a low solidification rate (V) resulting on formation of cellular crystallisation. Above the cellular zone it is found out that the structure is made up of coarse dendrites and eutectic laves, this is due to a very low solidification rate and a reduced thermal gradient at the middle of the clad. This shows that the formed liquid stayed long to allow for growth of dendrites and more evidence can be seen by existence of porosity at the center of the clad. These micrographs are a result of low scanning speed and low feed rate or in short, high energy density. At the near surface, the size of dendrites was reduced and also consisting of many of finer dendritic structures. The surface provides free face for energy dissipation and as result, there is a low G/V ratio and dendrites become more finer (Awasthi *et al.*, 2010).

The second region is formed from low feed rates (5-10 g/min) and higher scanning speeds (20-25 mm/sec). The region is characterized by primary and eutectic laves phases at the top of the clad (Figure 4.5b) while the clad middle (Figure 4.5g) is made of fine dendritic structures with small amounts of primary laves. This may be due to the thermal convection flows within the solid-liquid interface that caused more heat to be conveyed from the center to the surface. The effect of which is a higher solidification rate at the center while the near surface regions are continuously heated to temperatures above eutectic thus having fine dendrites and primary laves respectively. The cellular crystallization zone also exists at the clad-substrate

boundary (Figure 4.5i) but it does not cover a greater depth or height as compared to that of the first region. This is due to low laser energy density input which has reduced the temperature gradient at the base.

When the powder feed rate is slightly increased to a range of 10-15 g/min at high scanning speed of (15-25 mm/sec), a third region is formed. The near surface microstructure (Figure 4.5c) is then made up of large columnar dendrites and eutectic laves phase. The clad middle (Figure 4.5h) consists of primary and eutectic laves while above the fusion line (Figure 4.5m) only fine dendrites are observed. The increased amount of feed rate seems to have shielded some of the energy from reaching the base therefore a low thermal gradient was formed which was easily dissipated into the substrate hence no formation of cellular crystals but only fine dendritic structures. The effect is evidenced by the formation of pores at the clad-substrate boundary meaning that there is less fusion. At a higher level from the boundary towards the center, the heat input seems to have reached eutectic temperature causing formations of primary and eutectic laves phases. Solidification rate at this point was lower compared to the boundary section and allowed for formation of laves. The area near to the surface is composed of large columnar dendrites indicating most of the energy shielded was saturated at this point and forming a liquid phase which existed for a longer period hence formation of larger dendrites (Zimmermann *et al.*, 2009).

Region 4 was made from high feed rates above 15 g/min and high laser scanning speed above 15 mm/sec which resulted in thin columnar and fine dendritic structures at the near surface zone (Figure 4.5d). The middle of the clad (Figure 4.5i) has primary and eutectic laves phase while at the boundary (Figure 4.5n) the clad is made of fine dendrites. Similar as in region 3 above, there is less heat reaching the base of the clad hence low temperature gradient with an increased solidification rate due to heat sinking into the substrate, hence, formation of fine dendrites and boundary porosity. Also the primary laves were formed at the middle of the clad. The near surface region has thin columnar and fine dendrites formed from a low laser-material

interaction because the scanning speed was high to allow for formation of liquid phase that could give time for large dendrite formation (Zimmermann *et al.*, 2009).

From a high feed rate above 15 g/min at lower scanning speed rate less than 15 mm/sec, region 5 was formed. The microstructure near to the top (Figure 4.5e) of the clad consisted of large columnar dendrites whereas the middle of the clad (Figure 4.5j) and the close to the boundary there exists fine dendrites.

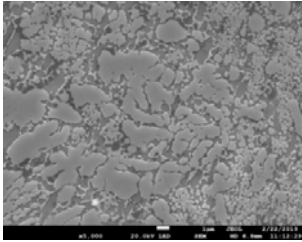
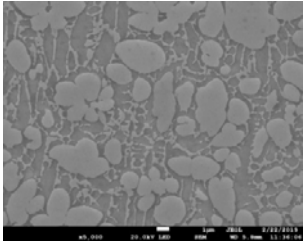
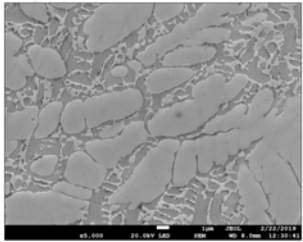
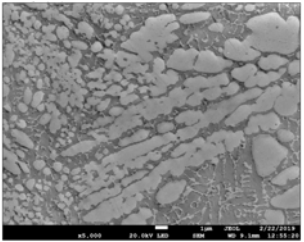
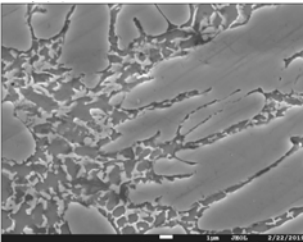
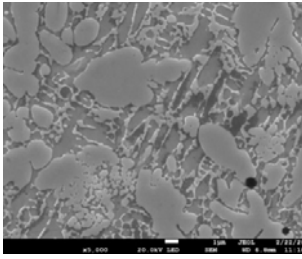
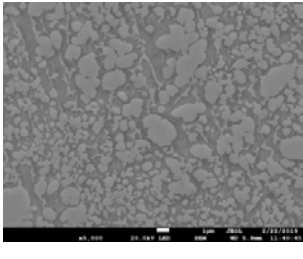
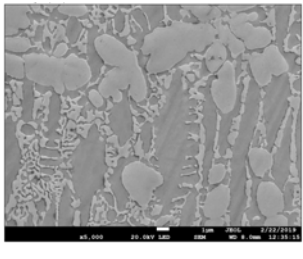
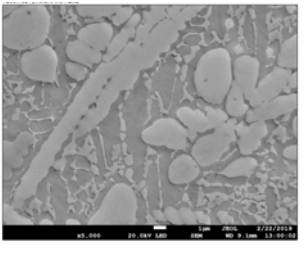
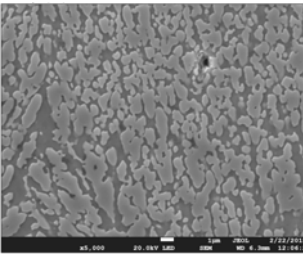
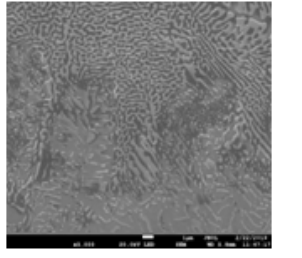
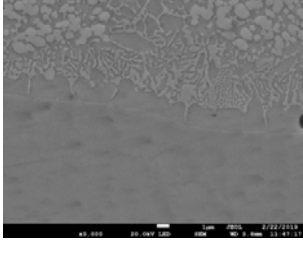
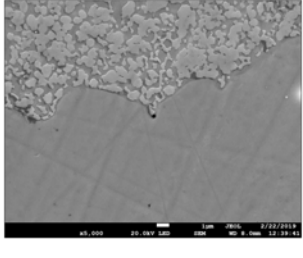
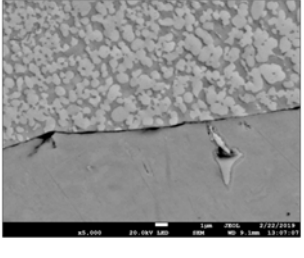
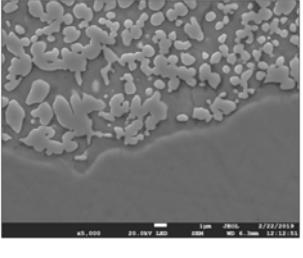
Region	1	2	3	4	5
Near surface	a. 	b. 	c. 	d. 	e. 
Track centre zone	f. 	g. 	h. 	i. 	j. 
Fusion zone	k. 	l. 	m. 	n. 	o. 

Figure 4.5. FE-SEM micrographs showing Tribaloy T800 clad cross-sections, microstructures at the clad center and at the clad-substrate boundary.

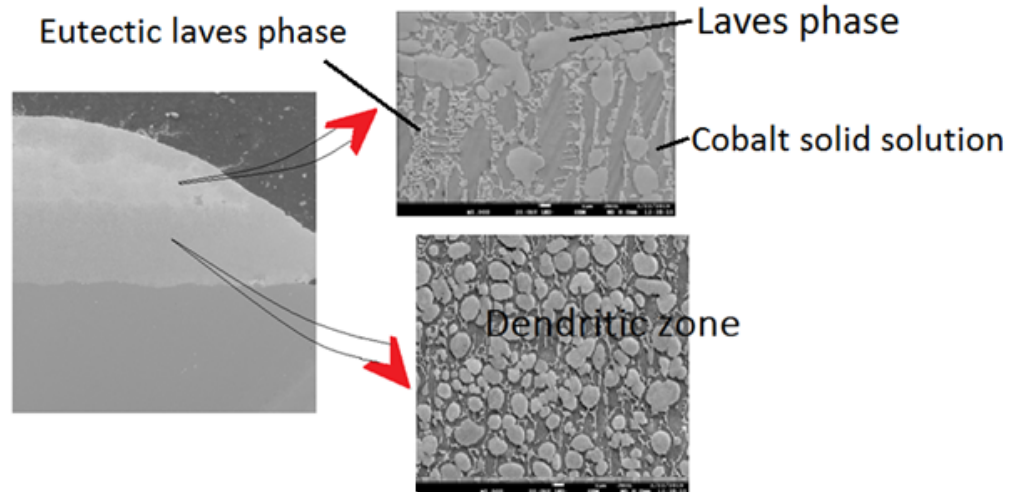


Figure 4.6 Laves zone and dendritic zone within a Tribaloy T800 coating.

Slower scanning rates with high feed rate allowed the top of the clad to form a liquid phase which was prolonged enough to allow formation of large dendrites. Also existence of Marangoni convection forces have kept heat distributed to the top surface. However, the center and the boundary experienced a low temperature gradient from the shielded energy input because of high material injection. This caused formation of fine dendrites and porosity.

Generally, it is not possible to get homogeneity on the clad structures and all regions differ in terms of the microstructure. Region 1 and 2 fusion zones have similar structures of cellular crystallization even though that of region 1 is larger whereas regions 3, 4 and 5 have a dendritic structure instead. The microstructure of the middle of the clads for regions 1, 3 and 4 have primary laves except for region 5 which has a fine dendritic structure while for region 2 it is a mixture of both. The near surface microstructure for region 3, 4 and 5 comprises of columnar dendrites while region 2 has primary and eutectic laves. Region 1 at near surface has a mixture of large and fine dendrites. Since a cellular structure seems ideal for proper fusion, homogeneity is required for the near surface and middle microstructure which shows to be better achieved in region 2.

4.1.3 Chemical analyses across the regions on the process map of laser-cladded Tribaloy T800 clads

Chemical composition analysis (Figures 4.7 and Table 4.1) of the laves indicates high contents of molybdenum (Mo) and cobalt (Co), with low contents of chromium (Cr) and silicon (Si). These laves are immersed in a solution containing high levels of Co while Cr, Mo and Si are lower. The eutectic laves phase is also seen to be formed from high level content of Mo not so much different from the primary laves composition but of the laves are lower in Co content compared to their matrix binder.

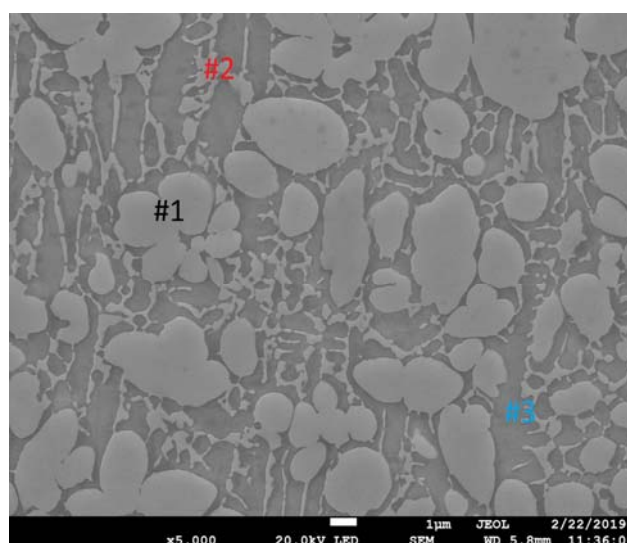


Figure 4.7. Micrograph of Tribaloy T800 showing locations of chemical analysis.

Table 4.1. chemical weight percentage analysis by EDS.

Weight %					
Elements	C	Si	Cr	Co	Mo
Primary Laves (#1)	5.01	4.66	12.88	38.92	38.53
Eutectic laves (#2)	4.34	3.21	16.82	41.43	34.2
Co-Matrix (#3)	3.98	1.79	21.98	56.56	15.68

Table 4.2: Chemical composition by EDS of laser-cladded T-800 clads as a function of the regions on the processing map.

Region	Location	Si (wt%)	Cr (wt%)	Fe (wt%)	Co (wt%)	Ni (wt%)	Mo (wt%)
1	Surface	3.13	16.68	4.75	41.88	-	26.96
	Centre	3.12	17.12	4.45	42.94	-	25.81
	Fusion	2.42	17.02	16.87	32.69	3.16	20.82
2	Surface	3.14	16.91	-	44.8	-	26.44
	Centre	3.16	16.62	-	45.28	-	26.07
	Fusion	2.87	16.24	5.95	42.03	-	25.35
3	Surface	3.45	16.86	-	45.57	-	29.68
	Centre	2.75	18.85	-	49.93	-	24.3
	Fusion	3.33	17.63	-	47.31	-	27.62
4	Surface	3.58	16.91	-	45.44	-	28.91
	Centre	3.00	18.47	-	48.34	-	25.42
	Fusion	3.23	16.33	-	44.77	-	29.01
5	Surface	3.32	17.21	-	44.91	-	26.39
	Centre	3.23	16.45	-	45.70	-	28.12
	Fusion	3.24	16.96	-	44.79	-	26.62

The first region clads are composed of high cobalt (Co) content as expected because of being fabricated from a cobalt based alloy while other main constituents are molybdenum (Mo) and chromium (Cr) (Table 4.2). There is increased content of Mo at the surface relative to the middle and fusion zones of the tracks while increased content Co and Cr is observed at the middle of the clad. This means the middle of the clad is dominated by Co solution matrix while the surface has more Mo for the formation of laves phases and dendrites. However, it is observed that there is Iron (Fe) content in most of parts of the clad with highest concentrations found at the fusion zone. This could be due to diffusion of elements when the melt pool was formed. The Marangoni convection currents acted from the bottom of the clad and dissipating energy to the surface and in the process some of the Fe content was transferred to other location within the clad. Studies show that there is a correlation between laser energy input to fluid flow velocity and dilution depth due to hydrodynamic effects imposed by Marangoni forces, thermocapillary or surface

tensions (Morville *et al.*, 2012). High energy density generates a melt pool with high fluid velocity and also more depth of dilution hence transfer of elements during fluid flow. More Iron diffused from the base metal but as for Nickel it is only found close to the fusion zone. It is established that iron diffuses more readily in comparison to nickel (MacEwan *et al.*, 2006).

As the energy density is reduced (feed rate of 5-10 g/min and scanning speed of 20-25 mm/s) in the second region of the process map, the cobalt composition remains the highest constituent at the clad center and similarly Mo also has high concentrations close to the clad surface (Table 4.2). This is an indication that the Co matrix also continues to dominate the middle of the clad compared to other regions and the formation of dendritic structures and laves dominates the parts that are closer to the surface. Distinctively, the Fe content is not found at the top and middle of the clad. However, a smaller amount of Fe is found close to the base metal. This is an indication the heat input was not high enough to melt the substrate excessively and introduce too much elemental diffusion. This is also evidenced by lack of nickel on any part of the clad. Region 3 of the process map is a result of a powder feed of 10-15 g/min and scanning speed of 15-25 mm/s. Clads indicate a high concentration of Co solution matrix at the clad center than at the surface. The high Cr content at the middle of the clad can be explained to be due to formation of eutectic laves phase. The molybdenum content is higher at portions near the clad surface but lower at the middle of the clad because of the formation of columnar dendrites. There is no Fe nor Ni content in the clad because the feed rate was more hence not allowing the heat input to induce sufficient melting of the substrate and no diffusion of elements occurred. Instead the Co content is more than the amount found close to the surface.

As more feed was introduced at a high scanning speed for region 4, the Marangoni effects were forced to act where there is temperature gradients which is at the center hence less heat reached the base and no melting of the substrate. Therefore, no diffusion of Fe is seen in the clad composition rather, it seen that the Mo content is more close to the fusion this may be due to formation of more dendrites. The clad

center continues to be dominated by the cobalt matrix. No indication of elemental diffusion was found in any part of the clad. Feed rates above 15 g/min at slow scan speeds in region 5 of the process map resulted on high Cr content in areas close to the surface while more Co and Mo is found at the central part of the clads. This clad is dominated by columnar dendrites at the surface which means they are high in Cr content whereas the middle of clads is characterised by fine dendrites which means they are dominated by the surrounding Co solution. In summary, the first region in the process map has shown possibilities of the formation of intermetallics which degrades the quality characteristics of the clads. The second has a little of intermetallics at the fusion zone which can be allowable for proper bonding **while other regions 3,4 and 5 have very little amounts of elemental diffusion which indicates insufficient bonding.** The Mo and Si contents are seen to be more in Primary Laves phases as seen in Figure 4.7a. Laves phases are more important in providing abrasive resistance to clads (Przybylowicz & Kusinski, 2002). Another important aspect is homogeneity, regions 1,3,4 and 5 show too much variations in Mo content which is an important element for formations of laves phase. A lower variation is indicated by region 2 which means it is more homogenous compared to other regions on the process map.

4.2 Process maps of laser-cladded Stellite 6 (ST6) coating

Stellite 6 (ST6) clads were fabricated by varying process parameters, and the images were plotted as shown in Figure 4.9. The clad tracks were characterised by different colours, geometry, cracks and porosity. These features were also used in process maps of the ST6 clads following similar criteria that were used herein for producing the process maps for T-800 clads. A summary of the data set collected for ST6 is shown in Appendix F. Stellite 6 clads base colour was considered as light grey when there is no oxidation. Various regions on the process map of 6 are described as seen in Figure 4.9. The process maps were categorised as (1) Wide, thick, continuous and light grey track, (2) Narrow, thick, continuous and light grey track, (3) Wide, thick, continuous and Brown track, (4) Narrow, thick, discontinuous and/or discolored track and (5) Wide, thick and/or discontinuous and discolored.

4.2.1 Regions across the process maps of laser-cladded Stellite 6 (ST6) coating

4.2.1.1 Region 1 (Wide, thick, continuous and light grey track)

The region of wide, thick, continuous and coating with no discolourations appears on the process maps at lowest feed rates and scanning speeds. Region 1 for Stellite 6 clads is characterised by of continuous and wider tracks (2655-2894 μm). The region only appears at a feed rate of 5 g/min and scan speed of 5-10 mm/s. The scanning speed allows for a longer laser-material interaction thus generating an excessive melt pool. Due to difference in the thermal gradient between the centre of the melt pool and the edge of the pool, surface tensions were generated in which they attract the low tension fluid to flow towards the surface resulting in outward fluid flow (Morville *et al.*, 2012). Outward Fluid flow is the main cause for formation of wide clad tracks.

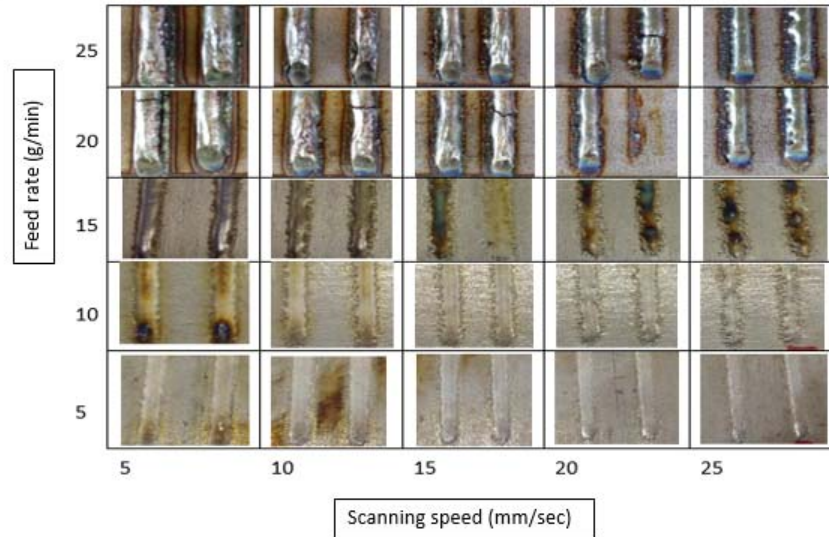


Figure 4.8. As-deposited single tracks of Stellite 6 clads developed with varying feed rate and scan speed.

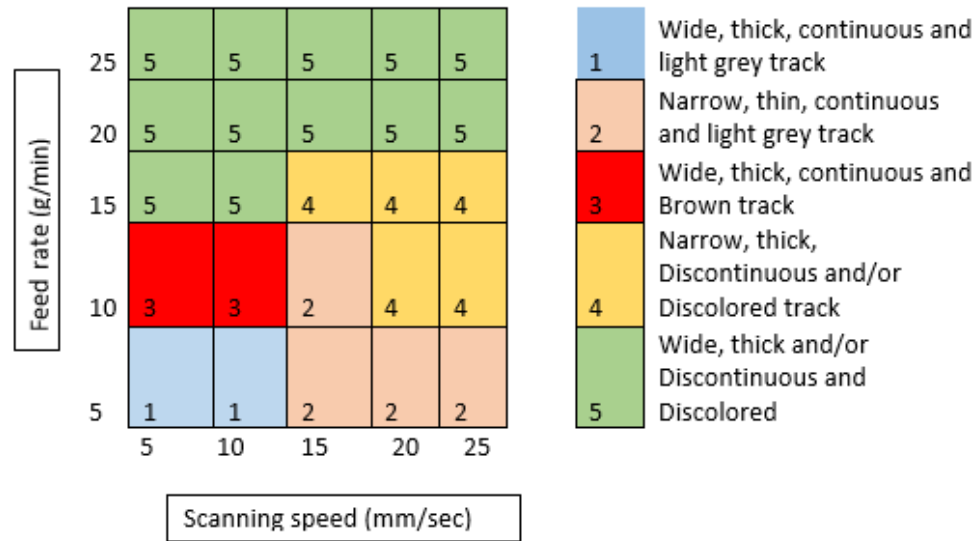


Figure 4.9: Process map for Stellite 6.

Stellite 6 clad tracks in this region are rougher in texture in comparison to those of Tribaloy in the same region. Clad continuity occurs due to the agglomeration of inter-particle bonding during melting and solidification. In agreement with Gu *et al.*, (2015), high energy densities were found to improve the wetting conditions of the metal powders which in turn increases the densification of the microstructures. This explains the existence of continuous tracks in region 1. The clads attained a uniform light grey colour which is the base colour for ST 6 clads and also indicating that there is no oxidation within the process.

4.2.1.2: Region 2 (Narrow, thin, continuous and light grey track)

Clad tracks in this region are narrower in width ($< 2500 \mu\text{m}$) and thinner in height ($< 200 \mu\text{m}$) compared to those in region 1, but they are continuous in similarity to those in region 1. The region is formed by adopting low feed rates of 5-10 g/min and high scanning speeds (15-25 mm/s). The narrowness of these clad arose from laser beam interacting with the material at high speed and not allowing for excessively long residence time between powder and laser beam nor dissipation of heat that can allow for excessive melt pool flow. The melt pool that was formed existed at a high

viscosity for which thermocapillary forces could not exceed in order to induce fluid flow (Limmaneevichitr & Kou, 2000). The energy density only allows sufficient melting to support the interlocking of powder particles and form continuous clads. The clads in this region do not show any indication of oxidation as there is no discolouration.

4.2.1.3: Region 3 (Wide, thick, continuous and brown track)

As the feed rate increases to 10 g/min, at scanning speed of 5 to 10 mm/s, the clads become thicker than the regions 1 and 2. This is due to more material being fed into the system and low laser beam speed allowing for this material to be fully melted which upon solidification resulted in increased clad heights. The slow speed of the laser beam also allows formation of wider and continuous tracks from excess melt pool. The hydrodynamics in the melt pool are due to generation of Marangoni convection currents as the temperature gradient from within the melt pool and its edge is steep. If the temperature gradient is greater than the change in surface tension, the fluid flow will be directed outwards from the center of the pool to its edge, thus a wider pool is formed (Kou *et al.*, 2011). However, the difference in this region to region 1 is that the clads are brownish. This may be as a result of the shielding gas at this feed no longer being sufficient in preventing oxidation or this may be due to excessive temperature input. In an inspection of TIG welds by Maske (2015), similar effects were found on EMK 6 Bohler welded on AISI 1020. The weldments were discolored at high feeds indicating that oxidation has occurred, the effects of which were attributed to excessive temperature input.

