



Original article

Influence of nanocrystalline nickel powder on oxidation resistance of spark plasma sintered Ni-17Cr6.5Co1.2Mo6Al4W7.6Ta alloy

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ABSTRACT

An investigation was carried out to evaluate the isothermal oxidation behavior of nickel based superalloys at 1100 °C for 120 h. The alloys which were microstructured and nanostructured were developed by spark plasma sintering system. The oxidation test was carried out at a temperature of 1100 °C in air at different exposure time up to 120 h. The nanostructured nickel based alloy possessed better oxidation resistance with the formation of continuous adherent alumina, chromia scales, and spinel on the alloy surface and with no significant internal oxides. The oxidation rate of NiO was observed to be lower in the nanostructured nickel superalloy due to low diffusion rate of Nickel. The oxidation kinetics followed a parabolic oxidation law.

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1. Introduction

Due to increasing interest for improvement in the efficiency of aerospace engine and thermal plants, nickel based superalloys have been required to attain higher oxidation resistance at higher temperatures (Latief et al., 2013; Sun et al., 2018). The oxidation resistance of an alloy relies on a continuous, slow growing, dense and adherent oxide scale that can effectively reduce the oxidation growth rate and improve the service life. Consequently, a protective oxide depends on the selective oxidation of elements such as Cr, Al and so on. Alumina and chromia oxide scales are known to be protective, but beyond 900 °C chromia scale is prone to volatility. The adherent and continuous alumina on the surface of the alloy implies a high content of aluminium in the alloy and consequently results into reduced mechanical properties (Niu et al., 2006; Asteman et al., 2013; Yu et al., 2017; Sun et al., 2018). In

addition, the increased growth rate of NiO contributes immensely to the oxidation rate of nickel based superalloy. High oxidation rate of superalloys is a factor which reduces the long life span of high temperature components (Saber et al., 2017). The oxidation process of superalloys are affected by key factors such as the processing route, alloy composition (Khalid et al., 1999), and material state in terms of particle size. Nanostructured materials are materials with sizes of few nanometers with novel properties. Under oxygen rich environment, materials with smaller size and higher defect densities are anticipated to oxidize rapidly, since higher defect density can influence oxidation by accelerating the reactivity of oxygen with elements and increase oxygen diffusion (Liu et al., 2017). On the contrary, it was shown that the reduced size of nanostructured materials can significantly offer enhanced oxidation resistance (Liu et al., 2017). As it can proffer benefits such as reducing porosity which is one factor that can promote internal oxidation. The development of nickel based superalloy using conventional processing routes such as casting, induction melting are prone to inducing defects (Babalola et al., 2018a,b) such as alloy segregation, have their density dependent property reduced due to the present of pores (Shongwe et al., 2015). For improved oxidation resistance behaviour of superalloys, porosity which can aggravate internal oxidation should be reduced or eliminated if possible. Thus, it is essential to develop and improve on the oxidation beha-

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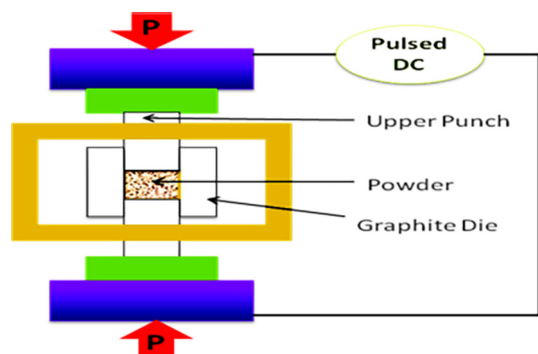


Fig. 1. Schematic diagram of the Spark Plasma Sintering technique.

viour of the bulk alloy through advanced processing route. Spark plasma sintering (SPS) is widely accepted as a rapid growing and novel pressure assisted sintering technology which exhibits fast heating rate, a relatively low sintering temperature and a short isothermal holding time (Shongwe et al., 2015; Shongwe et al., 2016b; Stanciu, Cernea et al., 2017; Babalola et al., 2018a,b) (see Fig. 1). The SPS starts from pulsed electrical current through a graphite die and powder under uniaxial load, the powder is heated via the joule heating generated through the spark plasma within conductive particles, hence densification is promoted (Shongwe et al., 2015; Shongwe et al., 2016a; Shinohara, Fujii et al., 2017). Advantages of SPS over other conventional processing routes is that powders are consolidated into bulk materials within relative short sintering time at relatively lower temperatures below their absolute melting point (Babalola et al., 2018a,b), and defects such as grain growth, coarsening of fine grains are minimized (Hassanzadeh-Tabrizi, 2017; Wynne et al., 2016; Bahrami et al., 2017). In addition, mechanically milling of powders result in particle size reduction and morphological changes which leads to higher densification (Kennedy et al., 2013) by reducing porosity during sintering, and consequently help to reduce oxidation rate. In this study, the effects of nanocrystalline nickel powder developed by high energy ball milling was investigated on the isothermal oxidation behaviour of spark plasma sintered nickel based superalloy at 1100 °C in an uncoated condition.

2. Experimental procedures

2.1. Fabrication and preparation of samples

In this experiment, raw elemental powders of Cobalt (50 mesh, 99.99% purity) and Molybdenum (2–4 µm, 99.99% purity) both supplied by Cerac/Pure Advanced Speciality Inorganics; Nickel (3–7 µm, 99.8% purity) and Tungsten (25 µm, 99.9% purity) supplied by Goodfellow; Chromium (10 µm, 99.2% purity) supplied by FloMaster Metal Powder; Aluminium (25 µm, 99.8% purity) supplied by TLS Technik GmbH & Co.; and Tantalum (–325 mesh, 99.9% purity) supplied by Aldrich Chemistry were used. The elemental composition of the alloy produced is given in Table 1. Microstructured nickel based superalloys was developed by adding Ni, Cr, Co, Mo, Al, Ta and W according to stoichiometry and mixed

thoroughly to attain homogeneity prior to sintering by Tubular mixer for 8 hr at 49 rpm. The process to develop dense nanostructured nickel superalloy from the as-received state micrometric commercial powder entails two step: (i) mechanical milling of nickel powder was carried out using Restch PM-400 planetary ball mill for a duration of 10 hr to attain nanoscale, (ii) the milled nickel powder was blended and mixed thoroughly using Tubular mixer for 8 hr in dry state with other elements according to stoichiometry to attain homogeneity prior to sintering. Commercial nickel powder was milled under vacuum in a stainless steel pot, using stainless steel balls with a ball to powder weight ratio (BPR) of 10:1, and ethanol was used as the process control agent. The set milling parameters were milling speed of 350 rpm, milling interval of 2 hr, and break interval at 15 min. The nanostructured and microstructured nickel based mixed powders were poured into a graphite die with a diameter of 20 mm separately and sintered in the spark plasma sintering system (HHPD-25, FCT System GmbH). The alloys were sintered at sintering temperature of 1100 °C, sintering pressure of 50 MPa, heating rate of 100 °C/min and a holding time of 10 min in vacuum. The sintering temperature was monitored with the aid of an optical pyrometer implanted in the SPS system, positioned at 3 mm from the top of the sample surface. Prior to oxidation, the sintered alloys were polished with 180-grit silicon carbide and up to 