4.2.1.4: Region 4 (Narrow, thick, discontinuous and/or discolored track)

The fourth region was formed from high speeds of 15-25 mm/s and a feed rate of 10-15 g/min resulting in narrow clads (1620-1952 μm) which are also thick. Furthermore, the clads are discontinuous due to a mismatch of the cladding system delivering more feed at high scan speeds which subsequently results in inappropriate laser-material interactions. Having more feed and less time for laser-material interaction means no ample time for sufficient melting thus, less liquid phase and less

inter-particulate bonding. The melt pool may be formed at high viscosity which had internal stresses greater than the weak thermocapillary stresses formed from a lower thermal gradient and lower surface tension gradients (Limmaneevichitr & Kou, 2000). Therefore there was no fluid flow that occurred within the melt pool. Due to the low energy density, the heat is not easily dissipated across the melt pool thus there is poor clad-substrate bonding as some of the clads could be seen peeling off. The Stellite 6 clads in this region were discolored especially at a feed rate of 15 g/min.

4.2.1.5: Region 5 (Wide, thick and discontinuous and/or discolored)

Region 5 clads were achieved by feed rates ranging from 15 to 25 g/min and laser beam speed of 5 to 25 mm/s. Clads in this region similar to those of region 4, were not adequately bonded as some were peeling off during sample preparations. The feed rate >15 g/min does not permit for heat to reach the base metal to provide enough bonding. The tracks are characterised by very thick clads due to excessive feed input that piles up without good consolidation. Large cracks are also visible on some of the claddings. Therefore, this region is composed of different defects that make it extraneous for future applications. The intensity of discolouration is higher showing dark silvery clad tracks meaning that the intensity of oxidation has increased compared to previous regions.

4.2.2 Microstructural evolution of laser-cladded Stellite 6 coating

Process maps of ST6 clads are further studied in relation to the microstructural consolidation behaviour in order to obtain an overview of how cladded ST6 clads are built. The Stellite 6 clads are formed from a columnar or equiaxed dendritic structure surrounded by secondary dendrite arm spacings as shown in Figure 4.10. Similar criteria that was used in T-800 clads was also adopted for ST6 in which the micrograph imaging was analysed at near clad surface, clad center and at the fusion zone.

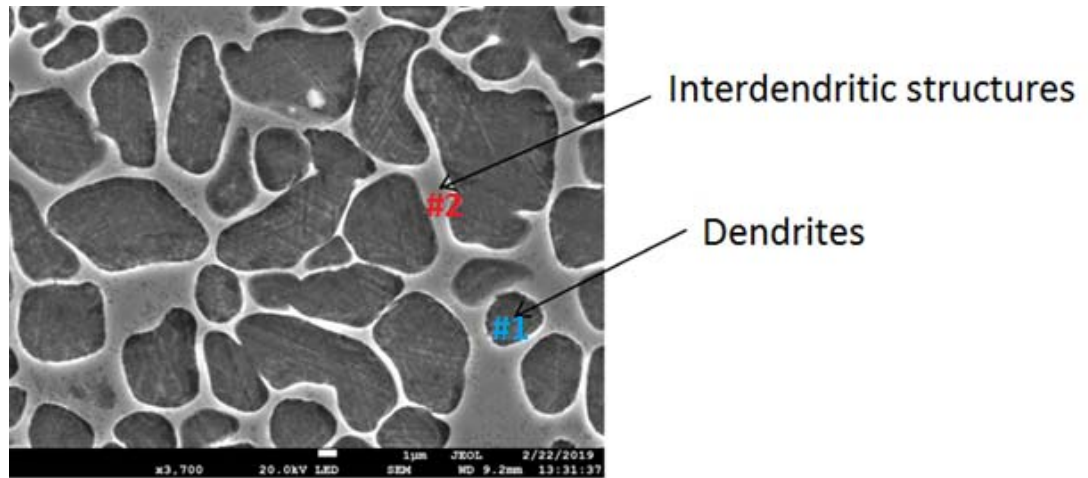


Figure 4.10. Microstructure of Stellite 6 clads consisting of the dendritic structure and dendrite arm spacing.

It can be seen in region 1 that the microstructure is characterized by fine columnar dendrites at areas close to the surface (Figure 4.11a.). However, the inter-dendritic structures or the eutectic appears to be not refined and fibrous with some portion still at separation stages. This is due to a high cooling rate which occurred at the surface not permitting for the refined formation of the inter-dendritic structures. It can be seen that as cooling rate becomes less at the clad middle, the inter-dendritic or eutectic structures improved in refinement and size (Figure 4.11f).

Table 4.3: Chemical weight percentage analysis by EDS.

Weight (%) Element	C	Cr	Fe	Co	W
Inter-dendritic structure (#1)	5.46	24.84	2.77	61.86	5.06
Dendrite (#2)	6	43.95	2.2	41.18	6.67

The dendrites formed close to the surface (Figure 4.11a.) and at center (Figure 4.11f) are aligned perpendicular to the substrate. Moreover, the dendrites became coarser

when approaching the fusion zone due to high thermal gradient and lower cooling rates. Similar findings established that areas within the cast sample with slow cooling rates were characterized by coarse dendrites compared to finer dendrites in fast cooling rates (Sun *et al.*, 2018). The second region of the process map consisted of fine columnar dendrites that are randomly orientated near the surface (Figure 4.11b.). This may be due to directional solidification of the melt pool being interrupted by Marangoni convection forces. At the center of the clad (Figure 4.11g.), the dendrites are still fine but they are equiaxed. Also, the eutectic structures increase in width due to a reduced solidification rate which allows for proper refinement and growth of the structures. Close to the fusion zone (Figure 4.11i.) the equiaxed dendrites were reduced in size as well as the eutectic structure due to increased solidification rate. Similar findings were reported in (Kaiser *et al.*, 2011).

Region 3 resulted in a mixture of fine dendrites and very long columnar dendrites (Figure 4.11c.) near the surface. The inter-dendritic structures are very thin in appearance due to rapid solidification from energy dissipation to the surface. The clad center consists of irregular shaped dendrites which seem to have solidified during a transition from fine dendrites to coarse dendrites. This can be seen by inter-dendrites that are disconnected and give space for dendrite growth (Figure 4.11h). As for the fusion zone, the microstructure is not refined as there is more feed than the laser energy can handle therefore the thermal gradient does not permit for proper dendrite nor cellular formation. This is attributed to pore formations that can be seen at the fusion line. Region 4 of the Stellite 6 process maps resulted in very fine columnar dendrites with very thin inter-dendrites (Figure 4.11d.) near the surface. This indicates that the cooling rate was very rapid. The clad middle is characterised by dendrites that were solidified during a transition from fine to coarseness. This can be explained through thickening of inter-dendritic threads in order to accommodate expansion of dendrites (Figure 4.11i) while other portions were becoming thin. The fusion zone is dominated by large pores and lack of fusion. The feed rate shielded most of the laser energy from reaching the substrate to allow for proper clad-substrate bonding.

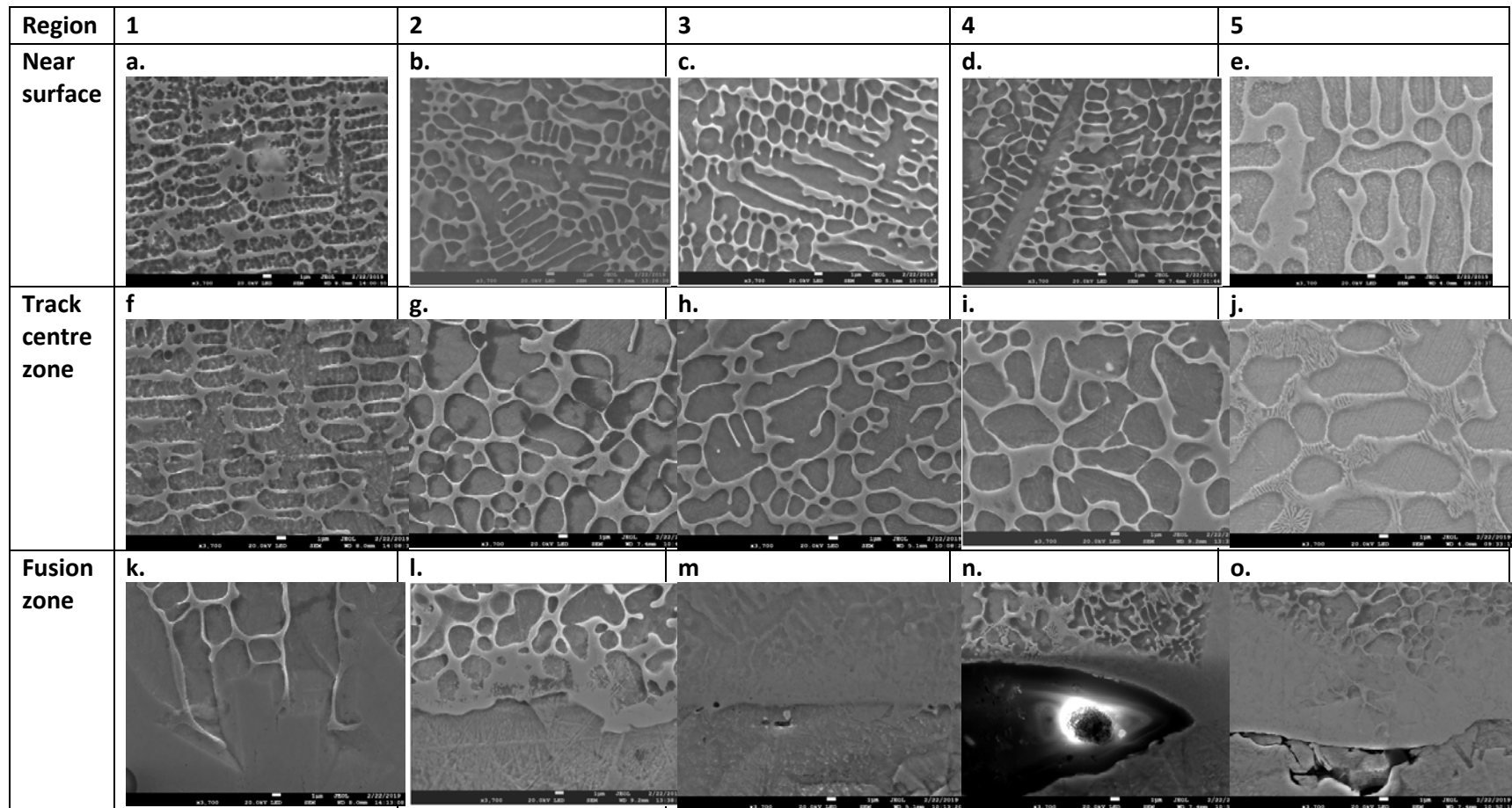


Figure 4.11. FE-SEM micrographs showing Stellite 6 clad cross-sections, microstructures at the clad center and at the clad-substrate boundary.

Region 5 is characterised with large columnar dendrites at near surface and the effects are similar to other process maps as the solidification process is slow. The center of the clad has a mixture of large and fine rounded dendrites. The energy input has reached the center allowing for formation and growth of some of the dendrites. The eutectic shows some formation of lamellar structures from within. The fusion zone is characterized by pores and weak bonding. This is the reason of the clads were peeling off during sample preparations. Comparing the microstructure across the process map regions, it can be seen that all regions were characterized by fine columnar dendrites at the surface excluding region 5 which has larger dendrites. The fine dendrites in region 1 also are not as refined as compared to those in other regions. As for the microstructure of the clad center, it can be seen that as the laser-energy density from region 1 to 5 reduces, the dendrites are transitioning from unrefined fine columnar (region 1) to refined fine equiaxed (regions 2,3 & 4) and in the end they become large equiaxed dendrites region 5). The fusion zones across all regions are characterized by equiaxed dendrites but the porosity on the fusion line starts to appear in region 3 and in the last 2 regions (regions 4 and 5). Fine and refined dendrites have been found to be important in providing an improved mechanical properties such as hardness and corrosion resistance (Ruan *et al.*, 2015). Region 1 and 2 have shown to have fine dendrites across all parts of the clad however, the ones in region 1 are not as refined as those in region 2.

4.2.3 Chemical analyses across the regions on the process map of laser-cladded stellite 6 (ST-6) clads

Figure 4.10 and Table 4.3 confirm that ST6 clads are characterised by Cr-rich dendrites (dark region) in random orientation and Co-rich inter-dendritic structures (grey) along the grain boundary. Other alloying contents within both structures were C, Fe and W. Similar findings established that Stellite 6 clads were enriched by the same constituents (Gupta *et al.*, 2010). Chemical analysis of different locations on the clad samples and across the regions of the process maps is presented in Table 4.4. The analysis was carried out at approximately 50 μm below the top surface, half-clad distance and approx. 50 μm above the substrate (Table 4.2). Region 1 was characterised by high levels of Co and Cr contents due to presence of dendrites and inter-dendritic structures.

Table 4.4: Chemical composition by EDS of laser-cladded ST6 clads as a function of the regions on the processing map.

Region	Location	C	O	Si	Cr	Fe	Co	W
1	Surface	10.25	3.53	1.59	27.67	2.93	48.01	5.07
	Centre	7.89	-	1.27	30.23	2.72	52.27	5.63
	Fusion	6.12	-	2.28	29.25	13.10	46.39	2.87
2	Surface	10.21	4.90	1.23	27.69	1.98	47.20	4.85
	Centre	7.29	-	-	30.65	2.71	53.37	5.98
	Fusion	8.07	-	1.14	29.81	2.83	50.42	6.66
3	Surface	12.62	-	1.09	29.34	2.65	48.57	4.88
	Centre	8.59	-	2.67	30.58	3.22	54.94	-
	Fusion	6.91	-	-	30.13	3.02	56.07	3.86
4	Surface	10.81	7.00	1.05	26.94	2.51	45.66	5.66
	Centre	8.77	-	2.73	29.48	2.72	56.30	-
	Fusion	9.04	-	1.32	29.64	2.90	51.96	5.15
5	Surface	13.30	-	2.62	27.68	3.03	53.38	-
	Centre	11.83	-	2.50	27.95	2.47	55.25	-
	Fusion	10.29	-	2.48	27.48	2.71	52.04	-

However, the concentration is higher at the center of the clad due to low solidification rate and low thermal gradient thus resulting in formation of these structures. The phase close to the fusion zone was characterised by low cobalt content but as for the Cr content, it was found to be higher than the one found close to the surface. The lower part of the clad has high thermal gradient between the substrate and molten metal of the clad causing rapid solidification. However, higher chromium content indicates that the solidification at the base of the clad was not as rapid compared to the surface as the formation of dendrites was allowed more time close to the substrate. The Fe content (Table 4.4) has shown to be highest at the fusion zone and is much more than that in the starting powder. This indicates that the degree of diffusion of elements from the substrate to the clad (which enhances the formation of undesirable inter-metallics) is highest at the fusion zone. Meanwhile, Fe content at the surface and middle of the clad is just within acceptable range of that in the starting powder.

The clad middle in region 2 has higher contents of Co and Cr compared to other areas of the clad. The region allows for formation and accumulation of dendrites/inter-dendritic structures due to a lower solidification rate. The accumulation of heat at the bottom of the clad also allows for formation of dendrites but not as much as the clad middle because here the substrate acts as a heat absorber which improves the solidification rate to be more rapid than in middle. The surface has lower chromium content which is due to low formation of the dendrites because of the rapid solidification due to energy escape to the surface. The Fe content across the locations of the clad appears to be fairly consistent with that found in the starting powder. This suggest that the process parameters in region 2 are just sufficient to initiate a moderate degree of substrate melting to promote good bonding of the clad to the substrate.

In region 3, it is seen that the concentration of carbon atoms increases in the following order across the locations on the clads: fusion < centre < surface. This is true for all the regions on the process maps. However, the contrary is true for Co in region 3 with its content being highest at the substrate/clad interface. Cr content is highest at the clad middle in comparison to other locations on the clad. This implies that the microstructure in the clad middle was already solidified while the dendrites were still in the process of growing whereas the surface which is made from finer dendrites has lower Cr content which correlates with structures being finer. The Fe content is more at the middle of the clad compared to other location on the clad thereby indicating lack of elemental diffusion from the substrate. This is also attributed by the formation pores at the fusion line meaning that the heat was not enough to melt the base metal and induce diffusion of elements. There is increased Fe content in the inter-dendritic structure which means the accumulation of inter-dendritic structure at the clad middle has absorbed more Fe content during formation.

Region 4 has Cr content lower than 30% across all locations of the clad suggesting that there is no accumulation in any of the locations on the clad. This region allows for formation of finer dendrites. The laser-material interaction is low and this is explained by large pores found at the clad-substrate boundary. However, the clad

middle continues to have more cobalt content. The Fe content is slightly uniform, thus, proving that the process parameters in this region did not impart fusion of the clad and substrate. Region 5 is characterised with microstructures having coarse dendrites. The size of the dendrites tends to be consistent across various locations on the clad. Coarsest dendrites at the middle of the clad could be attributed to increased Cr in this location. Moreover, the inter-dendritic spacing is as well slightly larger at the middle of the clad due to increased Co content, an indication of the correlation between chemical content and the microstructure. The Fe content is highest close to the surface of the clad. This may be accounted for by Marangoni forces acting towards the surface and resulting in elemental diffusion.

In studying the chemical analysis across process map regions, it can be seen that the Cr content which is responsible for corrosion resistance and formation of dendrites increases as follows across the regions: $3 > 2 > 1 > 4 > 5$. This finding provides relevant information in quantifying the formation of dendritic structures. The Co content responsible for formation of inter-dendritic regions is seen to be higher in regions 4 and 5 which explains the lower formation of dendrites compared to other regions. It can be seen also that regions 1 and 2 imparted fusion of the substrate and clad materials which resulted in diffusion of Fe to areas close the substrate-coating interface. Nevertheless, increased diffusion of Fe is noted in region 1. Again, it can be seen that regions 1 and 2 are characterised with W content across different locations on the clad. This could be elucidated by the parameters in regions 1 and 2 allowing for melting and diffusion of W into the clad melt pool whereas there is no uniformity in W content across locations on the clads in regions 3, 4 and 5. Region 1, 2 and 4 are characterized by oxygen contents closer to the surface.

4.3 Comparison of the LC processing and functional performance of the two metal alloy powders

4.3.1 Process maps

Both cobalt-based alloys Tribaloy (T-800) and Stellite-6 process maps have been developed and categorised into 5 regions each. Although they have equal number of

regions, the regions were not the same in terms of their locations within processing windows and their characteristics. Region 1 and 2 for both powders have shown to have less defects (discontinuity or discolourations) compared to other regions. Region 1 for Tribaloy clads occurred at scanning speeds 5-15 mm/sec and feed rates 5-15 g/min while for Stellite 6 region 1 was at a scanning speed range of 5-10 mm/sec and a feed rate of 5 g/min. For both powders the region was characterized by clads that are wide, thick, continuous without discolourations. Region 2 in T800 and ST6 both occurred at a scan speeds of 15-25 mm/sec and feed rates from 5 to 10 g/min. The clads for both metals were narrow, thin, continuous without discolourations. However, the third region for ST6 was established from a feed rate of 10 g/min and a scan rate of 5 to 10 mm/sec whereas that for T800 occurred at 10-15 g/min feed and laser beam speed of 15 to 25 mm/sec. Clads for ST6 became wide, thick, continuous with discolouration while those of T800 were narrow, thick, discontinuous without discolouration. This is the only region in which the clad descriptions are not similar. As for the fourth region for both alloys were found to be narrow, thick, discontinuous and discolored occurring at a feed rate of 15-25 g/min and a beam speed of 15-25 mm/sec for T800 while it occurred at a feed of 10-15 g/min and beam speed of 15-25 mm/sec for ST6. The fifth region is made from feeds of 15 g/min or more for both metal alloys but at 5-15 mm/sec for T800 and 5-25 mm/sec for ST6. The difference in the clads formed at region 3 could be due to the regions occurring at different process maps the other occurs at high energy density (ST6) and the other at low energy density (T800). There is also difference in the parameters for different regions between the metals which might be due to the difference in chemical composition. It was seen that the ST6 clads are characterized by contents of Fe and W in addition to the common contents of Co, Cr, Si and C whereas T800 has Mo. W has high melting point therefore more laser energy density will be required to melt more W hence the eutectic melting point is raised. This explains more clads being discontinuous in ST6.

4.3.2 Microstructural analysis

Tribaloy T800 clads were seen to be formed from a build-up of a eutectic matrix of Cobalt solid solution, cellular crystallization, eutectic laves phase, primary laves phase or fine dendritic structure. In comparison, Stellite 6 does not have lamellar

growths but it is made of columnar or equiaxed dendritic structures surrounded by secondary dendrite arm spacing but those at the surface may be embedded in a Fe-Co-O matrix solution which is concentrated than at the middle of the clad and this occurs in clads fabricated from high energy densities. Substrate-clad bonding from both metal alloys is affected similarly by laser-material interaction parameters with the regions 1 and 2 in the processing maps of T800 and ST6 metals imparting sufficient bonding that resulted from a higher energy input. Dendrites and laves from the Tribaloy clads constitutes of mainly Molybdenum and Stellite 6 dendrites mainly contain Chromium. Both matrix solution of which the structures are embedded within are rich in Co. Even though, both matrix solutions are rich in Co, they differ as Tribaloy has Mo while Stellite 6 contains Fe and W. As for the T800 mechanical bonding between the coating and substrate, it is evident from the fusion zone micrographs that the first two regions (Figure 4.5k, l) do not have a visible fusion line. This is an indication that there was a good bond occurring for region 1 and 2. EDS analysis gave an indication that there was more elemental diffusion for the two first regions (Table 4.2). However, regions 3 to 5 had lesser degree of elemental diffusion at the fusion zone indicating a weaker bonding compared to region 1 and 2. Furthermore, region 3 to 5 have a clear line of fusion (Figures 4.5m, n, o) separating the clad from the substrate which is also characterised by the presence of few and small pores ($<1\text{ }\mu\text{m}$). When studying the fusion zones for bond strength in ST6 clads, the micrograph for region 1 shows a less distinctive separation between the clad and the substrate even though the microstructure is not homogenous (Figure 4.11k). In comparison to region 1, region 2 (Figure 4.11l) seems to have a visible fusion line which can be understood to be an indication of a weaker bond. Region 3 to 5 (Figures 4.11m, n, o) fusion zones are characterised by porosity ($>1\text{ }\mu\text{m}$) and also EDS analysis indicates a lesser degree of elemental diffusion at the fusion zone compared to region 1 and 2 (Table 4.4). These features are a sign of very weak bonding between coatings and substrates. In comparison, T800 cladding has well fused bonds in the first two regions whereas with ST6 it occurs in region 1. Stellite 6 has shown the presence of large pores ($>1\text{ }\mu\text{m}$) along the fusion zones for regions 3 to 5 in comparison to smaller pores found in the same region for T800.

4.3.3 Phase composition analysis via X-ray diffraction (XRD) across the processing regions of the two powders

4.3.3.1 Tribaloy T800 XRD analysis

Phase analysis with X ray diffraction was carried out across different T800 process regions (Figure 4.12 a-e). The XRD patterns also indicates a crystalline structure of the phases. Region 1 was found to be characterized by 5 primary phases clearly confirming the existence of an fcc matrix Co based solid solution, Co (Cr) phase, $\text{Co}_3\text{Mo}_2\text{Si}$ laves phase with an addition of CoMoSi phase, and the eutectic lamellar phases which is Co_7Mo_6 . In addition to that, there is an existence of FeCo. A Phase which attributes to dilution of the clads from intermetallics of the substrate and the coating. Region 2 shows similar presence of the phases but at differing intensity. The Co matrix occurs at an intensity of approximately 2250 counts/sec for both two regions while laves phase for region 1 occur at heights of 2500 to 3000 counts/sec which is lower than that of region 2 occurring at approximately 4700 counts in a second. This effect may be due to proper development of laves structures in region 2 as the laser-material interaction resulted in a lower solidification rate compared to region 1 which was affected by rapid cooling. The laves phase $\text{Co}_3\text{Mo}_2\text{Si}$ were found to be linked with provision of wear resistant coatings (Przybylowicz & Kusinski, 2002). Eutectic phase was found to be at approximately 2400 and at 2400 counts/sec for region 1 and 2 respectively. Both regions 1 and 2 are characterised by FeCo phase. This may be due to the high energy density input being higher and melting the substrate thus allowing for formation of intermetallics. Similarly, EDS analysis of T800 for region 1 and 2 (Table 4.2) has also reflected the presence of Iron content which was rather high in region 1 compared to region 2.. Similar findings of intermetallics were found by Graf *et. al.* (2018) who studied the effect of dilution on the properties of CoCrMoSi alloy coatings produced by Plasma transferred arc welding. The study aforementioned, established that, with an increase in current/energy density there is more dilution that occurs evidenced by formation of carbides and Fe-SS phases a result of high Carbon and Iron content substrate. Region 3 phase analysis has shown to have a reduction in the total number of phases. The absence of FeCo phases can be linked with the reduction in dilution due to very high scanning speeds of $> 15 \text{ mm/s}$. The intensity of laves phases also is reduced which is an indication of lower formation of laves structures in the clad as the laser speed was

high thus there was a reduced amount of melting that occurred in the region. Another phase which was affected is the Co (Cr) which significantly reduced in intensity. Region 4 is attributed with an increment in Co fcc matrix intensity which may be linked with the increase of Co displayed by EDS analysis as well as the insufficient development of laves structures resulting in Co left in the matrix.

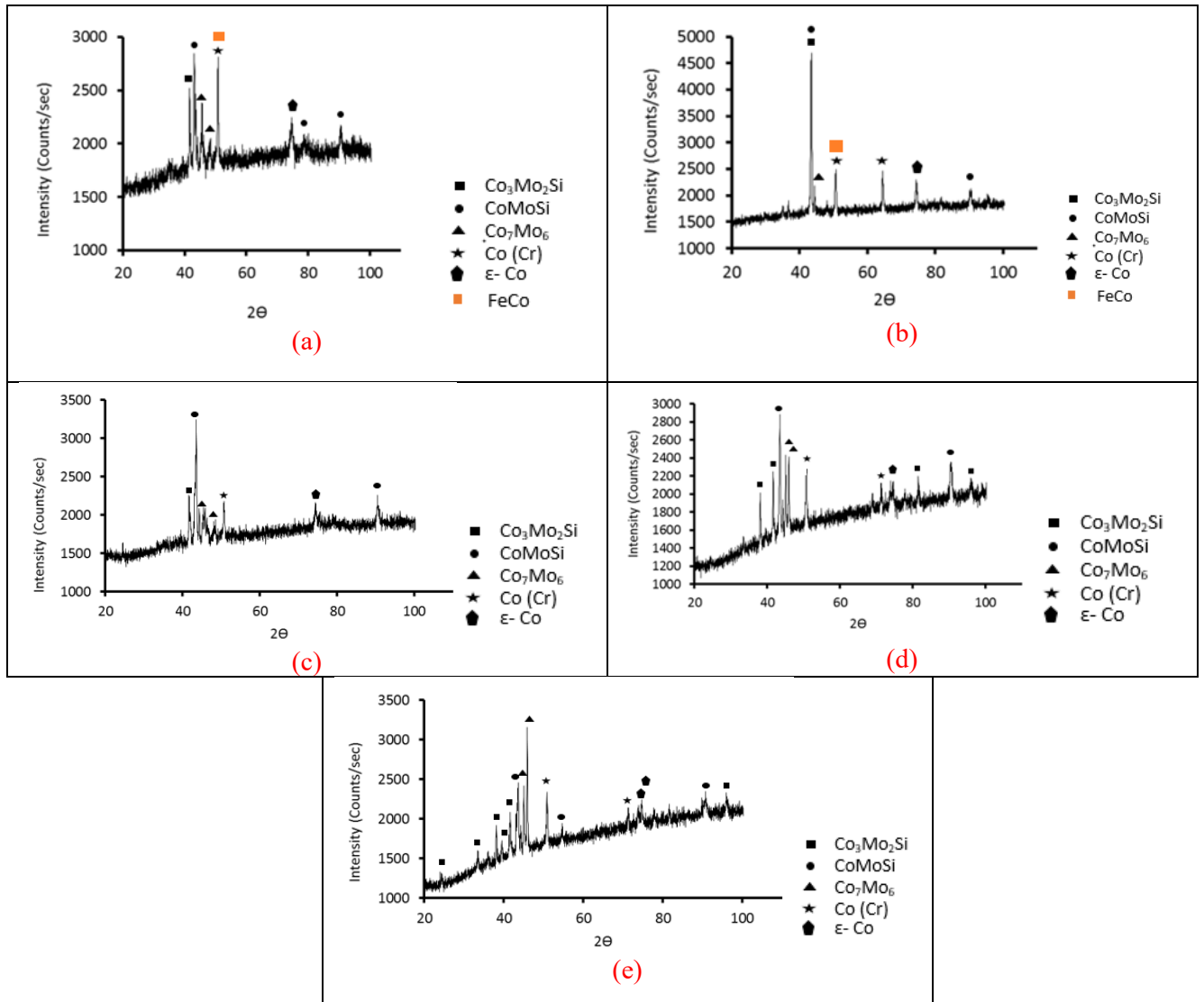


Figure 4.12 (a – e). X-ray diffraction pattern for T800 clads obtained from different regions of the process maps (a) Region 1; (b) Region 2; (c) Region 3; (d) Region 4; and (e) Region 5.

Furthermore, the amount of energy density was low with reference to the powder feed therefore no intensity of FeCo phases were found in the region as there is no

intermetallics effect. Table 4.2 of chemical composition analysis also confirmed the absence of Fe content for the last 3 regions. The energy input for the fifth region only allowed for the formation of cobalt matrix, laves and eutectic lamellar phases without any indication of intermetallics. However, the Co_7Mo_6 dominated the clads in contrast with other regions which were dominated by CoMoSi phase. Since the $\text{Co}_3\text{Mo}_2\text{Si}$ phase is linked with formation of laves and improved wear resistance, it is expected that region 3 to 5 will have less wear resistance in contrast two region 1 and 2 due to less formation of laves phases by the last 3 regions.

4.3.3.2 Stellite 6 XRD analysis

XRD analysis for Stellite 6 clads was also carried out and presented in Figure 4.13 a-e. The clads are characterised by 4 primary phases being Co, CrO, $(\text{Cr, Fe})_7\text{C}_3$ and W_2C phases. Stellite 6 coatings from region 1 which was formed by lower feed rates $< 5\text{g/min}$ and lower scan speed $< 5\text{-}10\text{ mm/s}$, the phase analysis consists of an fcc crystalline structure. The region is characterised by high intensity CrO, $(\text{Cr, Fe})_7\text{C}_3$ and Co phases with intensities ranging from 4000-4200 counts/sec. In region 2, the scan speed was increased to above 10 mm/s. A lower intensity of $(\text{Cr, Fe})_7\text{C}_3$ phase was found compared to in region 1 and which can be eluded as the effects of dilution. Low scan speeds allowed for sufficient laser material interaction in region 1 which might have caused the substrate to melt and mix with the coating. Second region was formed from high scan speed at lower feed rates therefore, the effects led to a lower intensity of the $(\text{Cr, Fe})_7\text{C}_3$. Data collected by EDS (Table 4.4) shows that there was more Fe content recorded from region 1 in comparison to region 2 which may be the reason for a high intensity of $(\text{Cr, Fe})_7\text{C}_3$ in region 1 rather than in region 2. However, the CrO and Co intensity increased to a higher intensity of approx. 5200 counts/sec. This may be due to the fact that the laser-material interaction allowed for proper formation of dendrites as the phases are responsible for the development of the dendritic structure. Region 3 was found to be enriched with high intensities of CrO and Co which also links with the high Co and Cr content found in the EDS analysis of the region. From the micrographs, the dendrites were found to be large at the center due to a lower solidification rate hence leading to the enlargement of the structures.

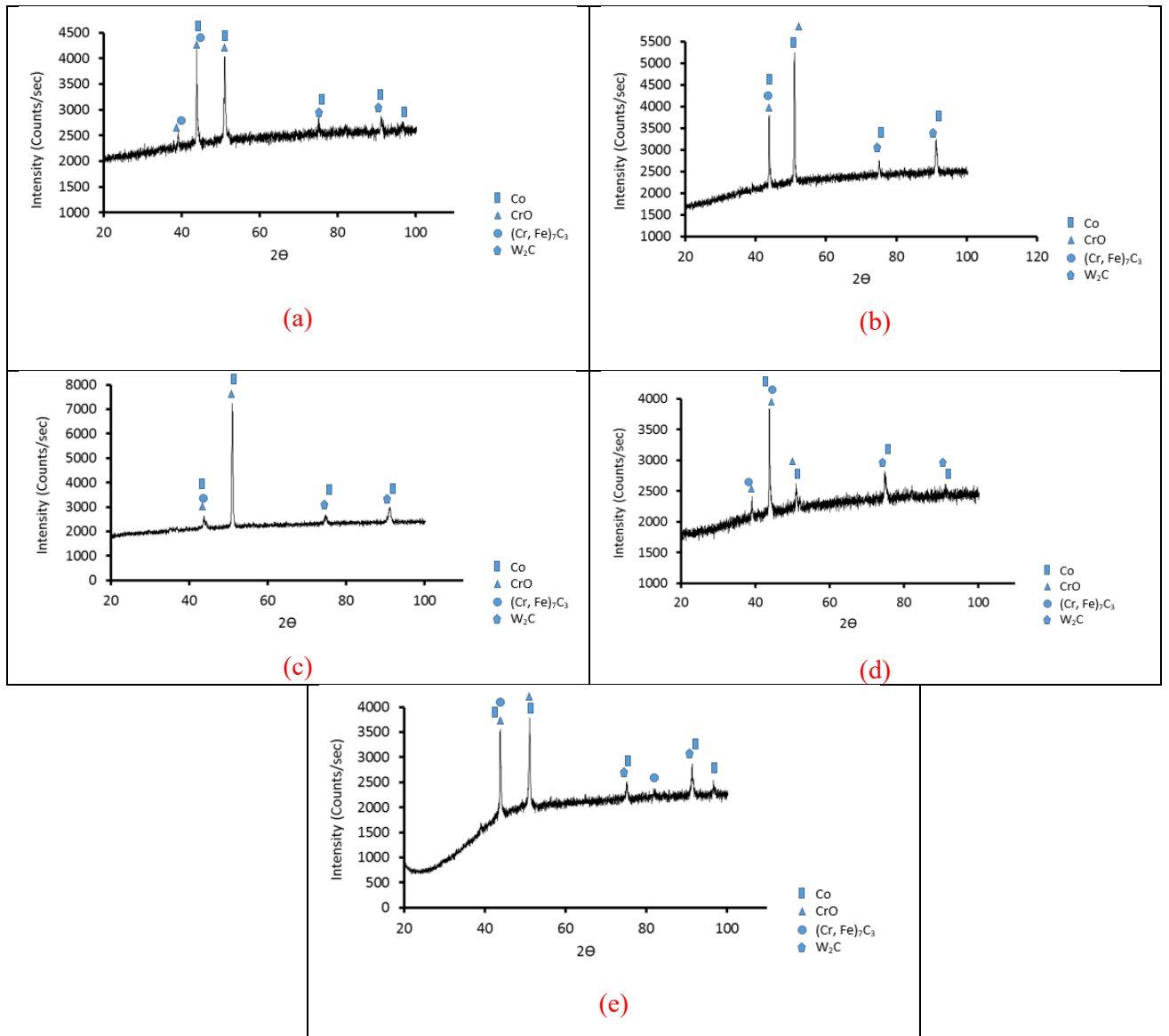


Figure 4.13 (a-e). X-ray diffraction pattern for Stellite 6 clads at different regions 1-5 respectively.

Dendrites are composed of high Cr content from EDS analysis (Table 4.3). as the feed rate was increased to ≥ 10 g/min, the CrO, $(Cr, Fe)_7C_3$ and Co phases were all reduced to intensities lower than 4000 counts/sec which is may be attributed to low development of dendritic structures. Dendrites were found to be the core center for improved hardness and corrosion resistance as indicated by Ruan et. al., 2015. Since Cr is required for formation of dendrites, it is therefore expected that, region 1, 2 and

3 have a higher hardness and corrosion resistance due to a high intensities of CrO and (Cr, Fe)₇C₃.

4.3.4 Microhardness Vickers test and wear resistance across the processing regions

Figure 4.14 compares the microhardness of both metal clads. The microhardness is an average of values obtained under each individual parameter variations at different regions (Appendix G and H). In terms of microhardness, Tribaloy T800 exhibits higher hardness compared to Stellite 6 which suggests its applicability in industries experiencing high equipment wear rates. Region 2 for both powders has the highest microhardness with region 1 becoming the second. Region 2 resulted from the lowest feed rate of 5 g/min and high scanning speeds above 10 mm/sec which indicates that there is proper consolidation within that region. As of region 1, the same feed rate of 5 g/min was used but at scan speed lower than 10mm/sec giving it lower hardness than region 2. This effect is due to high laser energy density causing excessive melting and due to the surroundings remaining very hot, the melt pool stays longer hence porosity occurs from introduction of air into the melt pool(Huang *et al.*, 2011; Xu *et al.*, 2015). Region 3 of Tribaloy clads is a result of >15 mm/sec scan speeds at 10 g/min feed whereas region 3 in Stellite 6 clads arises from the same feed but at <20 mm/sec laser speed. In that case, the microhardness for both metals does not follow the same trend and will have region 3 in Stellite 6 clads being the third in hardness compared to its own regions and the clads are characterised by continuous clads indicating sufficient bonding of powders whereas with T800 clads, region 3 is the lowest to all regions due to discontinuous tracks from high scan speeds.

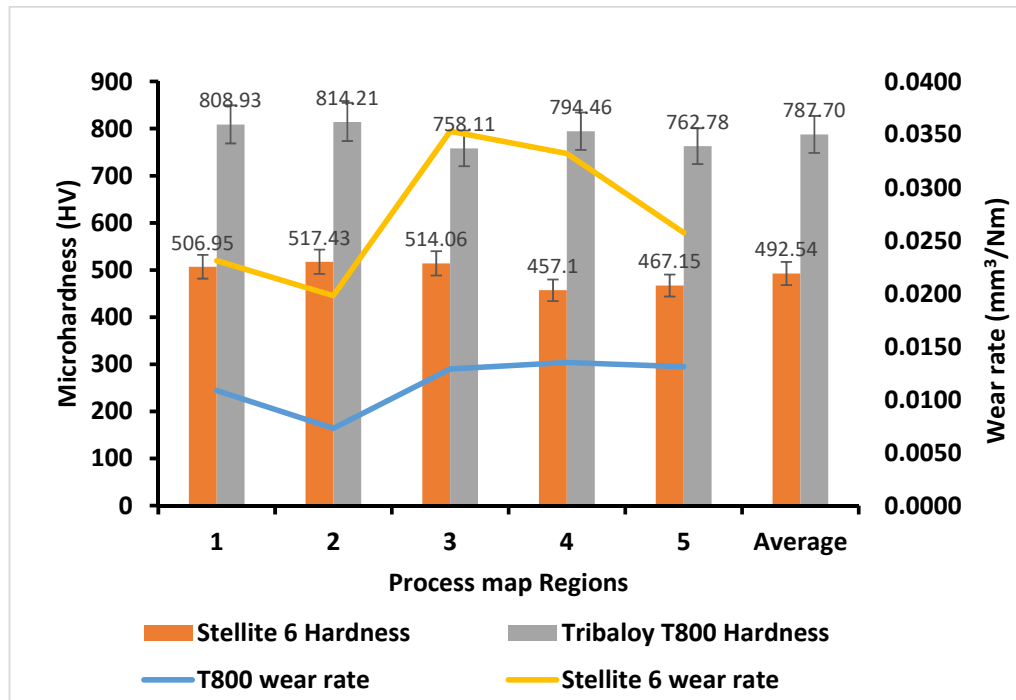


Figure 4.14. Microhardness values for both Tribaloy and Stellite 6 clads.

The regions of 4 and 5 in both metals have low microhardness values than regions 1 and 2 as they are either contaminated due to oxidation or discontinuous (indicating improper particle bonding). Average values of microhardness for both metals was found to be 787.70 HV and 492.54 HV for Tribaloy and Stellite 6 respectively. Other researchers who have worked on the same metal alloys (Navas, Cadenas, *et al.*, 2006; Piasecki & Bartkowski, 2013) were able to get maximum hardness values from laser clad clads of 850 HV and 633 HV for Tribaloy T800 and Stellite 6 respectively which means more research has to be conducted especially effects of other parameters of the process. Based on the analysis, Tribaloy T-800 is recommended for surface modification of pump impeller due to high hardness qualities in comparison to Stellite 6.

Wear test was then conducted for each region and represented in Figure 4.14. Wear rate of Tribaloy T800 was found to range between 0.0073 to 0.0135 mm³/Nm whereas that of Stellite 6 ranged from 0.0198 to 0.0353 mm³/Nm. It is evident that the wear rate for region 1, 2, 3, 4 and 5 was found to be 0.0109, 0.0073, 0.0129, 0.0135 and 0.0131 mm³/Nm respectively. The lowest wear rate was found at region

2 which also recorded the highest microhardness in the process map. This is the region which is also characterised by high intensity $\text{Co}_3\text{Mo}_2\text{Si}$ phases compared to other regions. That is the phase responsible for formations of the laves phases. The highest wear rate was found under region 4 which may be due to incomplete development of microstructural features as there is more feed and less energy density for sufficient melting. The $\text{Co}_3\text{Mo}_2\text{Si}$ laves phases here were very low in intensity as well as in region 5. As for Stellite 6, the lowest wear rate was also established from the second region. This is the region considered to have proper laser-material interaction thus proper inter-particle bonding and improved wear resistance. The XRD pattern analysis indicates high intensity of CrO and $(\text{Cr, Fe})_7\text{C}_3$ phases responsible for the formation of dendrites. Moreover, the highest wear rate was found under region 4 which also has the second lowest microhardness where dendrites are not as refined and fine as in region 2. It is clearly evident that, the microhardness and the specific wear rate have an inverse relationship. An increase in microhardness may result in a reduced amount of wear.

4.4 Conclusions

This study focused in the consolidation 112inimize of clads formed by Tribaloy T800 and Stellite 6 metal powders from varying laser processing parameters. It can be deduced from the content of the study that:

- Processing of clads from each metal resulted in 5 regions of different consolidation 112inimize and for Tribaloy (T-800) they are identified as: (1) Wide, thick, continuous and silvery clads, (2) Narrow, thin, continuous and silvery clads, (3) Narrow, thick, discontinuous and discolored clads, (4) Narrow, thick, discontinuous and/or discolored clads and (5) Wide, thick, discontinuous and/or discolored clads while that of Stellite 6 include (1) Wide, thick, continuous and light grey track, (2) Narrow, thick, continuous and light grey track, (3) Wide, thick, continuous and Brown track, (4) Narrow, thick, discontinuous and/or discolored track and (5) Wide, thick and/or discontinuous and discolored.
- In terms of geometry, thick clads can be attained effectively by increased feed rates while the width can be increased by increased energy input to allow the effect of Marangoni currents to act within the melt pool.
- The microstructural evolution in terms of dendritic structures for both metals is affected by the process parameters in variation. Also the chemical composition can be effected by the processing parameters. Fusion of the clad and the substrate can be altered or improved varying the feed rates and the scanning speed.
- T800 clads are formed from laves, cobalt matrix solution and lamellar structures while ST6 clads are characterized by dendrites and inter-dendritic structures both of which vary in shapes and sizes based on the process parameters.
- Both clads are characterized by contents of Co. Cr and Si. Distinctively ST6 contains Fe and W whereas T800 has Mo.
- XRD patterns for T800 clads show 5 primary phases which are Co matrix, Co (Cr) phase, $\text{Co}_3\text{Mo}_2\text{Si}$, CoMoSi and Co_7Mo_6 while the patterns of Stellite 6 indicate the presence of 4 primary phases being Co, CrO , $(\text{Cr, Fe})_7\text{C}_3$ and W_2C phases.

- Microhardness values for the two metal alloys were also measured under each region. The average microhardness recorded were 787.70 HV and 492.54 HV for Tribaloy and Stellite 6 respectively. The highest microhardness was recorded in region 2 for both metal alloys at which also, the wear rate was lower. The microhardness was found to have a direct relationship with the wear rate.

Chapter Five: Optimisation and modelling of the coefficient of friction, specific wear rate and microhardness of laser clad t-800 clads

5.0 Chapter summary: In this chapter, the metal alloy with the best clad in terms of microhardness was further explored in order to minimize and find mathematical models for its cladding process. In this case, the chosen metal alloy was Tribaloy T-800. So the study begins by minimizing the experimental runs from an L9 orthogonal array which was tested against S/N ratios, ANOVA and interaction contour plots. The microstructure and wear mechanisms on the clads were further explored.

5.1 Statistical analysis, Optimisation and modelling

5.1.1 S/N ratio analysis

Findings from the experimental measurements of the quality characteristics (specific wear rate, COF and microhardness) of LC deposited Tribaloy T-800 clads are presented in Table 5.1 as well as under Appendix J. The experimental outcomes were transformed into S/N ratio and presented in Table 5.1. In an attempt to gain insight into the role of LC process parameters on the wear resistance of the clads, the S/N ratios for all experimental runs were determined by using the statistical software (Minitab 18). The delta values for each factors were then derived from the values of the S/N ratios. Delta values for COF, specific wear rate and microhardness are presented in Tables 5.2, 5.3 and 5.4 respectively. S/N ratios for each response in Table 5.1 were found and were used to calculate individual S/N responses under each set of parameter in Tables 5.2, 5.3 and 5.4. For instance, when calculating the S/N ratio for feed under level 1 for the COF, all S/N ratios with level 1 of feed rate were averaged.

Table 5.1. Experimental results for wear performance and microhardness as well as corresponding S/N ratio.

Run	Feed rate (g/min)	Scan speed (mm/sec)	Spot Diameter (mm)	Specific wear rate (mm ³ /Nm)	S/N ratio	COF	S/N ratio	Microhardness HV	S/N ratio
1	5	15	4	0.0128	37.86	0.0824	21.68	838	58.46
2	7.5	5	4	0.00735	42.67	0.0794	22.00	918	59.26
3	10	10	4	0.0189	34.47	0.0811	21.82	663	56.43
4	5	5	2	0.0074	42.62	0.0828	21.64	879	58.88
5	7.5	10	2	0.0148	36.59	0.0793	22.01	765	57.67
6	10	15	2	0.037	28.64	0.0825	21.67	469	53.42
7	5	10	3	0.00982	40.16	0.0747	22.53	857	58.66
8	7.5	15	3	0.0228	32.84	0.0734	22.69	580	55.27
9	10	5	3	0.0265	31.54	0.0693	23.19	621	55.86

5.1.1.1 Coefficient of friction

The signal-to-noise ratio for the coefficient of friction (COF) considered '*the smaller the better*' desirability. The S/N ratio responses were recorded in Table 5.2 while the main effects plot for S/N ratios were depicted in Figure 5.1.

Table 5.2. Responses table for Signal-to-noise ratios (COF)

Level	Feed rate (g/min)	Scanning speed (mm/sec)	Spot diameter (mm)
1	21.95	22.28	21.77
2	22.23	22.12	22.8
3	22.23	22.01	21.83
Delta	0.28	0.26	1.03
Rank	2	3	1

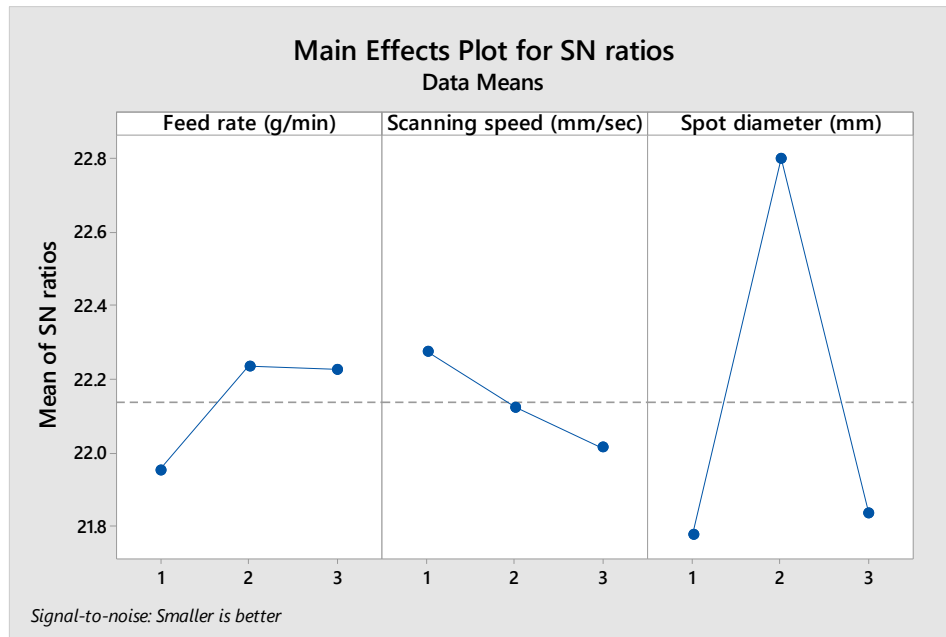


Figure 5.1. Main effects plot for S/N ration-Coefficient of friction.

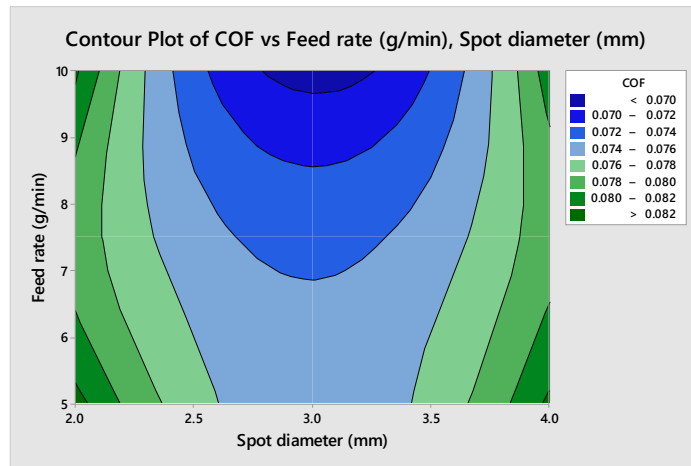
Table 5.2 reveals that the delta value for spot diameter was the highest, hence, suggesting that it imparted the highest influence on COF relative to feed rate and scanning speed. The process parameters influence the COF in the following order: spot diameter>feed rate>scan speed. Analysis of the main effects plot for S/N ration for COF (Figure 5.1) indicates the following relationships between the process parameters and COF: The S/N ratio for COF is seen to be increasing initially as the feed rate increases from level 1 to level 2 parameter setting while the S/N ratio remain constant as feed rate increases from level 2 to level 3. Initial decrease in COF reported for feed rate increment from level 1 to level 2 could be ascribed to low feed rates at level 1 which promoted excessive melting and formation of pores because of prolonged solidification, now being avoided when feed rate was increased to level 2. Pore formations might have caused high values of COF at level 1. Meanwhile, constant value of COF obtained with feed rate increasing from level 2 to level 3 could be elucidated by the energy density being enough and able to melt powders sufficiently from a feed of 7.5 to 10 g/min. Examination of how the scan speed influences S/N ratio (COF) suggest an indirect relationship in which highest S/N ratio was obtained at the lowest scanning speed. This implies that the clads are imparted with lowest COF at the lowest scan speed. This observation could be explained by the laser-material interaction from low scan speeds allowing for melting of powders

thus formation of surfaces with low friction (Che *et al.*, 2014; Gu *et al.*, 2015). The beam spot diameter is seen to impart the highest S/N ratio (COF) at level 2 (3mm) relative to levels 1 (2mm) and 3 (4mm) parameter settings. Analysis of Figure 5.1 shows that lowest COF (indicated by highest S//N ratio) can be obtained at the following parameter settings: feed rate (7.5 g/min), scanning speed (5 mm/sec) and spot diameter (3 mm).

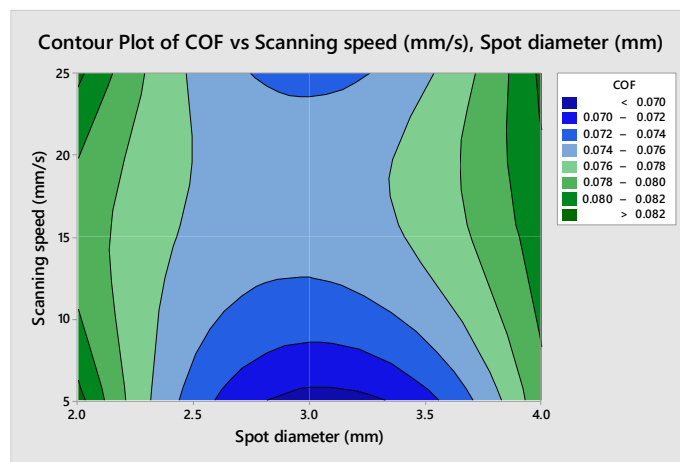
Figure 5.2 illustrates how the interactions between process parameters influence COF namely: (a) spot diameter, feed rate (b) spot diameter, scanning speed (c) feed rate, spot diameter. It could be confirmed from Figure 5.2a that COF is best minimized (<0.07) when laser beam spot diameter of 3 mm was employed in conjunction with feed rate above 9 g/min for LC deposition of the clads. Nevertheless, undesirable high COF values were obtained when laser beam spot diameter other than 3 mm was employed for LC deposition irrespective of powder feed rate. A lower COF (<0.07) was found to be achieved when cladding with feed rates above 9 g/min were introduced. At feed rates lower than 9 g/min, the COF became higher. Figure 5.2b shows an interaction between the scan speed and the spot diameter. It was seen that low COF (<0.07) was achieved when laser beam speed lower than 10 mm/sec was used. A spot diameter of 3 mm continues to give a lower COF compared to other diameters. It was further confirmed in Figure 5.2c, that, an interaction of high feed rate (>9 g/min) and low scan speed (<10 mm/sec) was responsible for very low COF (<0.07).

5.1.1.2 Specific wear rate

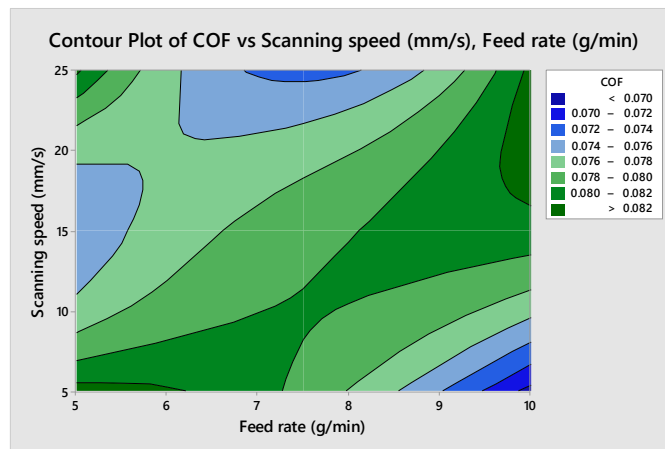
A higher delta for S/N ratios for the specific wear rate was found under the feed rate showing that the feed rate was more influential in comparison to the scanning speed and the spot diameter. The sequence of influence goes from feed rate, scanning speed to spot diameter respectively with spot diameter giving the lowest delta. The main effects plot for S/N ratios against specific wear rate is shown under Figure 5.3. Feed rate shows a great variation in S/N ratios. The plot of which begins with a very low wear rate found at 5 g/min and rapidly increases when the feed rate is increased to 7.5 g/min. The cause of which is due to the feed rate becoming more than what the energy density can handle thus a weak bond within the powders which can easily be worn away by abrasion.



(a)



(b)



(c)

Figure 5.2 (a-c). Contour plots of process variables against COF (a) Feed rate vs Spot diameter, (b) Scanning speed vs spot diameter and Feed rate vs scanning speed.

Table 5.3. Responses for signal-to-noise ratio (Specific wear rate).

Level	Feed rate (g/min)	Scanning speed (mm/sec)	Spot diameter (mm)
1	40.21	38.94	35.95
2	37.37	37.07	34.84
3	31.55	33.11	38.33
Delta	8.66	5.83	3.49
Rank	1	2	3

Similar effects were explained by Gu (2015) who attributed that high energy densities support wetting of powders causing them to be densified. Further increment of the feed rate to 10 g/min further increases the wear rate of the clad. Similar relationship was attributed by the scan speed which indicated a positive correlation with the wear rate. Low specific wear rate was achieved at the lowest scan speed of 5 mm/sec and as it was increased to 10 mm/sec, an initial increase of the wear rate was observed. Additionally, the scan speed was further increased to 15 mm/sec and a more rapid increase in the wear rate resulted. This is attributed to a laser-material interaction that provides sufficient melting due to a lower scan speed thus resulting in formation of fine dendritic structure from slow solidification rates. A fine dendritic structure was found to be significant for retaining of mechanical properties such as wear and corrosion resistance (Ruan *et al.*, 2015). As for the spot diameter, a different trend was observed where the wear rate was low for a spot diameter of 2mm then increased for a laser beam diameter of 3 mm. The high wear rate at a 3 mm diameter was short lived when the diameter was further increased to 4 mm and the lowest wear rate was recorded. A low wear rate is found to be given by process parameters of 5 g/min (feed rate), 5 mm/sec (scan speed) and 4 mm (spot diameter).

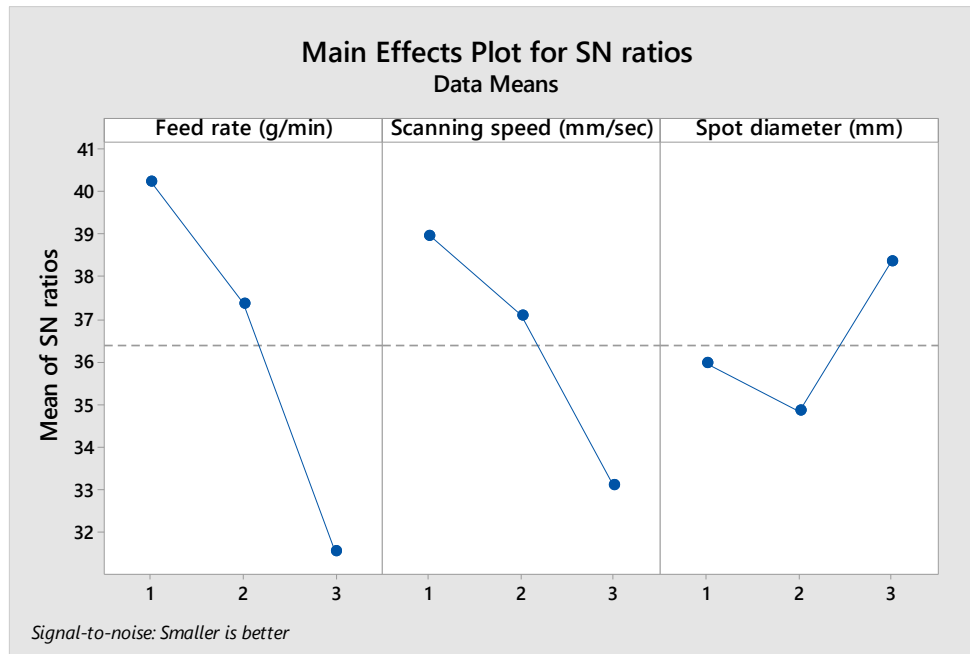
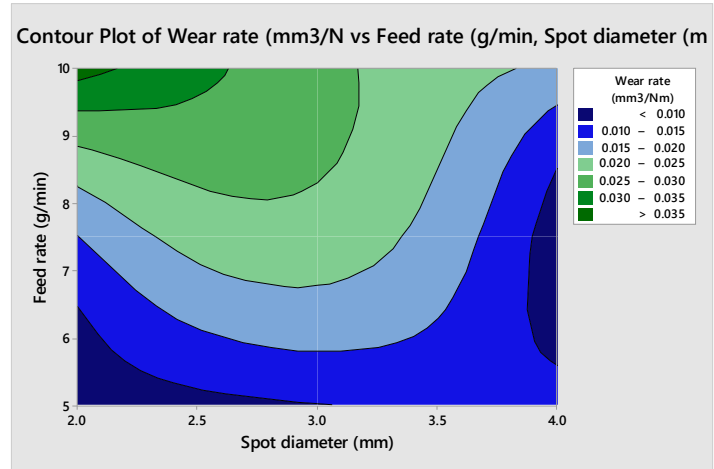
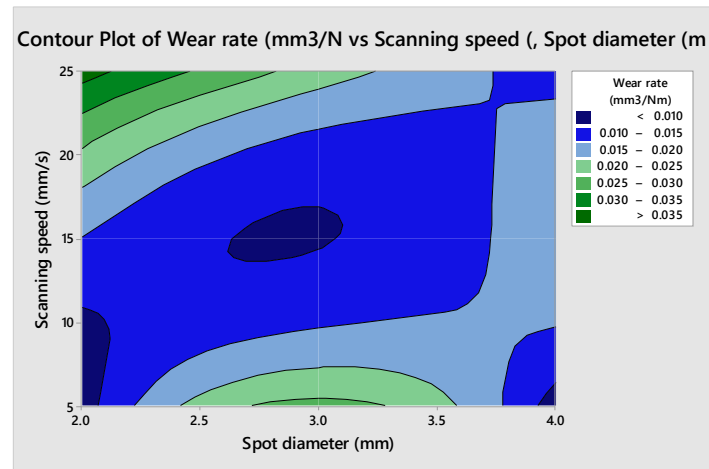


Figure 5.3. Main effects plot for S/N ratio-Specific wear rate.

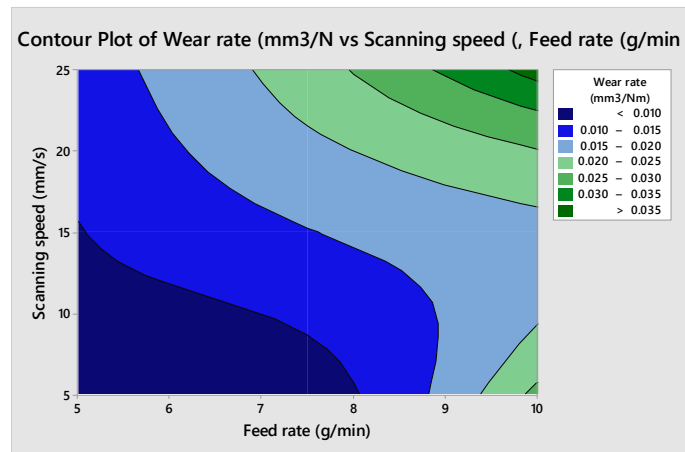
Contour plots for the process variables against the specific wear rate are illustrated below in Figure 5.4 (a-c). The interaction between the feed rate and spot diameter in Figure 5.4a shows that, specific wear rate below 0.010 was achieved by feed rates of 8 g/min and below whereas the spot diameter has to be lower or above that of 3 mm. Illustration of the interaction between the laser beam speed and spot diameter shows a different relationship with the wear rate. Low wear rates (<0.010) were achieved when scan speed is below 20 mm/sec while all distinctive values of spot diameter were able to provide lower feed rates. Lastly, Figure 5.4c shows an interaction of feed rate and laser beam speed against the wear rate. An interaction of lower scan speed <15 mm/sec and feed rates <8 g/min gave low specific wear rates below $0.010 \text{ mm}^3/\text{Nm}$.



(a)



(b)



©

Figure 5.4 (a-c). Contour plots of process variables against Specific wear rate
(a) Feed rate vs Spot diameter, (b) Scanning speed vs spot diameter and Feed
rate vs scanning speed.

5.1.1.3 Microhardness

A summary of the raw microhardness values for the various samples is shown under Appendix K and an average further used. The DoE analysis for microhardness considered the aspect as ‘*the large the better*’ as this outcome was desired for clad durability. Similarly, the responses to S/N ratios were obtained and studied. Table 5.4 shows a summary of the responses of S/N ratios as well as Figure 5.5 which shows the main effects for S/N ratios plotted against their variables. High S/N ratios are a good indication of optimum parameters and they were bolded as seen on the table.

Table 5.4. Responses for signal-to-noise ratio (Microhardness).

Level	Feed rate (g/min)	Scanning speed (mm/sec)	Spot diameter (mm)
1	58.67	58	56.66
2	57.4	57.59	56.6
3	55.24	55.72	58.05
Delta	3.43	2.28	1.45
Rank	1	2	3

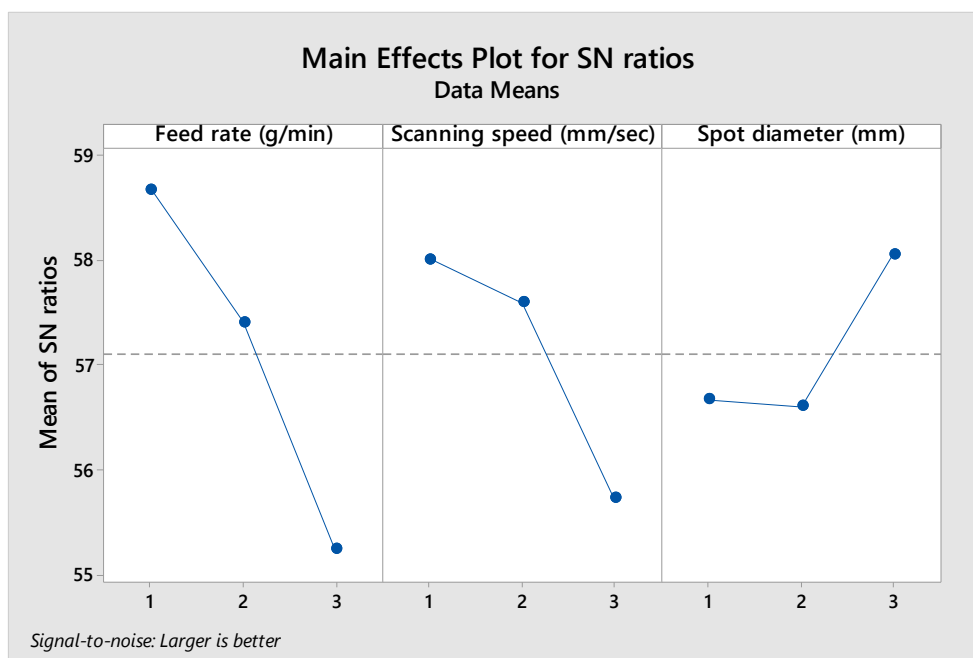
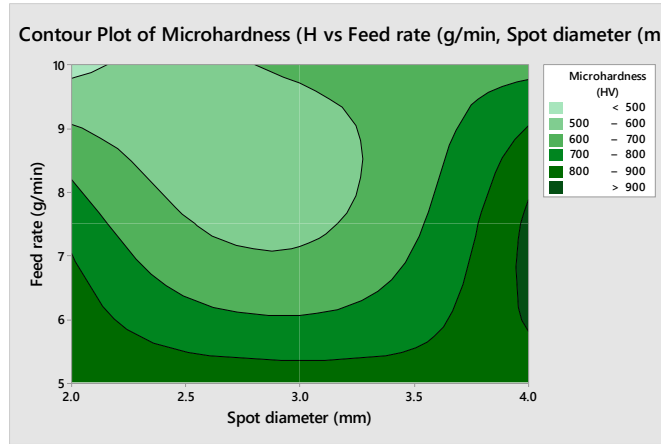


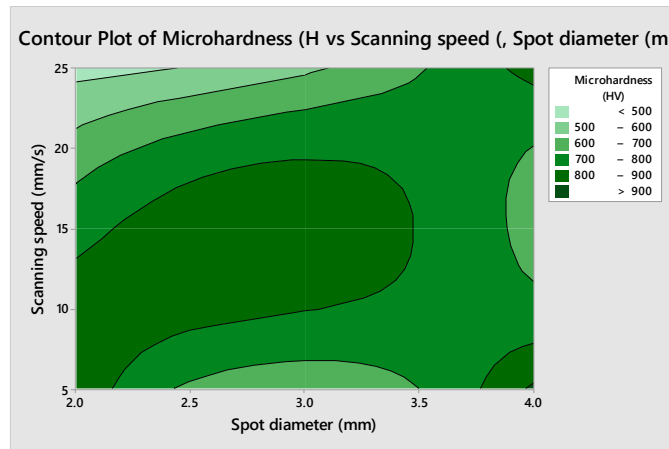
Figure 5.5. Main effects plot for S/N ration-Microhardness.

As shown by the delta values under Table 5.4, microhardness was found to be highly influenced by the feed rate followed by scanning speed and lastly by Spot diameter. Figure 5.5 shows that, a greater T-800 hardness value was achieved by cladding at 5 g/min. A decrease in feed to an amount of 7.5 g/min, resulted in a rapid drop in the microhardness. Further decrease in the feed rate to 10 g/min caused the microhardness to further drop to lower values. As for the scan speed, a 5 mm/sec speed began with a higher microhardness and which reduced as soon as a scan speed of 10 mm/sec was used, the microhardness was reduced rapidly when the scan speed was increased to 15 mm/sec. These lower parameters for scan speed and feed rate were found to promote sufficient melting and similar effects to those were found by Gedda, (2004) in which clads were wide due to excessive melting. A graphical illustration for the spot diameter shows a slight reduction of microhardness from a spot diameter of 2 to 3 mm but when a diameter was changed to 4 mm, the microhardness was rapidly increased. The highest microhardness was found to be attained from a combination of process parameters of feed rate at 5 g/min, scan speed at 5 mm/sec and spot diameter of 4 mm.

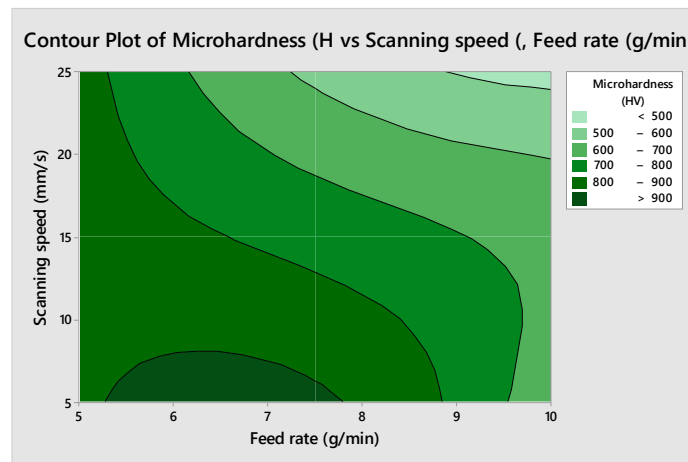
Finally, the effects on microhardness were investigated (Figure 5.6). The plot for feed rate against the spot diameter in figure 5.6a indicated that, the microhardness values above 900 HV were achieved when the feed rate was at 9 g/min and below whereas the spot diameter has to be at 4 mm. As for the plot of scanning speed against spot diameter, scanning speeds below 20 mm/sec were responsible for >900 HV while spot diameter had to be at < 3.5 mm. Lastly, the interaction between feed rate and scan speed achieved great hardness values when a laser beam speed lower than 10 mm/sec was introduced with a feed rate lower than 8 g/min into the cladding process. In summary of the S/N ratio analysis, it was found that the spot diameter highly influenced the coefficient of friction whereas feed rate significantly influenced the specific wear rate and microhardness. This is what is expected when carrying out the Analysis of variance tests (ANOVA). Furthermore, the S/N ratios response were very high for the microhardness followed by those for wear rate and COF respectively. This is an indication that the varied process parameters were very influential on the microhardness of Tribaloy T-800 clads.



(a)



(b)



©

Figure 5.6 (a-c). Contour plots of process variables against Microhardness (a) Feed rate vs Spot diameter, (b) Scanning speed vs spot diameter and Feed rate vs scanning speed.

5.1.2 ANOVA and Multiple Linear regression model

Minitab 18 was further used for mathematical modelling of T-800 clads. Multiple linear regression statistical tool was employed to predict from the data sets, the amount of each process parameters required to adjust the wear rate, microhardness and COF. ANOVA and coefficients tables were generated in order to come up with the equations.

Calculation of the F values was carried out by calculating the standard deviation within the factors and between factors. The standard deviations were used to calculate the adjusted sum of squares for the factors and for the residual error respectively. Sum of squares is the sum of variances from the mean value. Mean squares were then calculated by dividing the sum of squares by the degree of freedom (Df). The Df is the count of factors minus one. The F-value was determined by Factor mean square divide by residual error mean square. The residual error mean square is the level of noise and an F-ratio more than one means the factors have an effect more than the level of noise. Since it cannot be understood the error of the noise or at what level above the noise the effects could be considered practical, the p value is then used. The p value is obtained from a DF versus F test table. It gives statistical value which can be determined if it falls within the probability range of the noise or not. If the p value is less than 5% it is then not in the range of the noise and the effects are considered to be practical.

It was noted after running the Minitab software that there was no interaction between factors and therefore the analysis was only carried out for factors individually. Table 5.5 below was found to measure the level of significance of the three factors (feed rate, laser beam speed and spot diameter) on the coefficient of friction. The F value for scanning speed was below 1 which means it is not practical. Those for feed rate and spot diameter were more than one at values of 1.54 and 19.47 respectively. The p value then was used to further test the significance and less than 0.005 was regarded as significant therefore feed rate was still within the insignificant range despite a positive F-value. The only significant factor for the COF is the spot diameter.

Table 5.5: ANOVA analysis for the Coefficient of friction. (Pooled).

Source	DF	SS	MS	F-Value	P-Value
Feed rate (g/min)	2	0.000012	0.000006	Pooled	
Scanning speed (mm/sec)	2	0.000008	0.000004	Pooled	
Spot diameter (mm)	2	0.000155	0.000077	19.47	0.049
Error	2	0.000008	0.000004		
Total	8	0.000183			

*Note $F'_{0.05}(2, 2) = 9.00$ for 95% confidence.

The parameters varied against specific wear rate were also tested for their level of significance on the response (Table 5.6). Parameters which had values more than 9.00 for the F-test were an indication that they have were significant at 95% confidence towards the wear rate as a result those which were not significant were pooled. However, the P- values were 0.011, 0.027 and 0.059 for feed rate, scanning speed and spot diameter respectively. These results indicate that the Feed rate and scan speed were significant against the specific wear rate but the highest influence is with feed rate.

Table 5.6. ANOVA analysis for the Specific wear rate.

Source	DF	SS	MS	F-Value	P-Value
Feed rate (g/min)	2	0.000485	0.000243	86.16	0.011
Scanning speed (mm/s)	2	0.000204	0.000102	36.16	0.027
Spot diameter (mm)	2	0.00009	0.000045	15.95	0.059
Error	2	0.000006	0.000003		
Total	8	0.000785			

*Note $F'_{0.05}(2, 2) = 9.00$ for 95% confidence.

Furthermore, a significance test was carried for the microhardness on Table 5.7. The F values were significantly high for all factors (more than 9.00). However, the P values tell a different story as it could be seen that the feed rate is significant but as for the scanning speed the value is at 0.053 which is not significant but arguably could be considered significant as literature has shown that the scan speed works hand in hand with the feed rate. For instance, for a feed rate of 5 g/min and a laser beam speed of 5 mm/sec there could be sufficient melting without excessive melting

but a reduction to 3 mm/sec could cause an excessive melting. A researcher Barekat *et al.*, (2016), when studying effects of scanning speed and feed rate on clad geometry found similar discoveries.

Table 5.7. ANOVA analysis for the Microhardness. (Pooled).

Source	DF	SS	MS	F-Value	P-Value
Feed rate (g/min)	2	114540	57270	40.13	0.024
Scanning speed (mm/sec)	2	50895	25447	17.83	0.053
Spot diameter (mm)	2	25220	12610	Pooled	
Error	2	2854	1427		
Total	8	193510			

*Note $F'_{0.05}(2, 2) = 9.00$ for 95% confidence.

Multi-linear regression analysis was then carried using the software for mathematical modelling of the LC process parameters based on the preferred response. Regression equations 5.1, 5.2 and 5.3 were obtained for the adjustment of process variables in order to control the COF, microhardness and specific wear rate respectively.

R-sq = 8.95%

Regression equation:

$$\text{COF} = 0.0821 - 0.00117 F + 0.00113 S - 0.00028 D \quad (5.1)$$

Where **F** = feed rate in g/min, **S** = scanning speed (mm/sec) and **D** = spot diameter (mm/sec).

R-sq = 90.4%

Regression equation:

$$\text{Microhardness (HV)} = 1127.1 - 136.8 F - 88.5 S + 51.0 D \quad (5.2)$$

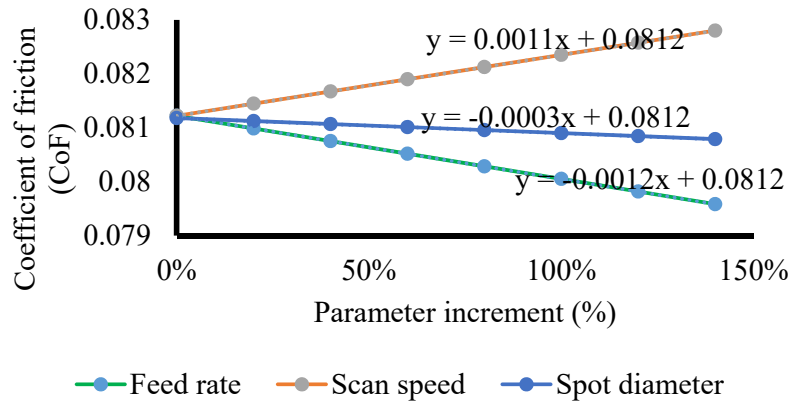
R-sq = 87.77%

Regression equation:

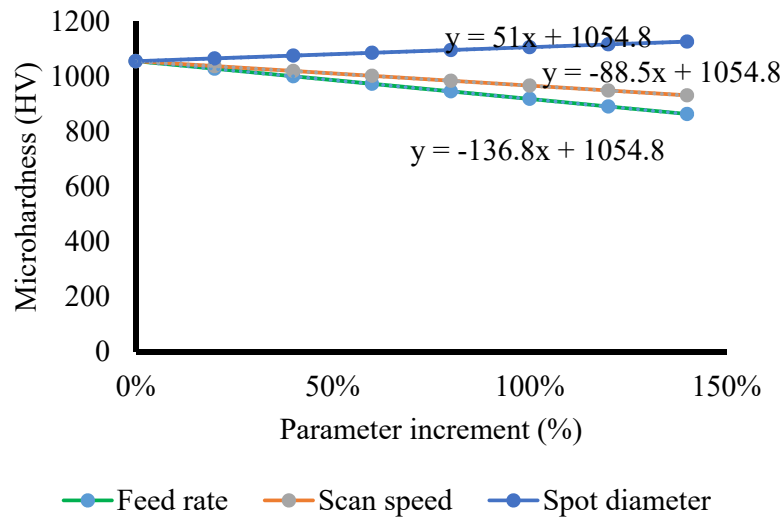
$$\text{Wear rate (mm}^3/\text{Nm)} = 0.00219 + 0.00873 F + 0.00523 S - 0.00336 D \quad (5.3)$$

5.2 Sensitivity Analysis

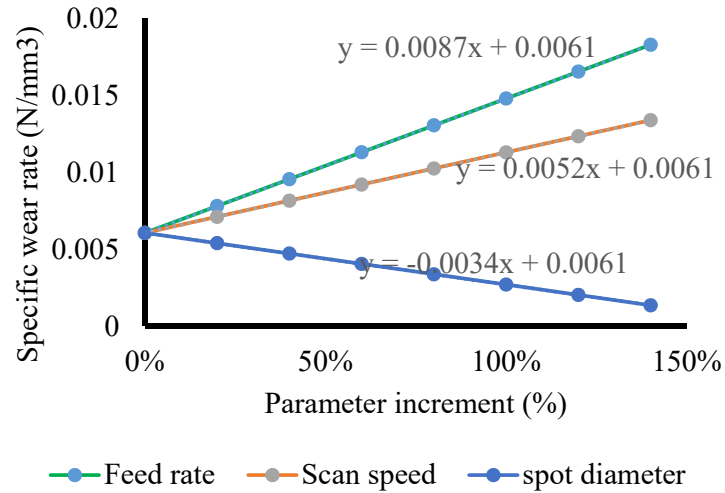
Sensitivity analysis was used to measure the effects of change on individual process parameters towards the clad characteristics. In order to achieve this, a single parameter within the ones measured (feed rate, scanning speed and spot diameter) was varied while others were kept at constant. 0% change is measured as the lower level input and increased at intervals of 20% to a maximum of 140%. The increment were then inserted into the regression equation to obtain the outcome. The results are shown in Figures 5.7 (a-c). The level of sensitivity can be measured based on the gradient of the linear relationship found on the illustrations (Figure 5.7 a-c). The CoF was found to be highly sensitive to change in feed rate which shows a gradient of 0.0012 seconded by the scanning speed at a gradient of 0.0011 and lastly spot diameter at 0.0003. The increment for both feed rate and spot diameter has a negative correlation towards the CoF. Furthermore, microhardness was also found to be highly sensitive to feed rate followed by laser beam speed and beam diameter in that order but at gradients of 136.8, 88.5 and 51.0 respectively. The gradients for scan speed and feed rate are negative, which is an indication of an inverse relationship with the clad hardness. The magnitude of the gradient is essential in measuring the steepness or level of sensitivity without considering whether it is negative or positive. Moreover, the specific wear rate of the clads were also evaluated for sensitivity. The outcome of which depicted that wear rate is more sensitive to the change in feed rate which shows a positive correlation similar with the correlation of scan speed. The sensitivity towards the scan speed is less in comparison to that of the feed rate. In contrast, the spot diameter has a negative correlation with the wear rate and has less impact. In summary, it has been discovered from this study that the CoF, specific wear rate and microhardness were highly sensitive towards the feed rate followed by the scanning speed and lastly the spot diameter.



(a)



(b)



(c)

Figure 5.7. Sensitivity analysis of process parameters towards (a) Coefficient of friction, (b) microhardness and (c) specific wear rate.

5.3. Microstructural characterisation and wear mechanism of the clads

5.3.1 Microstructural characterisation

Figure 5.8 shows the samples after they were grinded and polished in order to reveal whether or not they have porosity and cracks. Samples 1, 3 and 4 had cracks of which Sample 3 showed the greatest number of cracks. Clad 1 and 4 only show a few individual cracks that run across the clad.

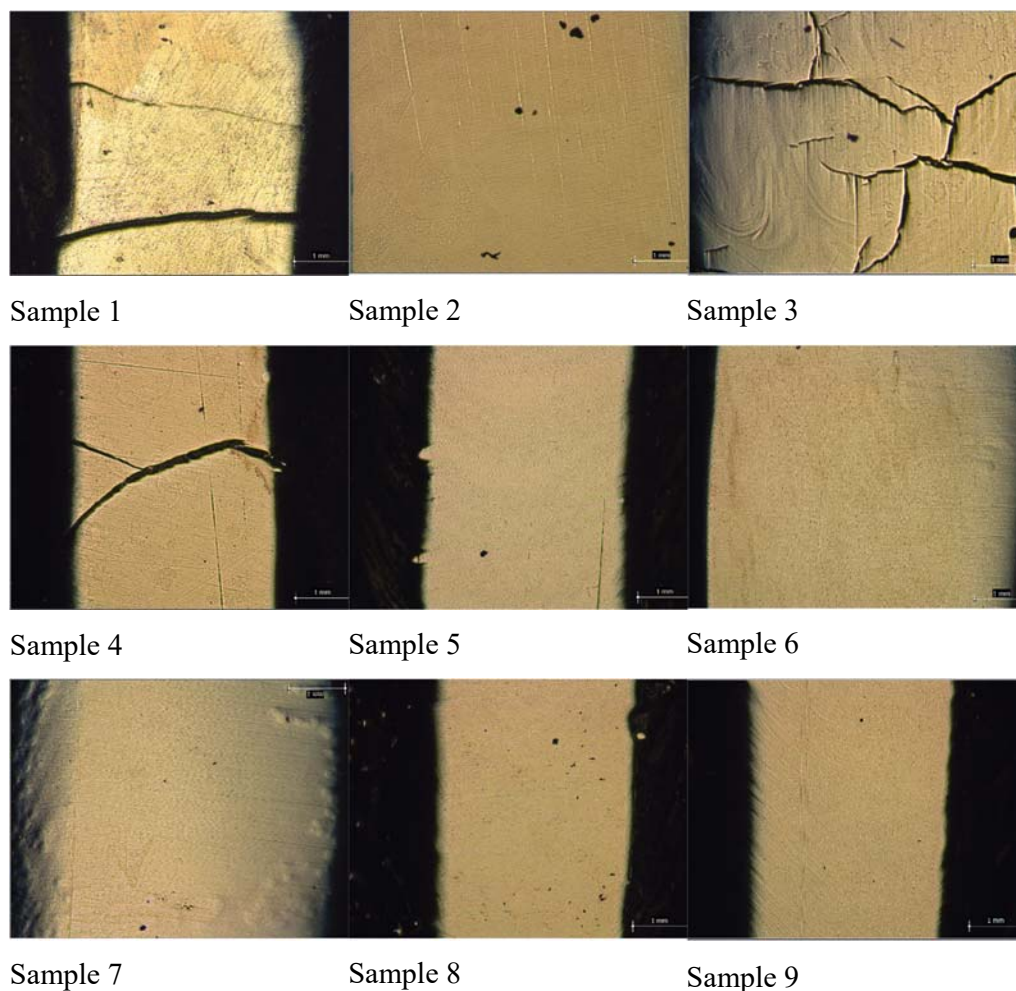


Figure 5.8. Showing transverse sections of Laser Cladded single track single layer clads

Sample 2 appears to have a fabrication characterized by a few sized pores. Similar findings are with sample 8 in which the clad was characterised by a few pores but they are smaller in comparison to those found in sample 2. Sample 5, 6, 7 and 9 are

seen to have unnoticeable defects. The presence of cracks and pores are not desirable in clads as they impart poor mechanical properties. Cracks have been associated with residual stresses that occur within the clad during the cooling process. For instance, Wang *et al.* (2015) studied residual stresses of laser cladmed composites and established that residual stresses were associated with the cracking behaviour of clads which mostly propagate from the clad-substrate boundary to the surface. Residual stresses at the clad-substrate boundary could be accounted for by high thermal gradient existing between the substrate initially at room temperature and the high temperature incident laser beam. The effects of these defects on the functionality of the clads will be discussed later on especially on the wear rate.

Microstructures of the samples with high (sample 6), medium (sample 8) and low (sample 2) wear rate were shown in Figure 5.8 below. The sample with the medium wear rate was found by locating the value ($0.022 \text{ mm}^3/\text{Nm}$) between the highest ($0.037 \text{ mm}^3/\text{Nm}$) and the lowest ($0.00735 \text{ mm}^3/\text{Nm}$). The closest with the value ($0.022 \text{ mm}^3/\text{Nm}$) was $0.028 \text{ mm}^3/\text{Nm}$ from sample 6. Sample 2 was found to have a very fine dendritic structure with an estimated individual size of approximately $1 \text{ }\mu\text{m}$. This was a result of a feed rate of 7.5 g/min and a lower scan speed of 5 mm/sec which allowed for proper laser-material interaction hence fine dendrites formed. Sample 8 consists of a mixture of eutectic laves structures and a secondary eutectic matrix. In this case the feed rate is minimal to that of sample 2 but the scan speed was higher at 25 mm/sec and this amount of laser material interaction, the heat is only retained at the surface due to Marangoni convection currents and less heat reaches the base therefore there is no heat flow to the base metal thus formation of large laves at the surface. Estimated size of a singular average laves structure was found to be approx. $7 \text{ }\mu\text{m}$. Sample 6 similarly consisted of eutectic primary laves as seen in Figure 5.9.

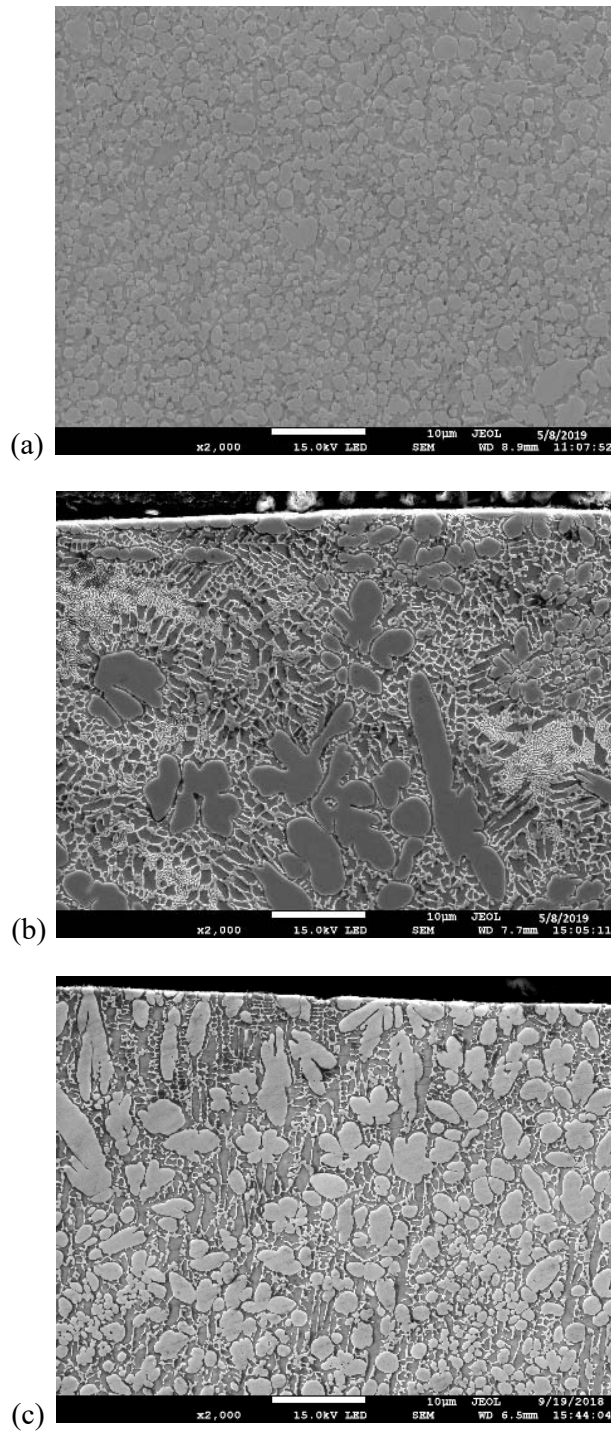


Figure 5.9. SEM imaging of samples with (a) low (sample 2), (b) medium (sample 8) and high (sample 6) specific wear rate respectively.

5.3.2 Wear analysis

Wear mechanism was investigated at micro level under the scanning electron microscope (SEM) in order to understand the wear behaviour of the coating as parameters are varied. The wear scar was found to be characterised by secondary features such as plows, valleys and peaks as shown on Figure 5.10 in similarity to findings by Raymond (2004) when studying the abrasive behaviour of polymer surfaces. Plowing and cutting were found to be associated with abrasive wear for most samples (Bayer, 2004; Navas *et al.*, 2006).

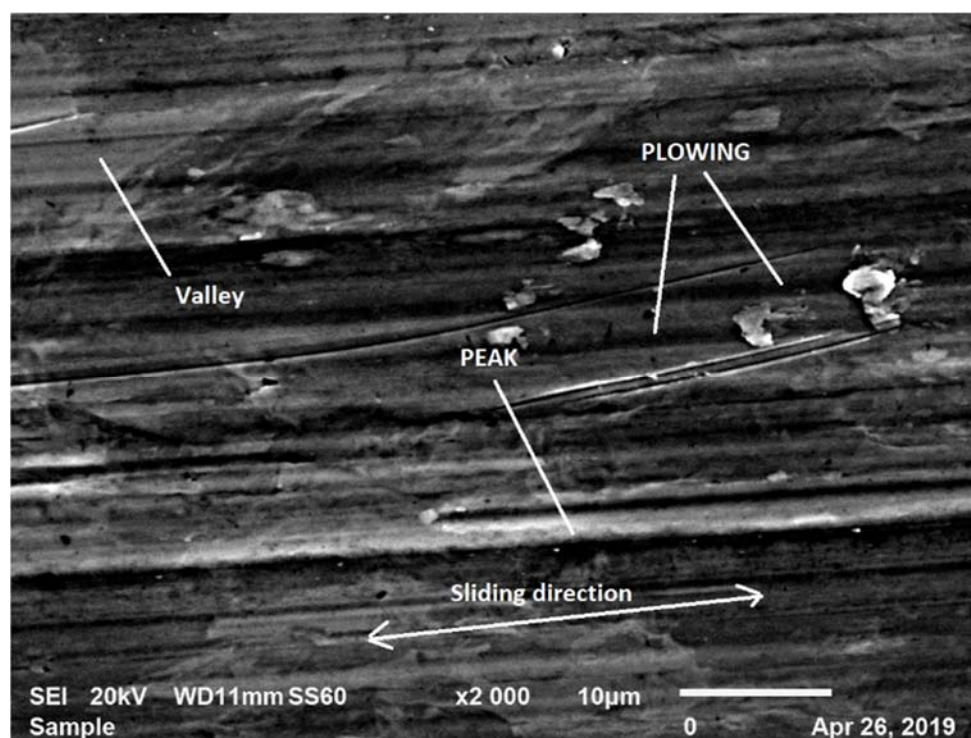
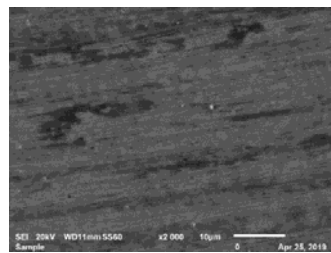


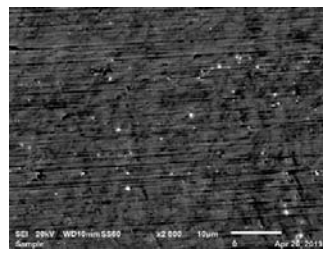
Figure 5.10. Secondary features found within wear scars.

It was found out that the wear scars differed in appearance across all samples as shown on Figure 5.11. The secondary features appeared to be mild or severe which is similar to findings by Raymond (2004) in a study of wear morphology of lubricated and unlubricated wear scars. Sample 1 showed a mild wear morphology by which the surface appeared to have traces of plows that were very faint. This is an indication that the wear was not much. The faint plows were estimated to be of 3 μm in width and peak to peak distance of 7 μm . Sample 2 in the other hand was characterised by

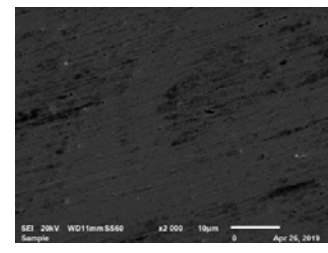
lightly severe wear scars that feature very thin plows indicating that the asperities from the wear ball were able to plow their way through the specimen surface. The plow appeared to be distributed across the scar and were numerous with an individual width estimated at $0.5\ \mu\text{m}$. The peak to peak distance can be seen as approximately $1\ \mu\text{m}$ and the plenty small plows might amount to a greater amount of surface area hence a greater amount of wear. Sample 3 has similar morphology with sample 2 through the existence of fine plows across the wear scar. In distinction, sample 3 possesses mild secondary structure and appear to be very faint which could amount to lower wear volume. The fourth sample bears a scar with a mixture of both severe and mild scars with large plowing appearing as mild and fine plowings showing as severe. The peak-to-peak distance of the large mild features was estimated at $15\ \mu\text{m}$ while for the fine severe features at $2\ \mu\text{m}$.



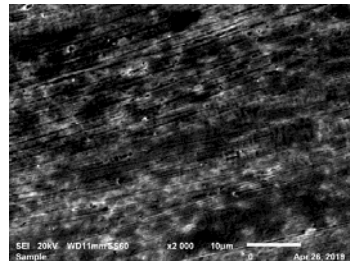
Sample 1



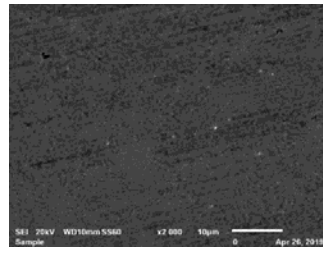
Sample 2



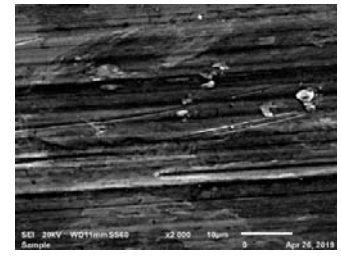
Sample 3



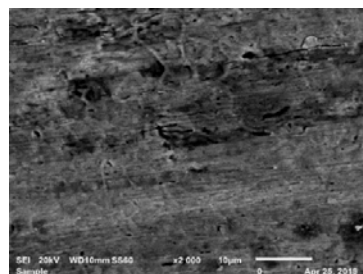
Sample 4



Sample 5



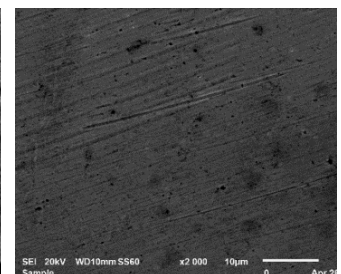
Sample 6



Sample 7



Sample 8



Sample 9

Figure 5.11. Showing 2000x magnification SEM images of wear test scars as they appear on different T-800 samples.

The wear mechanism micrograph for sample 5 shows a mild wear which occurred very lightly and amounting to almost a polished surface. The direction of sliding could be easily identified but the plowing is very faint. Sample 6 has an indication of high severe wear with visibly deep and wide plowing similar to those found by (Rajesh *et al.*, 2012). The estimated plow width was estimated at approximately 4 microns and peak-to-peak distance of 5 microns. The plowing is very deep which means the impact of asperities from the wear ball was high enough. There were also white loose particles observed from the micrograph which may be wear debris removed from the specimen surface during plowing. Similar effects were found on sample 7 which showed no or light fabricated plows. Sample 8 reveals that there were few troughs formed. The plowing was observed to be large at an approximate width of 30 microns and appears at very low numbers on the surface. The rest of the scar is similar to a polished surface. Finally, sample 9 wear scar was analysed and there was an indication of dry scratching as there were fine plow marks with a width of about 0.5 microns. In contrast, the scratches leave more space between the next and also, they were mild.

In summary of the wear scar analysis, the most severe wear was observed on sample 6 and this might have been because of the formation of large dendrites as indicated by the SEM imaging in Figure 5.10. Also, a fine dendritic structure is considered for retaining mechanical properties of which the structure here had very large laves instead. A lower indication of wear was observed at sample 1, 2, 5 and 7 with the remaining falling within the medium range. Sample 2 was earlier on found to have the lowest wear rate which may explained by the SEM imaging that shows a very fine dendritic structure is critical in providing improved mechanical properties (Che *et al.*, 2014; Ruan *et al.*, 2015).

The highest amount of wear recorded was found under sample 5 at values of 0.0133 mm³ and 0.037 mm³/Nm in terms of wear volume and wear rate respectively. The sample was fabricated from processing parameters of 7.5 g/min feed, 15 mm/sec scanning speed and 2 mm spot diameter. The least amount of wear recorded was sample 3 but the process parameters will be nullified at the optimisation stage because of the presence of cracks as well as those of sample 1 and 4. This is because cracks are the last defects to be considered on the functionality of clads. The second least

wear amounted to 0.00279 mm^3 and $0.00774 \text{ mm}^3/\text{Nm}$ for wear volume and specific wear rate respectively under sample 6. Sample 6 was achieved as a result of adjusting LC process parameters to 10 g/min powder feed, 25 mm/sec and 2 mm spot diameter. The significance of these variables still needs further statistical approaches to understand individual and combined contributions.

5.3.3 Correlation between microhardness and wear rate

As samples were tested for both hardness and wear, a graphical illustration (Figure 5.12) was used to observe the relationship between the two. It can be seen that there is a cluster of points that are skewed in an inverse direction. To further analyse the data, the Pearson's correlation method is used to check for the amount of correlation between the two attributes. A P- value less than 0.05 means there is correlation while a negative correlation coefficient means an inverse relation between the attributes and positive coefficient indicates a direct relation.

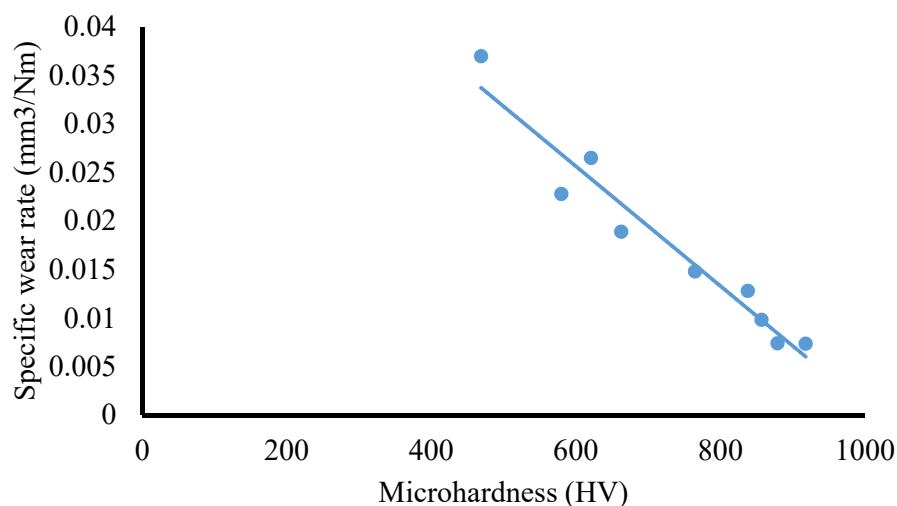


Figure 5.12: Specific wear rate versus microhardness.

Table 5.8 shows a p-value of 0 which indicates the existence of correlation between hardness and wear rate. In agreement with the graphical illustration Figure 5.12, the Pearson's coefficient is negative also implying the existence of an inverse relationship. The more closer the coefficient to +1 or -1 implies the correlation is very strong therefore, the hardness of the samples have a strong inverse relationship with

the specific wear rate. There is a higher chance of getting a lower wear rate from a harder sample.

Table 5.8: Pearson’s correlation coefficient

Correlations	
Pearson correlation	-0.97
P-value	0

5.4 Estimation of optimum parameters

The choice of optimum parameters in this study is premised on the ground that the deposited clads should be devoid of porosity and micro-cracks while the microstructure should be homogenous and characterised with fine dendrites which ensure a reasonable degree of high microhardness. All these microstructural characteristics are critical to the clads exhibiting minimum wear rate and COF. While Table 5.1 suggests a direct relationship between coating’s wear rate and microhardness. However, it is clear from Table 5.1 that a coating exhibiting highest microhardness and lowest wear rate may not have the lowest COF. Hence, it is important that optimum process parameters are selected in an attempted balance the interactions of processing issues with the quality characteristics. In this study, one of the objectives was to strengthen the LC clads by retaining an improved wear resistance performance and hardness at low coefficient of friction. The optimisation of these responses were found to be achievable with a feed rate of 5 g/min and scan speed of 5 mm/sec. The two parameters were significant against the microhardness and specific wear rate also considering the laser-material-interaction for this parameters will be adequate to provide fully dense clads. The spot diameter is considered to be fixed at 3 mm which was also attributed by the contour plotting that a lower coefficient of friction was achievable in that spot diameter range. The optimum parameters were attained from the multi response prediction under Table 5.9. The characteristics range for the 95% confidence interval and population interval were also shown.

Table 5.9. Multi response prediction.

Variable	Setting			
Feed rate (g/min)	5			
Scanning speed (mm/s)	5			
Spot diameter (mm)	3			

Response	Fit	SE Fit	95% CI	95% PI
Microhardness (HV)	885.6	33.3	(742.2, 1028.9)	(668.8, 1102.3)
Wear rate (mm ³ /Nm)	0.00849	0.00148	(0.00212, 0.01486)	(-0.00114, 0.01812)
COF	0.07296	0.00176	(0.06539, 0.08052)	(0.06152, 0.08439)

5.5 Confirmation test

A confirmatory test was carried out to establish if the optimum parameters were repeatable and also check if the responses were improved. The process settings followed were laser power (1800 watts), shielding gas (8.0 l/min), carrier gas (2.0 l/min), feed rate (5 g/min), scan speed (5 mm/sec) and spot diameter (3 mm). The SEM micrograph of the optimum sample is shown in Figure 5.13.

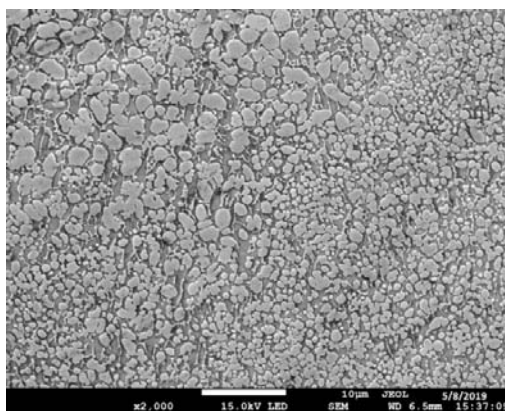


Figure 5.13. SEM image showing the microstructure of the confirmatory sample.

It is evident that the microstructure of the confirmatory sample was characterised with very fine dendritic structures with no defects visible. The outcome of analyses on the confirmatory sample revealed the following: 900 HV (microhardness), 0.0743 (COF) and 0.0071 mm³/N/m (specific wear rate) as shown in Table 5.10. These values were found to be within the 95% predicted confidence interval (CI) and 95% predicted population interval (PI) characteristics.

X-ray diffraction analysis (Figure 5.14) was carried out on the confirmatory sample and it is evident that the phases from the powder were retained without any addition of impurities or intermetallics. The pattern was characterized by 5 primary phases clearly Co based solid solution, Co (Cr) phase, Co₃Mo₂Si, CoMoSi and the eutectic lamellar phase which is Co₇Mo₆. The existence of the Co₃Mo₂Si phase implies the formation of the laves phase and a higher intensity means there is a higher concentration of the laves structure which provides a higher wear resistance for clads. The absence of FeCo phase there is no dilution effect from the process.

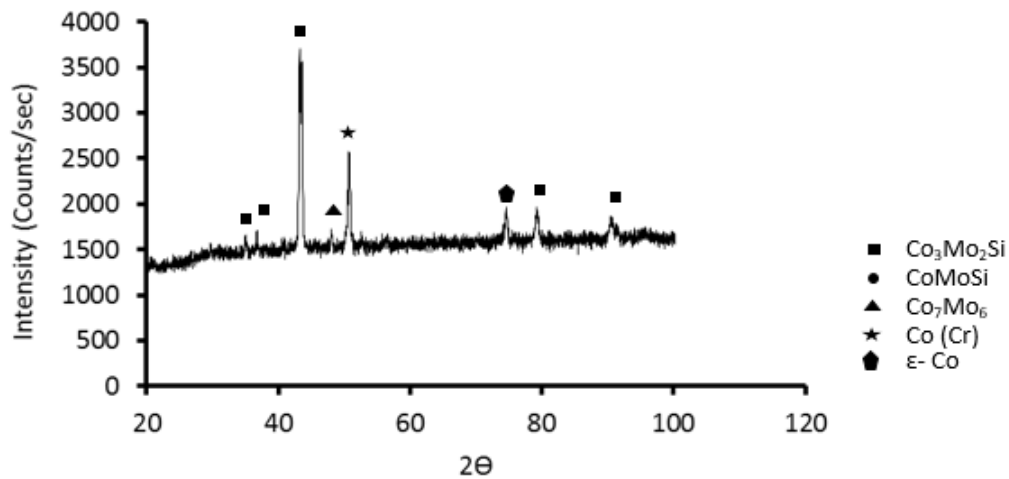


Figure 5.14. XRD pattern for the confirmatory sample.

Results from the confirmatory test were compared with those of the predicted model through error analysis (see Table 5.10). Error analysis was calculated as shown in equation 5.4.

$$Error (\%) = \frac{|\sigma_E - \sigma_P|}{\sigma_E} \times 100 \% \quad (5.4)$$

Where σ_E is the experimental values and σ_P is the predicted value. It can be confirmed from Table 5.10 that the predictive models have very low error percentages for the microhardness and coefficient of friction at 1.6% and 1.8% respectively. However, as for the specific wear rate the error is a bit higher at 19.6 % but the model is still reliable as all values fall within the 95% confidence index of the model.

Table 5.10: Comparison of experimental measurements against predicted models as obtained by multiple regression analysis

Cladding parameters: P = 1800 watts, S = 5 mm/s, F = 5 g/min, D = 3 mm	Experimental values (σ_E)	Predicted values (σ_P)	Error analysis (%)
Microhardness (HV)	900	885.6	1.6
Specific wear rate (mm³/Nm)	0.0071	0.00849	19.6
COF	0.0743	0.07296	1.8

5.6 Conclusion

In this study, the laser cladding optimisation of Tribaloy T-800 clads was successfully implemented. The cladding was run through L9 orthogonal array with 9 experimental runs. The samples were analysed and it was observed that sample 1, 3 and 4 were characterised by cracks and others had less amount of defects. Dry sliding wear test was conducted on the sample to obtain the COF and specific wear rate. In addition, measurement of microhardness was carried out on the samples. Furthermore, the Minitab 18 software was employed to optimise the T-800 clads for a greatest microhardness, lowest COF and lower wear rate. The responses for signal-to-noise ratios for COF gave optimum parameters as Feed rate = 7.5 g/min, Scanning speed = 5 mm/sec and Spot diameter = 3 mm. As for a higher microhardness, optimum parameters were found at a feed rate of 5 g/min, scanning speed of 5 mm/sec and a spot diameter of 4 mm. Wear rate was also found lower for the variables of 5 g/min feed rate, 5 mm/sec laser beam speed and 4 mm spot diameter. Multiple linear regression model on Minitab 18 was further used to obtain mathematical model

equations for LC process control factors based on specific wear rate, microhardness and COF of T-800 clads. Later on, a confirmatory test was conducted to verify the model and it was found out lower error was obtained (1.6%, 1.8% and 19.6% for microhardness, COF and wear rate respectively) when compared against the optimum predicted values. Sensitivity analysis was conducted and it was also discovered from this study that the CoF, specific wear rate and microhardness were highly sensitive towards the feed rate followed by the scanning speed and lastly the spot diameter. Furthermore, a correlation between the microhardness and wear rate was established as a direct inverse relationship in which an increment in hardness results in a reduction in wear rate. Microstructural observation and wear scar analysis was also investigated on the samples and it was found that the scar was characterised by plowing and micro-cutting of either mild or severe intensity. The most severe plowing was observed on sample 6 and the lowest among samples 1, 5 and 7. Moreover, the samples were then studied for microhardness, with the highest value recorded by sample 2 whereas sample 6 has the lowest hardness value. The estimated optimum parameters were predicted to be at a feed rate of 5 g//min, scan speed of 5 mm/sec and a spot diameter pf 3 mm. The responses obtained from the confirmatory test were found to be 900 HV, 0.0071 mm³/N/m and 0.0743 COF for the microhardness, specific wear rate and coefficient of friction respectively and these compare favourably well with the predicted values obtained from the model equations.

Chapter Six: Conclusions and Future work

6.0 Chapter summary: This chapter begins by re-introducing the aims/ objectives of the project. It further summarizes the findings from the research work in relation to the objectives of the study. Lastly, recommendations and future work are mentioned.

6.1 Conclusions

This research work began by stating the aims/objectives under chapter 1 of which were:

- v. Develop LC process maps for Stellite 6 and Tribaloy T800 clads with a view to understanding the effects of variation in process parameters on their consolidation behaviour.
- vi. Comparing the geometrical characteristics, microstructural properties and consolidation behaviour of the two powders with a view to identifying a suitable clad material for the substrate material (304 L austenitic Stainless steel) of which the slurry pump impeller is made of.
- vii. Determine the effects of process parameters on the microhardness, coefficient of friction and wear properties of the suitable clad material for the substrate material.
- viii. Optimise the quality characteristics (microhardness and wear properties) of the suitable clad material as process parameters are varied via Taguchi design of experiment (DoE).

6.1.1 Findings from this study

Main findings from this study are hereby presented as follows:

6.1.1.1 Consolidation behaviour of laser clad Stellite 6 and Tribaloy T800 clads

- i. The variation in process parameters has successfully provided the process map regions for the two metal alloys. The cobalt based alloys were found to each have 5 regions of consolidation behaviour. It was found that wide

tracks in terms of geometry were associated with low feed rates and scan speed with the influence of Marangoni convection currents that allows for the flow of material.

- ii. It was gathered from this study that the effects of the varying parameters was also found to have effects on the microstructure of the Stellite 6 and T800 clads which varied based on the morphology, size and distribution of the dendritic structures for ST6 and laves for T800.
- iii. The Vickers hardness test was carried out on the laser clad samples and it was discovered that the microhardness varied in relation to the process parameter variations.

6.1.1. 2 Comparison of the two cobalt based metal alloys (Stellite 6 and Tribaloy T800)

- i. 5 regions of consolidation behaviour for ST6 clads were found to be (1) Wide, thick, continuous and light grey track, (2) Narrow, thick, continuous and light grey track, (3) Wide, thick, continuous and Brown track, (4) Narrow, thick, discontinuous and/or discolored track and (5) Wide, thick and/or discontinuous and discolored while that for T800 was found to be (1) Wide, thick, continuous and silvery clads, (2) Narrow, thin, continuous and silvery clads, (3) Narrow, thick, discontinuous and discolored clads, (4) Narrow, thick, discontinuous and/or discolored clads and (5) Wide, thick, discontinuous and/or discolored clads.
- ii. The findings indicated that the microstructure between the cobalt based alloys differ in terms of microstructure. The ST6 clads were found to be characterised by dendrites and inter-dendritic structures while T800 clads were found to have eutectic laves phase and eutectic matrix of cobalt solid solution.
- iii. XRD phase analysis was conducted on the alloys and it was found that, Stellite 6 had 4 primary phases (1) $(\text{Cr, Fe})_7\text{C}_3$, (2) W_2C , (3) Co and (4) CrO phases whereas Tribaloy T800 have 5 primary phases (1) Co matrix, (2) Co_7Mo_6 , (3) Co (Cr) phase, (4) $\text{Co}_3\text{Mo}_2\text{Si}$ and (5) CoMoSi phases.
- iv. The microhardness of the two powders was investigated and it was discovered that, T800 samples (787.70 HV) showed a higher microhardness than those of ST6 which averaged at 492.54 HV.

- v. Varying of process parameters for the two metal alloys also gave variations on the microhardness and the wear rate. The two outcomes have an inverse relationship indicating that, an increase in microhardness also means a reduction in wear rate for both metal alloys.

6.1.2 Effects of process variables on the Coefficient of friction, specific wear rate and microhardness of T800 clads

- i. . It could be confirmed from the contour plots that, the COF was best minimised (<0.07) when laser beam spot diameter of 3 mm was employed in conjunction with feed rate above 9 g/min for LC deposition of the clads. Furthermore, undesirable high COF values were obtained when laser beam spot diameter other than 3 mm was employed for LC deposition irrespective of powder feed rate. In an interaction between the laser beam speed and spot diameter, it was seen that low COF (<0.07) was achieved when laser beam speed lower than 10 mm/sec was used. A spot diameter of 3 mm continued to give a lower COF compared to other diameters. It was further confirmed that, an interaction of high feed rate (>9 g/min) and low scan speed (<10 mm/sec) was responsible for very low COF (<0.07).
- ii. As for the secific wear rate, the interaction between the feed rate and spot diameter indicated that, a specific wear rate below 0.010 can be achieved by feed rates of 8 g/min and below whereas the spot diameter has to be a value other than of 3 mm. The interaction between the laser beam speed and spot diameter indicated that, low wear rates (<0.010) were achieved when scan speed is below 20 mm/sec while this occurred for all values of spot diameter. Lastly, an interaction of lower scan speed <15 mm/sec and feed rates <8 g/min gave low wear rates below $0.010 \text{ mm}^3/\text{Nm}$.
- iii. A sensitivity analysis was conducted between the outcomes (wear rate, microhardness and coefficient of friction) and the process parameters (feed rate, scan speed and laser beam diameter). It was discover that, the outcomes were highly sensitive towards the feed rate, scanning speed and spot diameter respectively in that order.
- iv. Finally, the effects on microhardness were investigated and it could be seen that microhardness values above 900 HV were achieved when the feed rate

was at 9 g/min and below, whereas the spot diameter has to be at 4 mm. As for the plot of scanning speed against spot diameter, scanning speeds below 20 mm/sec were responsible for >900 HV while spot diameter had to be at < 3.5 mm. Lastly, the scan speed continued to show lower significance to achieve a great hardness value when lower than 10 mm/sec while the feed rate has to be lower than 8 g/min.

6.1.3 Optimisation and modelling of T800 clads

- i. S/N ratios from Taguchi (DoE) were used to determine the optimum values of each variable on the investigated responses of Coefficient of friction, specific wear rate and microhardness of T800 clads.
- ii. The ANOVA was further used to test the significance of each variable towards the characterisation responses. The COF was found to be influence by spot diameter whereas microhardness and specific wear rate were found to be influenced by feed rate and scan speed.
- iii. Multi linear regression equations were obtained for the LC processing of T800 clads. The equations were found to e a s follows:
 - $COF = 0.0821 - 0.00117 F + 0.00113 S - 0.00028 D$
 - $Microhardness (HV) = 1127.1 - 136.8 F - 88.5 S + 51.0 D$
 - $Wear rate (mm^3 /Nm) = 0.00219 + 0.00873 F + 0.00523 S - 0.00336 D$

(5.3)

Where **F** = feed rate in g/min, **S** = scanning speed (mm/sec) and **D** = spot diameter (mm/sec).

- iv. Optimum process variables were finally obtained of which they are feed rate (5 g/min), scan speed (5 mm/sec) and spot diameter (3 mm) giving response range of microhardness at (742.2 - 1028.9 HV 95% confidence interval or 668.8 - 1102.3 HV 95% population interval), coefficient of friction at (0.06539 - 0.08052 confidence interval 95% or 0.06152 - 0.08439 population interval 95%) and specific wear rate at (0.00212 - 0.01486 mm³/Nm 95% confidence interval or 0.00114 - 0.01812 mm³/Nm 95% population interval). The fitted model values were 885.6 HV, 0.00849 mm³/Nm and 0.07296 which were compared against results from the confirmatory test for error analysis. The error was found to be 1.6%, 1.8% and 19.6% for microhardness, COF and wear rate respectively.

- v. A confirmatory test was carried out and the response analysis were found to be 900 HV, $0.0071 \text{ mm}^3/\text{N/m}$ and 0.0743 COF for the microhardness, specific wear rate and coefficient of friction respectively and these were found to compare well with the predicted optimum values.

6.2 Recommendation for future work

Even though most of the gap in knowledge has been investigated, further studies need to be carried in this work. The work is as follows and not limited to:

Models that relate to the exact and measured geometries need to be developed for the T800 clads. Most work carried out is associated with the functionality of the clads. Most researchers would be interested in the geometries and also the formation of multi-tracks and multi-layer clads so as to achieve industrial applications from the clads. The other work needed to be done is the corrosion study on the samples so that the applicability on slurry pump impeller which is exposed to corrosive media could be achieved. Furthermore, simulation and practical work has to be understood for the clad deposition to refurbish complex geometries of the pump impellers. The deposition mechanisms suitable to carry out the refurbishment need to be investigated in order to select the suitable one as there are many approaches in place investigated by other researchers, such methods as mesh deposition, chess pattern deposition e. t. c.

This study shows the feasibility of laser cladding as it has been found that the clads with low defects and low specific wear rate were successfully fabricated. Further refurbishment on the slurry pump impellers is what needs to be done and the functionality of the component investigated.

List of References

- Addie, G. R., Roudnev, A. S., & Sellgren, A. (2007). The new ANSI/HI centrifugal slurry pump standard. *Journal of the Southern African Institute of Mining and Metallurgy*, 107(6), 403–409.
- Amin, S., Panchal, H., & Professor, A. (2016). A Review on Thermal Spray Coating Processes. In *International Journal of Current Trends in Engineering & Research Scientific Journal Impact Factor* (Vol. 2, pp. 556–563). Retrieved from <http://www.ijcter.com>
- Antony, K., & Arivazhagan, N. (2015). Studies on energy penetration and marangoni effect during laser melting process. *Journal of Engineering Science and Technology*, 10(4), 509–525.
- Arrizubieta, J. I., Ruiz, J. E., Martinez, S., Ukar, E., & Lamikiz, A. (2017). Intelligent nozzle design for the Laser Metal Deposition process in the Industry 4.0. *Procedia Manufacturing*, 13, 1237–1244. <https://doi.org/10.1016/j.promfg.2017.09.043>
- Aucott, L. (2017). *Mechanism of solidification cracking during welding of high strength steels for subsea linepipe of High Strength Steels for Subsea Linepipe*. Universiter of Leceister.
- Awasthi, R., Kumar, S., Srivastava, D., & Dey, G. K. (2010). Solidification and microstructural aspects of laser-deposited Ni-Mo-Cr-Si alloy on stainless steel. *Pramana - Journal of Physics*, 75(6), 1259–1266. <https://doi.org/10.1007/s12043-010-0215-4>
- Bajwa, R., Khan, Z., Nazir, H., Chacko, V., & Saeed, A. (2016). Wear and friction properties of electrodeposited Ni-based coatings subject to nano-enhanced lubricant and composite coating. *Acta Metallurgica Sinica (English Letters)*, 29(10), 902–910. <https://doi.org/10.1007/s40195-016-0470-6>
- Baldenebro-Lopez, F. J., Gomez-Esparza, C. D., Corral-Higuera, R., Arredondo-Rea, S. P., Pellegrini-Cervantes, M. J., Ledezma-Sillas, J. E., ... Herrera-Ramirez, J. M. (2015). Influence of size on the microstructure and mechanical properties of an AISI 304L stainless steel-a Comparison between bulk and fibers. *Materials*, 8(2), 451–461. <https://doi.org/10.3390/ma8020451>
- Barekat, M., Shoja, R., & Ghasemi, A. (2016). Optics & Laser Technology Nd : YAG laser cladding of Co – Cr – Mo alloy on γ -TiAl substrate. *Optics and Laser Technology*, 80, 145–152. <https://doi.org/10.1016/j.optlastec.2016.01.003>
- Barnes, N., Joseph, T., & Mendez, P. F. (2015). Issues associated with welding and surfacing of large mobile mining equipment for use in oil sands applications. *Science and Technology of Welding and Joining*, 20(6), 483–493. <https://doi.org/10.1179/1362171815Y.0000000060>
- Bascetin, A., Tuylu, S., Adiguzel, D., & Ozdemir, O. (2017). *Field properties and performance of surface paste disposal. Paste Tailings Management*. Springer, Cham. https://doi.org/10.1007/978-3-319-39682-8_6
- Bayer, Raymond, G. (2004). *Mechanical wear fundamentals and testing*. Marcel Dekker Inc., USA (2nd ed.). New York: Taylor & Francis Inc. <https://doi.org/doi:10.1201/9780203021798.fmatt>
- Beuth, J., Fox, J., Gockel, J., Montgomery, C., Yang, R., Qiao, H., ... Klingbeil, N. (2013). Process Mapping for Qualification Across Multiple Direct Metal Additive Manufacturing Processes. In *Solid freeform fabrication proceedings*

- (pp. 655–665). Austin: Solid Freeform Fabrication Symposium.
- Bishop, C. A. (2015). Chapter 11 – Polymer Coating Basic Information. In *Vacuum Deposition Onto Webs, Films and Foils* (pp. 221–239). <https://doi.org/10.1016/B978-0-323-29644-1.00011-6>
- Bloch, Heinz, P., & Budris, Allan, R. (2014). *Pump User's Handbook: Life Extension* (4th ed.). Lilburn, GA: The Fairmont Press Inc.
- Borges, B., Quintino, L., Miranda, R. M., & Carr, P. (2010). Imperfections in laser cladding with powder and wire fillers. *International Journal of Advanced Manufacturing Technology*, 50(1–4), 175–183. <https://doi.org/10.1007/s00170-009-2480-2>
- Cavaliere, P., & Silvello, A. (2014). Processing parameters affecting cold spray coatings performances. *International Journal of Advanced Manufacturing Technology*, 71(1–4), 263–277. <https://doi.org/10.1007/s00170-013-5465-0>
- Che, L., Sun, W., Huang, Y., & Chao, Y. (2014). Laser cladding technology in the repair of shearer picks. *COMPUTER MODELLING & NEW TECHNOLOGIES*, 18(12A), 563–565.
- Chong, K., Zhang, H., Xiao, G., Xu, H., & Zhao, W. (2018a). Effects of initial powder size on microstructure and corrosion resistance of in situ (Ti-V)C reinforced Fe-based laser cladding layers. *International Journal of Electrochemical Science*, 13(7), 6858–6869. <https://doi.org/10.20964/2018.07.27>
- Chong, K., Zhang, H., Xiao, G., Xu, H., & Zhao, W. (2018b). Effects of Initial Powder Size on Microstructure and Corrosion Resistance of in situ (Ti-V)C Reinforced Fe-based Laser Cladding Layers. *International Journal of Electrochemical Science*, 13, 6858–6869.
- Chrysosolouris, G., Zannis, S., Tsirbas, K., & Lalas, C. (2002). An experimental investigation of laser cladding. *CIRP Annals - Manufacturing Technology*, 51(1), 145–148. [https://doi.org/10.1016/S0007-8506\(07\)61486-3](https://doi.org/10.1016/S0007-8506(07)61486-3)
- Cooke, R. (2007). Thickened and Paste Tailings Pipeline Systems : Design Procedure – Part 2. In *International seminars on Paste and Thickened Tailings*. Perth, Australia.
- Davis, J. R. (2004). *Thermal Spray Technology Edited by. (R. Davis, Joseph, Ed.)* (Illustrate). ASM International.
- de La Batut, B., Fergani, O., Brotan, V., Bambach, M., & El Mansouri, M. (2017). Analytical and Numerical Temperature Prediction in Direct Metal Deposition of Ti6Al4V. *Journal of Manufacturing and Materials Processing*, 1(1), 3. <https://doi.org/10.3390/jmmp1010003>
- Drezet, J., Pellerin, S., Bezençon, C., & Mokadem, S. (2004). Modelling the Marangoni convection in laser heat treatment. *Journal of Physics*, 120(4), 299–306.
- Espallargas, N. (2015). *Introduction to thermal spray coatings. Future Development of Thermal Spray Coatings: Types, Designs, Manufacture and Applications*. New York, USA: Elsevier Ltd. <https://doi.org/10.1016/B978-0-85709-769-9.00001-4>
- Faravar, A. (2014). *Design , Implementation and Control of a Robotic Arm Using PIC 16F877A Microcontroller*. Eastern Mediterranean University.
- Fu, F., Zhang, Y., Chang, G., & Dai, J. (2016). Optik Analysis on the physical mechanism of laser cladding crack and its influence factors. *Optik - International Journal for Light and Electron Optics*, 127(1), 200–202. <https://doi.org/10.1016/j.ijleo.2015.10.043>

- Gaebler, B. F., Riedelsberger, H., & Gmbh, C. (2013). New Laser Cladding Technology Provides Throughput, Precision, and True Metallurgic Bonding, *20*(2).
- Gedda, H. (2004). *Laser Cladding : An Experimental and Theoretical Investigation. PhD Thesis, Luleå University of Technology, 2004.*
- Ghelichi, R., & Guagliano, M. (2009). Coating by the Cold Spray Process : A state of the art. *Frattura Ed Integrità Strutturale*, *8*, 30–44.
<https://doi.org/10.3221/IGF-ESIS.08.03>
- Gomez, V. A. O., de Macêdo, M. C. S., Souza, R. M., & Scandian, C. (2015). Effect of abrasive particle size distribution on the wear rate and wear mode in micro-scale abrasive wear tests. *Wear*, *328–329*, 563–568.
<https://doi.org/10.1016/j.wear.2015.03.015>
- Goodarzi, D. M., Pekkarinen, J., & Salminen, A. (2015). Effect of process parameters in laser cladding on substrate melted areas and the substrate melted shape. *Journal of Laser Applications*, *27*(S2), S29201.
<https://doi.org/10.2351/1.4906376>
- Grujicic, M., Tong, C., DeRosset, W. S., & Helfritch, D. (2003). Flow analysis and nozzle-shape optimization for the cold-gas dynamic-spray process. *Proceedings of the Institution of Mechanical Engineers, Part B: Journal of Engineering Manufacture*, *217*(11), 1603–1613.
<https://doi.org/10.1243/095440503771909980>
- Gu, D. (2015a). Laser Additive Manufacturing (AM): Classification , Processing Philosophy , and Metallurgical Mechanisms. In D. Gu (Ed.), *Laser Additive Manufacturing of High-Performance Materials* (Illustrate, p. 311). Berlin: Springer. <https://doi.org/10.1007/978-3-662-46089-4>
- Gu, D. (2015b). Laser additive manufacturing of high-performance materials. *Laser Additive Manufacturing of High-Performance Materials*, (Lm), 1–311.
<https://doi.org/10.1007/978-3-662-46089-4>
- Gu, D., Wang, H., Dai, D., Chang, F., Meiners, W., Hagedorn, Y.-C., ... Poprawe, R. (2015). Densification behavior, microstructure evolution, and wear property of TiC nanoparticle reinforced AlSi10Mg bulk-form nanocomposites prepared by selective laser melting. *Journal of Laser Applications*, *27*(S1), S17003.
<https://doi.org/10.2351/1.4870877>
- Gupta, D., Mordike, B. L., Shariff, S., & Padmanabhan, G. (2010). Laser Surface Cladding of EN19 Steel with Stellite 6 for Improved Wear Resistance. *Interface*, *19*, 317–330.
- Hasan, M. S., Mazid, A. M., & Clegg, R. (2017). The Basics of Stellites in Machining Perspective. *International Journal of Engineering Materials and Manufacture*, *1*(2), 35. <https://doi.org/10.26776/ijemm.01.02.2016.01>
- Higgins, T. V. (1995). The Three Phases of Lasers: Solid-state, Gas, and Liquid. *Laser Focus World*, *31*(7), 73–85.
- Hrabovsky, M. (2009). Thermal Plasma Generators with Water Stabilized Arc. *The Open Plasma Physics Journal*, *2*, 99–104.
<https://doi.org/10.2174/1876534300902020099>
- Hsiao, W.-Y., Wang, S.-H., Chen, C.-Y., Yang, J.-R., & Lee, W.-S. (2008). Effects of dynamic impact on mechanical properties and microstructure of special stainless steel weldments. *Materials Chemistry and Physics*, *111*(1), 172–179.
<https://doi.org/10.1016/j.matchemphys.2008.04.004>
- Huang, F. X., Jiang, Z. H., Liu, X. M., Lian, J. S., & Chen, L. (2011). Effects of Process Parameters on Microstructure and Hardness of Layers by Laser

- Cladding. *ISIJ International*, 51(3), 441–447.
<https://doi.org/10.2355/isijinternational.51.441>
- Irissou, E., Legoux, J.-G., Ryabinin, A. N., Jodoin, B., & Moreau, C. (2008). Review on Cold Spray Process and Technology: Part I—Intellectual Property. *Journal of Thermal Spray Technology*, 17(4), 495–516.
<https://doi.org/10.1007/s11666-008-9203-3>
- Joseph, G. B., Mageshwaran, G., Kumar, R. A., Raj, R. B. D., Jeevahan, J., & Kunjan, M. (2016). Characteristics studies of stainless steel (AISI type 304L) welded by ER310L filler using tig welding. *International Journal of Chemical Sciences*, 14(4), 2527–2534.
- Jr. Tucker, R. C. (1993). Thermal Spray Coatings. In *Handbook Surface Engineering* (Vol. 5, pp. 1446–1471). ASM International.
<https://doi.org/10.1007/s11548-011-0634-9>
- Kaiser, R., Browne, D. J., & Williamson, K. (2011). Investigation of the effects of cooling rate on the microstructure of investment cast biomedical grade Co alloys. In *IOP Conference Series: Materials Science and Engineering* (Vol. 27). <https://doi.org/10.1088/1757-899X/27/1/012071>
- Kansake, B. A., & Suglo, R. S. (2015). Impact Of Availability And Utilisation Of Drill Rigs On Production At Kanjjole Minerals Limited, 4(6), 1524–1537.
- Karman, G. P., McDonald, G. S., New, G. H. C., & Woerdman, J. P. (1999). Fractal modes in unstable resonators. *Nature*, 402(6758), 138.
<https://doi.org/10.1038/45960>
- Kato, K., & Adachi, K. (2001). Wear Mechanisms. In *Modern Tribology Handbook. Vol 1* (p. 28). CRC Press.
<https://doi.org/10.1201/9780849377877.ch7>
- Kausar, A. (2018). Polymer coating technology for high performance applications: Fundamentals and advances. *Journal of Macromolecular Science, Part A: Pure and Applied Chemistry*. <https://doi.org/10.1080/10601325.2018.1453266>
- Kinder, J. (2003). Corrosion Books: Metallographer's Guide - Practices and Procedures for Irons and Steels. By: Bruce L. Branfitt, Arlan O. Benscoter - Materials and Corrosion 6/2003. *Materials and Corrosion*, 54(6), 468–469.
<https://doi.org/10.1002/maco.200390105>
- Kivisäkk, U. (2010). *Influence of hydrogen on corrosion and stress induced cracking of stainless steel*. KTH Royal Institute of Technology.
- Ko, M., Sk, M. H., Laycock, N., Ryan, M. P., Abdullah, A. M., Williams, D. E., & Ingham, B. (2017). Effect of Cr/Mo on the Protectiveness of Corrosion Scales on Carbon Steel in Sweet Medium under High Flow Regime. *ECS Transactions*, 80(10), 509–517. <https://doi.org/10.1149/08010.0509ecst>
- Kou, S., Limmaneevichitr, C., & Wei, P. S. (2011). Oscillatory Marangoni flow: A fundamental study by conduction-mode laser spot welding. *Welding Journal*, 90(12), 229. Retrieved from
<http://connection.ebscohost.com/c/articles/67336515/oscillatory-marangoni-flow-fundamental-study-by-conduction-mode-laser-spot-welding>
- Kumarasamy, M., & Natarajan, S. (2016). Selection and characterisation of HVOF cermet coatings for volutes of high capacity pumps of opencast lignite mines. *Surface Engineering*, 32(3), 229–237.
<https://doi.org/10.1179/1743294415Y.0000000015>
- Kusmoko, A. (2016). Effect of laser power and steel and nickel-based alloy substrates on hardness and wear resistance of laser-deposited stellite coatings, 143.

- Kusmoko, A., Dunne, D., Li, H., & Nolan, D. (2014). Effect of Two Different Energy Inputs for Laser Cladding of Stellite 6 on P91 and P22 Steel Substrates. *Procedia Materials Science*, 6, 26–36. <https://doi.org/10.1016/j.mspro.2014.07.005>
- Laschkow, A., Lauffs, A., Weingaertner, Walter, L., Pereira, M., Gutjahr, J., & Pereira, Adriano de Souza, P. (2017). Wire and powder laser cladding of Inconel 625. *Engineering for a Changing World*, 45(6).
- Li, C. C. (1980). Characterization of thermally sprayed coatings for high temperature wear protection applications. *Thin Solid Films*, 73(1), 59–77. [https://doi.org/10.1016/0040-6090\(80\)90329-6](https://doi.org/10.1016/0040-6090(80)90329-6)
- Limmaneevichitr, C., & Kou, S. (2000). Visualization of Marangoni convection in simulated weld pools containing a surface-active agent. *Welding Journal*, 79(11), 324s–330s.
- Liu, G., Xu, Y. L., Yang, C. X., Xiao, X. S., Chen, X. M., Zhang, X. K., & Meng, X. J. (2015). Effects of alloy elements on oxidation resistance and stress-rupture property of P92 steel. *Acta Metallurgica Sinica (English Letters)*, 28(2), 129–138. <https://doi.org/10.1007/s40195-014-0172-x>
- Liu, J., Yu, H., Chen, C., Weng, F., & Dai, J. (2017). Research and development status of laser cladding on magnesium alloys: A review. *Optics and Lasers in Engineering*. <https://doi.org/10.1016/j.optlaseng.2017.02.007>
- Loto, R. T. (2017). Study of the corrosion resistance of type 304L and 316 austenitic stainless steels in acid chloride solution. *Oriental Journal of Chemistry*, 33(3), 1090–1096. <https://doi.org/10.13005/ojc/330304>
- Lupoi, R., Sparkes, M., Cockburn, A., & O'Neill, W. (2011). High speed titanium coatings by supersonic laser deposition. *Materials Letters*, 65(21–22), 3205–3207. <https://doi.org/10.1016/j.matlet.2011.07.014>
- MacEwan, J. R., MacEwan, J. U., & Yaffe, L. (2006). DIFFUSION OF Ni 63 IN IRON, COBALT, NICKEL, AND TWO IRON–NICKEL ALLOYS . *Canadian Journal of Chemistry*, 37(10), 1629–1636. <https://doi.org/10.1139/v59-237>
- Mahamood, R. M., & Akinlabi, E. T. (2017). Scanning Speed Influence on the Microstructure and Micro hardness Properties of Titanium Alloy Produced by Laser Metal Deposition Process. In *Materials Today: Proceedings* (Vol. 4, pp. 5206–5214). <https://doi.org/10.1016/j.matpr.2017.05.028>
- Mahbub, H. (2005). *High-Velocity Oxy-Fuel (HVOF) Thermal Spray Deposition of Functionally Graded Coatings*. Dublin City University.
- Mathur, S. (2012). A Simple Weather Forecasting Model Using Mathematical Regression, I (Volume I), 161–168.
- Mcdonald, G. (2015). Analysis of Thermal Spray in the industries of Western Canada. In *ASM International*. Edmonton, Canada: ASM International.
- Mishra, D. P., Sugla, M., & Singha, P. (2013). Productivity Improvement In Underground Coal Mines – A Case Study. *Journal of Sustainable Mining*, 12(3), 48–53. <https://doi.org/10.7424/jsm130306>
- Moolman, P. L., & Vietti, A. (2012). Tailings disposal: an approach to optimize water and energy efficiency. *The Southern African Institute of Mining and Metallurgy. Platinum*, 767–780.
- Morville, S., Carin, M., Peyre, P., Gharbi, M., Carron, D., Le Masson, P., & Fabbro, R. (2012). 2D longitudinal modeling of heat transfer and fluid flow during multilayered 2D longitudinal modeling of heat transfer and fluid flow during multilayered. *Journal of Laser Appli-Cations*, 24(3), 1–9.

- Mukhuthu, Z. (2014, June 20). Hope springs (not) eternal in spat over toxic sludge. *Mail & Guardian*. Retrieved from <https://mg.co.za/article/2014-06-19-hope-springs-not-eternal-in-spat-over-toxic-sludge>
- Myers, D. (2002). Friction, Lubrication, and Wear. In *Surfaces, Interfaces, and Colloids* (pp. 448–475). <https://doi.org/10.1002/0471234990.ch18>
- Naeem, M. (2013). Laser Processing of Reflective Materials. *Laser Technik Journal*, 10(1), 18–20. <https://doi.org/doi:10.1002/latj.201390001>
- Navas, C., Cadenas, M., Cuetos, J. M., & de Damborenea, J. (2006). Microstructure and sliding wear behaviour of Tribaloy T-800 coatings deposited by laser cladding. *Wear*, 260(7–8), 838–846. <https://doi.org/10.1016/j.wear.2005.04.020>
- Navas, C., Colaço, R., Damborenea, J. De, & Vilar, R. (2006). Abrasive wear behaviour of laser clad and flame sprayed-melted NiCrBSi coatings, 200, 6854–6862. <https://doi.org/10.1016/j.surfcoat.2005.10.032>
- Nsoesie, S., Liu, R., Jiang, K., & Liang, M. (2013). High-temperature Hardness and Wear Resistance of Cobalt-based Tribaloy Alloys. *Www.ijm-me.org International Journal of Material and Mechanical Engineering*, 2(3), 48–56.
- Okechukwu, C., Dahunsi, O. A., Oke, P. K., Oladele, O. I., & Dauda, M. (2017). Review on Hardfacing As Method of Improving the Service Life of Critical Components Subjected To Wear in Service. *Nigerian Journal of Technology*, 36(4), 1095–1103.
- Olakanmi, E. O., & Doyoyo, M. (2014). Laser-assisted cold-sprayed corrosion- and wear-resistant coatings: A review. *Journal of Thermal Spray Technology*. <https://doi.org/10.1007/s11666-014-0098-x>
- Olakanmi, E. O., Nyadongo, S. T., Malikongwa, K., Lawal, S. A., Botes, A., & Pityana, S. L. (2019). Multi-variable optimisation of the quality characteristics of fiber-laser clad Inconel-625 composite coatings. *Surface and Coatings Technology*, 357, 289–303. <https://doi.org/https://doi.org/10.1016/j.surfcoat.2018.09.063>
- Pangsrivini, S. (2016). *High Throughput Functional Material Deposition*. CASE WESTERN RESERVE UNIVERSITY.
- Parekh, R., Buddu, R. K., & Patel, R. I. (2016). Multiphysics Simulation of Laser Cladding Process to Study the Effect of Process Parameters on Clad Geometry. *Procedia Technology*, 23, 529–536. <https://doi.org/10.1016/j.protcy.2016.03.059>
- Paschotta, R. (2008). *Encyclopedia of Laser Physics and Technology*. *Laser Physics* (Vol. 1). Retrieved from <http://www.rp-photonics.com/encyclopedia.html>
- Piasecki, A., & Bartkowski, D. (2013). Laser cladding of Stellite 6 on low carbon steel for repairing components in automotive applications using disk laser. *Archives of Mechanical Technology and Automation*, 33(2), 2–11. Retrieved from http://atmia.put.poznan.pl/Woluminy/Fil/ATMiA_33_2_3.pdf
- Pogorelsky, I. V. (2004). Gas Lasers for Strong Field Applications. In *AIP Conference Proceedings* (pp. 109–124). <https://doi.org/10.1063/1.1842538>
- Przybyłowicz, J., & Kusinski, J. (2002). Laser cladding and erosive wear of Co–Mo–Cr–Si coatings. *Surface and Coatings Technology*, 125(1–3), 13–18. [https://doi.org/10.1016/s0257-8972\(99\)00563-0](https://doi.org/10.1016/s0257-8972(99)00563-0)
- Ragoth Singh, R., & Nataraj, M. (2012). Parametric study and optimization of pump impeller by varying the design parameter using computational fluid dynamics. *International Review of Mechanical Engineering*, 6(7), 1581–1585.

- Rajesh, S., Rajakarunakaran, S., & Pandian, R. S. (2012). Modeling and optimization of sliding specific wear and coefficient of friction of aluminum based red mud metal matrix composite using taguchi method and response surface methodology, *15*, 150–166.
- Ruan, Y., Chang, S. Y., & Dao, M. (2015). Comparative Effect of Rapid Dendrite Growth and Element Addition on Microhardness Enhancement of Fe-Based Alloys. *Crystal Growth and Design*, *15*(12), 5661–5664. <https://doi.org/10.1021/acs.cgd.5b01167>
- Rulli, S. (2006). Cobalt Coating in High Temperature Aircraft Engine, 1–11.
- Salleh, M. S., Ramli, M. I., Yahaya, S. H., Engineering, M., Teknikal, U., & Durian, P. P. (2011). Study On Mechanical Properties And Microstructure Analysis Of Aisi 304l Stainless Steel Weldments On Mechanical Properties And Microstructure Analysis Of Aisi 304l Stainless Steel Weldments. *Journal of Mechanical Engineering and Technology*, *3*(2).
- Sawyer, W. G., Argibay, N., Burris, D. L., & Krick, B. A. (2014). Mechanistic Studies in Friction and Wear of Bulk Materials. *Annual Review of Materials Research*, *44*(1), 395–427. <https://doi.org/10.1146/annurev-matsci-070813-113533>
- Schneider, M. (1998). *Laser cladding with powder effect of some machining parameters on clad properties. Twente (Netherlands), University, Dissetation*. Retrieved from <http://scholar.google.com/scholar?hl=en&btnG=Search&q=intitle:LASER+CLADDING+WITH+POWDER#9>
- Sellgren, A., Addie, G., Visintainer, R., & Pagalthivarthi, K. (2005). Prediction of slurry pump component wear and cost. *Annual Texas A&M Dredging Seminar*. Retrieved from [http://pure.ltu.se/portal/en/publications/prediction-of-slurry-pump-component-wear-and-cost\(ae7d46f0-9782-11db-8975-000ea68e967b\).html](http://pure.ltu.se/portal/en/publications/prediction-of-slurry-pump-component-wear-and-cost(ae7d46f0-9782-11db-8975-000ea68e967b).html)
- Shankarling, G. S., & Jarag, K. J. (2010). Laser dyes. *Resonance*, *15*(9), 804–818. <https://doi.org/10.1007/s12045-010-0090-9>
- Shebani, A., & Pislaru, C. (2015). Wear Measuring and Wear Modelling Based on Archard , ASTM , and Neural Network Models. *International Journal of Mechanical, Aerospace, Industrial and Mechatronics Engineering*, *9*(1), 177–182.
- Siegman, A. E. (2000). Laser beams and resonators: The 1960s. *IEEE Journal on Selected Topics in Quantum Electronics*, *6*(6), 1380–1388. <https://doi.org/10.1109/2944.902192>
- Singh, L., Chawla, V., & Grewal, J. S. (2012). A Review on Detonation Gun Sprayed Coatings. *Journal of Minerals and Materials Characterization and Engineering*, *11*(3), 243–265. <https://doi.org/10.4236/jmmce.2012.113019>
- Sun, J., Sheng, C., Wang, D. P., Zhao, J., Zhang, Y. H., Zhong, H. G., ... Zhai, Q. jie. (2018). Fine equiaxed dendritic structure of a medium carbon steel cast using pulsed magneto-oscillation melt treatment. *Advances in Manufacturing*, *6*(2), 189–194. <https://doi.org/10.1007/s40436-017-0206-5>
- Tadamalle, A. P., Reddy, Y. P., Ramjee, E., & Reddy, V. (2014). Evaluation of Nd: YAG Laser Welding Efficiencies for 304L Stainless Steel. *Procedia Materials Science*, *6*, 1731–1739. <https://doi.org/10.1016/j.mspro.2014.07.160>
- Torims, T. (2013). *The Application of Laser Cladding to Mechanical Component Repair, Renovation and Regeneration. DAAAM International Scientific Book*. <https://doi.org/10.2507/daaam.scibook.2013.32>

- Vardelle, A., Moreau, C., Themelis, N. J., & Chazelas, C. (2015). A Perspective on Plasma Spray Technology. *Plasma Chemistry and Plasma Processing*, 35(3), 491–509. <https://doi.org/10.1007/s11090-014-9600-y>
- Villafuerte, J. (2010). Current and future applications of cold spray technology. *Metal Finishing*, 108(1), 37–39. [https://doi.org/10.1016/S0026-0576\(10\)80005-4](https://doi.org/10.1016/S0026-0576(10)80005-4)
- Wang, D., Hu, Q., & Zeng, X. (2015). Residual stress and cracking behaviors of Cr13Ni5Si2 based composite coatings prepared by laser-induction hybrid cladding. *Surface and Coatings Technology*, 274, 51–59. <https://doi.org/10.1016/j.surfcoat.2015.04.035>
- Wang, D., Hu, Q., Zheng, Y., Xie, Y., & Zeng, X. (2016). Optics & Laser Technology Study on deposition rate and laser energy efficiency of Laser-Induction Hybrid Cladding. *Optics and Laser Technology*, 77, 16–22. <https://doi.org/10.1016/j.optlastec.2015.08.019>
- Weber, E. M. J. (1999). *Handbook of Laser Wavelength*. (E. M. J. Weber, Ed.) (1st ed.). CRC Press.
- Weman, K. (2011). *Welding Processes Handbook*. (K. Weman, Ed.), Woodhead Publishing Ltd (2nd ed.). United Kingdom: Woodhead Publishing Ltd. <https://doi.org/10.1533/9780857095183.105>
- Wills, B. A., & Napier-munn, T. (2006). *Mineral processing technology* (7th ed.). Elsevier science and technology books.
- Xu, M., Li, J., Jiang, J., & Li, B. (2015). Influence of powders and process parameters on bonding shear strength and micro hardness in laser cladding remanufacturing. *Procedia CIRP*, 29, 804–809. <https://doi.org/10.1016/j.procir.2015.02.088>
- Yanqin, F., Xiang, L., & Ruijie, Y. (2018). The Surface Modification Methods for Constructing Polymer-Coated Stents. *International Journal of Polymer Science*, 2018(3891686), 7.
- Yin, S., Cavaliere, P., Aldwell, B., Jenkins, R., Liao, H., Li, W., & Lupoi, R. (2018). Cold spray additive manufacturing and repair: Fundamentals and applications. *Additive Manufacturing*. <https://doi.org/10.1016/j.addma.2018.04.017>
- Yu, H., Ahmed, R., Lovelock, H. de V., & Davies, S. (2009). Influence of Manufacturing Process and Alloying Element Content on the Tribomechanical Properties of Cobalt-Based Alloys. *Journal of Tribology*, 131(1), 11601. <https://doi.org/10.1115/1.2991122>
- Zekovic, S., Dwivedi, R., & Kovacevic, R. (2007). Numerical simulation and experimental investigation of gas-powder flow from radially symmetrical nozzles in laser-based direct metal deposition. *International Journal of Machine Tools and Manufacture*, 47(1), 112–123. <https://doi.org/10.1016/j.ijmachtools.2006.02.004>
- Zhang, X., You, W. M., Zhou, Y. J., Li, Y. S., & Qiu, S. J. (2013). Analysis on the Corrosion Failure of MDS Mine Multistage Centrifugal Pump Impeller. *Advanced Materials Research*, 650, 344–349. <https://doi.org/10.4028/www.scientific.net/AMR.650.344>
- Zimmermann, G., Sturz, L., Walterfang, M., & Dagner, J. (2009). Effect of melt flow on dendritic growth in AlSi7-based alloys during directional solidification. *International Journal of Cast Metals Research*, 22(1–4), 335–338. <https://doi.org/10.1179/136404609x368154>

Appendices

Appendix A: Research equipment and software employed in this study.

Equipment/Software	Tests/Activities conducted
Eskom IPG YLS 5000 Fire laser system	Laser cladding sample generation
5 axis Kuka robotic arm	
Siemens GTV PF 2/2 multi-hopper system	
Operator's remote control	
Struers Labotom-15 metal cutting machine	Cutting and resizing of samples
Struers 50A35 disk	
Struers CitoPress-5 mounting machine	Mounting of samples
MRC M-1B grinding and polishing machine	Grinding and polishing of sample surfaces
1.5V Frederiksen Direct current (DC) power supply	Electrolytic etching
stainless steel electrodes	
JSM-7100F Scanning electron microscope (SEM) installed with Energy Dispersive X-Ray Spectroscopy (EDS)	Analysis of chemical composition on samples, Microstructural analysis
Innovatest 4000 series tester	Microhardness test
Rtec tribometer installed with a computer based Rtec MFT17 software	Wear test
Minitab 18 software	Statistical analysis

Appendix B: Summary of Experimental work conducted

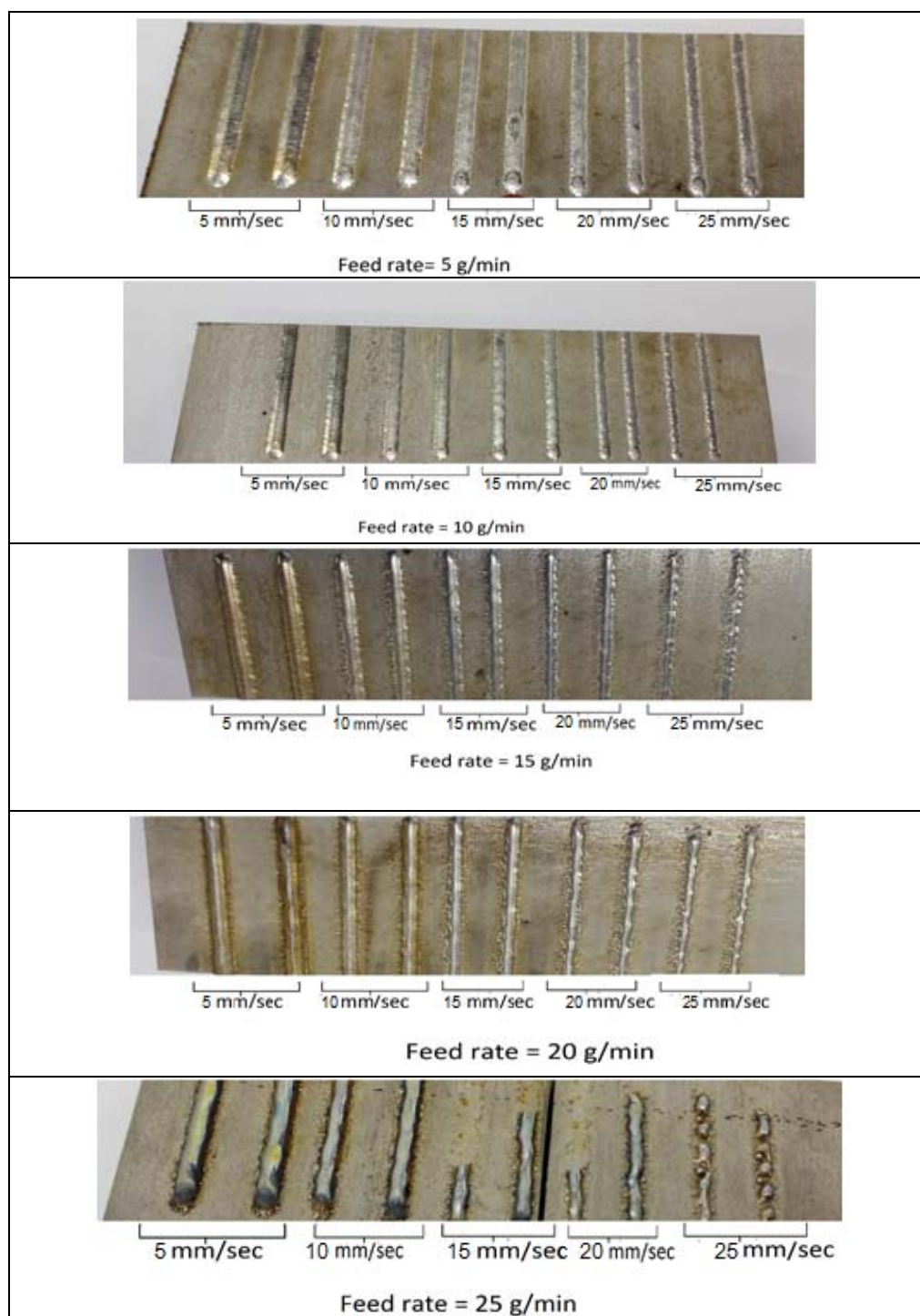
Appendix B1: Preliminary Experiment

Process variables	Levels of parameters				
	1	2	3	4	5
Powder feeding rate (g/min)	5	10	15	20	25
Scanning speed (mm/sec)	5	10	15	20	25
	Fixed parameters				
Stand-off distance (mm)	12				
Laser power (Watts)	1800				
Spot size (mm)	4				
Carrier gas (L/min)	2				
Shielding gas (L/min)	8				

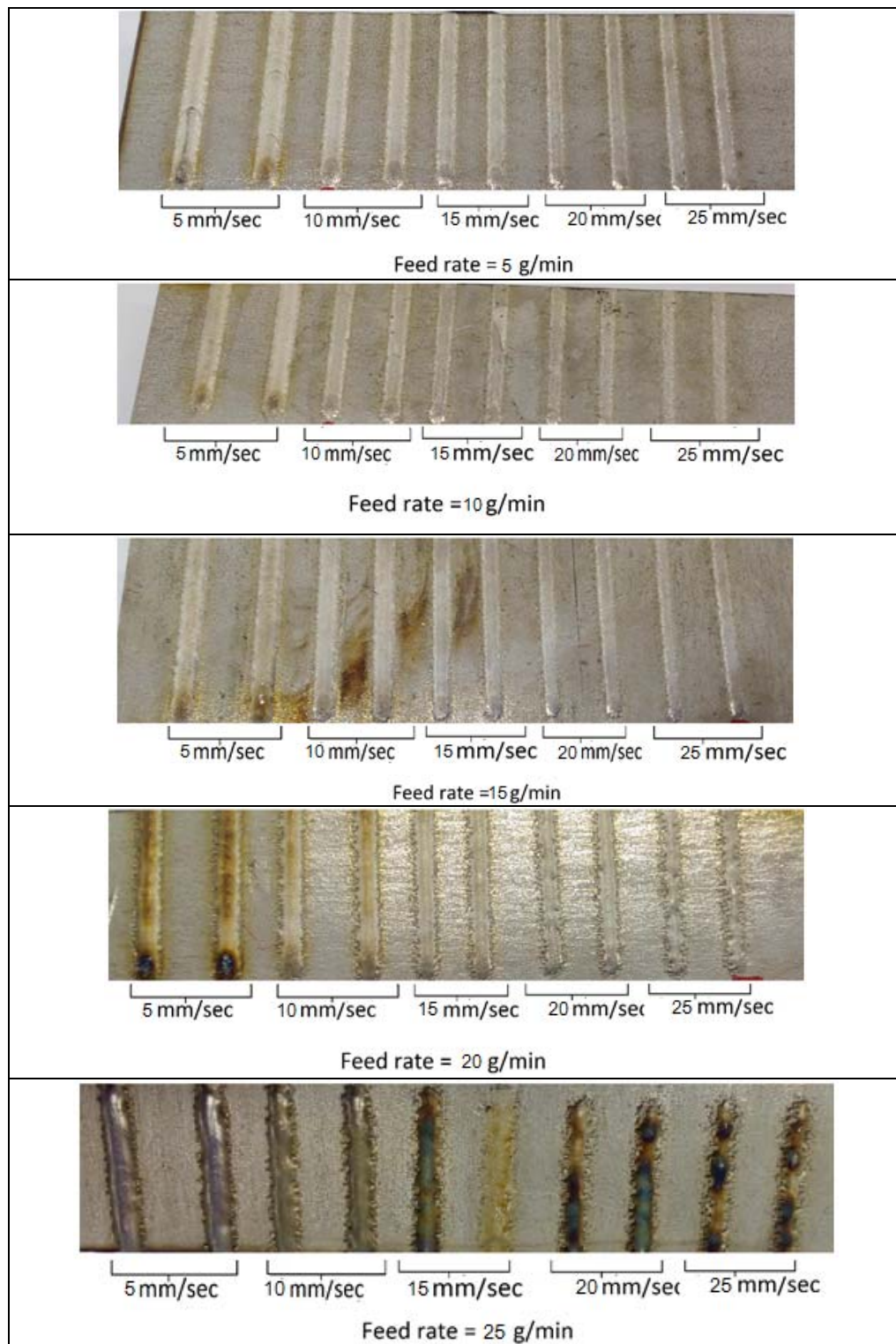
Appendix B2: Taguchi Design of Experiments (DoE)

Process variables	Levels of parameters		
	1	2	3
Feed rate (g/min)	5	7.5	10
Scanning speed (mm/sec)	5	10	15
Spot diameter (mm)	2	3	4
	Fixed parameters		
Stand-off distance (mm)	12		
Laser power (Watts)	1800		
Spot size (mm)	4		
Carrier gas (L/min)	2		
Shielding gas (L/min)	8		

Appendix C: Laser deposited T-800 samples for process mapping (Preliminary experiment).



Appendix D: Laser deposited Stellite 6 samples for process mapping (Preliminary experiment).



Appendix E: Data set from Tribaloy T800 coatings

Feed rate (g/min)	Scan speed (mm/sec)	Width-sample 1	Width-sample 2	Av. Width (μm)	Std.Dev.	Error (Width)	Height-sample 1	Height-Sample 2	Av. Height (μm)	Std.Dev.	Error (Height)	w/h ratio
5	5	3423.56	3408.84	3416.20	10.41	7.36	310.42	330.25	320.34	14.02	9.91	10.66
	10	2970.43	2954.41	2962.42	11.33	8.01	263.53	272.52	268.03	6.36	4.50	11.05
	15	2779.35	2794.38	2786.87	10.63	7.52	200.35	208.53	204.44	5.78	4.09	13.63
	20	2379.95	2420.47	2400.21	28.65	20.26	142.54	146.34	144.44	2.69	1.90	16.62
	25	2369.29	2375.63	2372.46	4.48	3.17	125.94	130.03	127.99	2.89	2.05	18.54
10	5	2968.57	2943.83	2956.20	17.49	12.37	345.67	352.59	349.13	4.89	3.46	8.47
	10	2810.71	2795.29	2803.00	10.90	7.71	261.48	265.53	263.51	2.86	2.02	10.64
	15	2505.23	2468.94	2487.09	25.66	18.15	209.13	219.42	214.28	7.28	5.15	11.61
	20	2215.23	2223.36	2219.30	5.75	4.07	165.51	169.23	167.37	2.63	1.86	13.26
	25	1971.49	1946.93	1959.21	17.37	12.28	130.46	130.57	130.52	0.08	0.05	15.01
15	5	3125.53	3100.97	3113.25	17.37	12.28	780.31	782.45	781.38	1.51	1.07	3.98
	10	2845.27	2829.42	2837.35	11.21	7.92	702.48	706.77	704.63	3.03	2.14	4.03
	15	2463.74	2484.25	2474.00	14.50	10.26	609.89	615.98	612.94	4.31	3.05	4.04
	20	2306.97	2319.62	2313.30	8.94	6.33	520.03	528.42	524.23	5.93	4.19	4.41
	25	2064.56	2025.09	2044.83	27.91	19.74	400.55	406.31	403.43	4.07	2.88	5.07
20	5	3123.59	3130.04	3126.82	4.56	3.22	823.87	818.58	821.23	3.74	2.64	3.81
	10	2723.94	2717.35	2720.65	4.66	3.30	710.85	719.48	715.17	6.10	4.32	3.80
	15	2170.43	2103.54	2136.99	47.30	33.44	523.52	528.97	526.25	3.85	2.73	4.06
	20	1993.55	2012.95	2003.25	13.72	9.70	450.67	456.86	453.77	4.38	3.10	4.41
	25	1849.59	1870.12	1859.86	14.52	10.27	395.79	401.68	398.74	4.16	2.94	4.66
25	5	3230.8	3240.23	3235.52	6.67	4.71	1454.02	1462.69	1458.36	6.13	4.34	2.22
	10	2806.49	2813.75	2810.12	5.13	3.63	1141.54	1145.70	1143.62	2.94	2.08	2.46
	15	2605.97	2615.64	2610.81	6.84	4.84	950.34	955.31	952.83	3.51	2.48	2.74
	20	2405.57	2497.12	2451.35	64.74	45.77	856.48	848.43	852.46	5.69	4.03	2.88
	25	2267.29	2278.08	2272.69	7.63	5.39	740.23	750.31	745.27	7.13	5.04	3.05

Appendix F: Data set from Stellite 6 coatings.

Feed rate (g/min)	Scan speed (mm/sec)	Width (sample 1)	width (sample 2)	Av. Width (μm)	Std.Dev.	Error (Width)	Height (sample 1)	Height (Sample 2)	Av. Height (μm)	Std.Dev.	Error (Height)	w/h ratio
5	5	2875.99	3010.78	2943.39	95.31	67.40	300.52	308.43	304.48	5.59	3.955	9.67
	10	2689.85	2623.64	2656.75	46.82	33.11	242.42	248.23	245.33	4.11	2.905	10.83
	15	2320.43	2310.83	2315.63	6.79	4.80	169.56	179.41	174.49	6.97	4.925	13.27
	20	2268.49	2289.42	2278.96	14.80	10.47	140.52	146.31	143.42	4.09	2.895	15.89
	25	1944.82	1936.39	1940.61	5.96	4.21	107.79	111.58	109.69	2.68	1.895	17.69
10	5	2955.91	2965.94	2960.93	7.09	5.02	308.23	316.42	312.33	5.79	4.095	9.48
	10	2627.62	2612.76	2620.19	10.51	7.43	248.75	254.38	251.57	3.98	2.815	10.42
	15	2343.64	2315.24	2329.44	20.08	14.20	174.82	178.42	176.62	2.55	1.8	13.19
	20	2155.94	2147.23	2151.59	6.16	4.36	146.42	152.56	149.49	4.34	3.07	14.39
	25	1742.58	1736.34	1739.46	4.41	3.12	120.78	128.85	124.82	5.71	4.035	13.94
15	5	2980.23	2990.48	2985.36	7.25	5.13	360.58	305.32	332.95	39.07	27.63	8.97
	10	2753.84	2759.1	2756.47	3.72	2.63	265.45	269.42	267.44	2.81	1.985	10.31
	15	2491.93	2497.08	2494.51	3.64	2.58	180.52	206.24	193.38	18.19	12.86	12.90
	20	2344.91	2352.64	2348.78	5.47	3.87	163.59	171.27	167.43	5.43	3.84	14.03
	25	2190.35	2182.58	2186.47	5.49	3.88	129.58	135.75	132.67	4.36	3.085	16.48
20	5	3143.59	3170.85	3157.22	19.28	13.63	332.67	342.52	337.60	6.97	4.925	9.35
	10	2983.68	2978.54	2981.11	3.63	2.57	269.26	273.23	271.25	2.81	1.985	10.99
	15	2837.84	2848.31	2843.08	7.40	5.23	230.38	234.65	232.52	3.02	2.135	12.23
	20	2740.54	2760.04	2750.29	13.79	9.75	210.47	222.97	216.72	8.84	6.25	12.69
	25	2590.3	2578.28	2584.29	8.50	6.01	197.75	205.79	201.77	5.69	4.02	12.81
25	5	3287.59	3283.74	3285.67	2.72	1.93	340.58	348.37	344.48	5.51	3.895	9.54
	10	3100.32	3110.03	3105.18	6.87	4.86	284.98	290.87	287.93	4.16	2.945	10.78
	15	2990.54	2978.85	2984.7	8.27	5.85	235.57	225.73	230.65	6.96	4.92	12.94
	20	2800.42	2806.34	2803.38	4.19	2.96	213.54	223.75	218.65	7.22	5.105	12.82
	25	2753.68	2763.09	2758.39	6.65	4.71	204.12	208.87	206.50	3.36	2.375	13.36

Appendix G: Microhardness values for Stellite 6 and Tribaloy T800.

Feed rate (g/min)	Scan speed (mm/sec)		Microhardness			Error (Microhardness)		Microhardness			Error (Microhardness)
			Sample 1	Sample 2	Average			Sample 1	Sample 2	Average	
5	5	Tribaloy T800	810.48	798.57	804.53	5.95	Stellite 6	497.75	504.79	501.27	3.52
	10		797.32	805.49	801.41	4.08		509.88	515.28	512.58	2.7
	15		807.42	811.58	809.50	2.08		518.48	522.13	520.31	1.825
	20		831.85	845.58	838.72	6.87		521.97	513.54	517.76	4.215
	25		794.55	799.53	797.04	2.49		518.34	510.84	514.59	3.75
10	5		810.78	818.38	814.58	3.80		506.23	513.42	509.83	3.595
	10		819.49	823.83	821.66	2.17		526.63	511.45	519.04	7.59
	15		804.57	806.12	805.35	0.77		518.42	516.32	517.37	1.05
	20		760.51	764.82	762.67	2.16		485.29	493.58	489.44	4.145
	25		749.83	757.74	753.79	3.95		467.38	463.58	465.48	1.9
15	5		807.94	813.49	810.72	2.77		515.23	509.79	512.51	2.72
	10		790.36	811.74	801.05	10.69		479.47	467.25	473.36	6.11
	15		748.38	768.74	758.56	10.18		463.52	467.23	465.38	1.855
	20		770.74	778.46	774.60	3.86		431.39	437.85	434.62	3.23
	25		747.85	755.28	751.57	3.71		425.23	435.51	430.37	5.14
20	5		812.74	804.68	808.71	4.03		501.45	496.25	498.85	2.6
	10		784.56	826.52	805.54	20.98		485.29	495.2	490.25	4.955
	15		778.85	782.75	780.80	1.95		473.57	479.54	476.56	2.985
	20		777.58	783.52	780.55	2.97		467.42	476.56	471.99	4.57
	25		753.54	759.67	756.61	3.07		471.48	465.38	468.43	3.05
25	5		781.46	787.92	784.69	3.23		467.75	463.85	465.80	1.95
	10		780.30	784.84	782.57	2.27		463.59	469.02	466.31	2.715
	15		761.53	763.56	762.55	1.01		435.93	441.39	438.66	2.73
	20		750.51	756.79	753.65	3.14		421.45	429.56	425.51	4.055
	25		750.74	756.83	753.79	3.05		410.39	422.8	416.60	6.205

Appendix H: Average microhardness of T800 and ST6 at different process regions

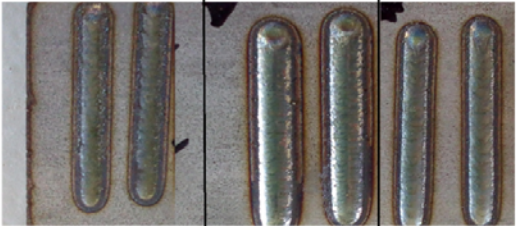
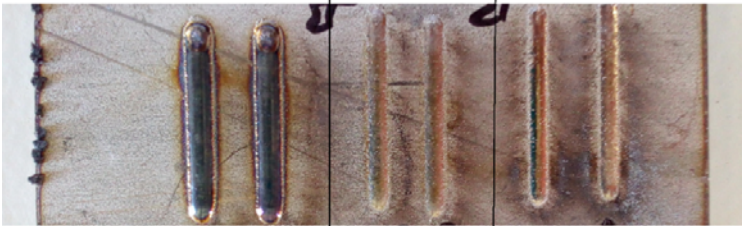
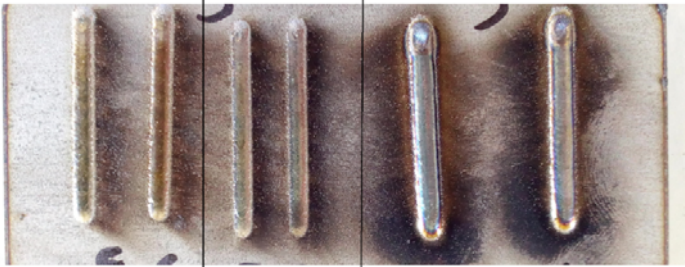
Tribaloy T800

Region	1	2	3	4	5
	804.5	838.7	762.7	810.7	774.6
	801.4	797.0	753.8	808.7	751.6
	809.5	805.3	758.6	805.5	780.8
	814.6			784.7	780.6
	821.1			782.6	756.6
	801.1			762.5	753.7
					753.8
Average	808.7	813.7	758.3	792.5	764.5
Sdt.Dev.	8.0	22.1	4.4	19.1	13.5
Error	3.3	12.7	2.6	7.2	5.5

Stellite 6

Region	1	2	3	4	5
	501.3	520.3	509.8	539.4	512.5
	512.6	517.8	519.0	465.5	473.4
		514.6		465.4	498.9
		517.4		434.6	490.2
				430.4	476.6
					472.0
					468.4
					465.8
					466.3
					438.7
					425.5
					416.6
Average	506.9	517.5	514.4	467.1	467.1
Std.Dev.	8.0	2.3	6.5	43.7	28.4
Error	5.7	1.2	4.6	19.5	8.2

Appendix I: Optimisation samples of T800 coatings (Taguchi Design of Experiment)

 A3 A2 A1
A1 (Spot diameter= 4 mm, Feed rate = 5 g/min, scan speed =15 mm/sec), A2 (Spot diameter= 4 mm, Feed rate = 10 g/min, scan speed =10 mm/sec) and A3 (Spot diameter= 4 mm, Feed rate = 15 g/min, scan speed =10 mm/sec).
 B3 B2 B1
B1 (Spot diameter= 3 mm, Feed rate = 5 g/min, scan speed =10 mm/sec), B2 (Spot diameter= 3 mm, Feed rate = 10 g/min, scan speed =15 mm/sec) and B3 (Spot diameter= 3 mm, Feed rate = 15 g/min, scan speed =5 mm/sec).
 C3 C2 C1
C1 (Spot diameter= 2 mm, Feed rate = 5 g/min, scan speed = 5 mm/sec), C2 (Spot diameter= 2 mm, Feed rate = 10 g/min, scan speed =10 mm/sec) and C3 (Spot diameter= 2 mm, Feed rate = 15 g/min, scan speed =15 mm/sec).

Appendix J: Specific wear rate and Coefficient of friction data (Taguchi design of experiment)

Run	Feed rate (g/min)	Scan speed (mm/sec)	Spot Diameter (mm)	Specific wear rate (mm ³ /Nm)					COF				
				sample 1	sample 2	Average	Std.Dev.	Error	sample 1	sample 2	Average	Std.Dev.	Error
1	5	15	4	0.0123	0.0133	0.0128	0.000707	0.0005	0.0835	0.0813	0.0824	0.001556	0.0011
2	7.5	5	4	0.00669	0.00801	0.00735	0.000933	0.00066	0.0823	0.0765	0.0794	0.004101	0.0029
3	10	10	4	0.0178	0.0199	0.0189	0.001485	0.00105	0.0816	0.0806	0.0811	0.000707	0.0005
4	5	5	2	0.0063	0.0084	0.0074	0.001485	0.00105	0.0846	0.0809	0.0828	0.002616	0.00185
5	7.5	10	2	0.0132	0.0164	0.0148	0.002263	0.0016	0.0811	0.0774	0.0793	0.002616	0.00185
6	10	15	2	0.0479	0.0261	0.037	0.015415	0.0109	0.0836	0.0813	0.0825	0.001626	0.00115
7	5	10	3	0.011	0.00857	0.00982	0.001718	0.00121 5	0.0736	0.0758	0.0747	0.001556	0.0011
8	7.5	15	3	0.0222	0.0234	0.0228	0.000849	0.0006	0.0714	0.0753	0.0734	0.002758	0.00195
9	10	5	3	0.0253	0.0277	0.0265	0.001697	0.0012	0.0712	0.0673	0.0693	0.002758	0.00195

Appendix K: Microhardness of T800 coatings (Taguchi Design of Experiment).

Run	1	2	3	4	5	6	7	8	9
Hardness	852	902	714	892	724	522	843	565	623
	851	938	626	903	729	457	865	609	622
	829	902	658	840	773	484	898	586	610
	824	914	645	848	796	438	831	583	624
	820	932	673	860	802	453	856	556	642
	829	914	631	902	786	423	843	563	603
	836	937	679	894	734	476	833	579	616
	863	903	680	890	775	495	884	597	625
Average Hardness	838	918	663	879	765	469	857	580	621
Std.Dev.	15.47	15.70	29.25	25.25	31.33	32.15	24.25	17.99	11.59
Error	5.47	5.55	10.34	8.93	11.08	11.37	8.58	6.36	4.1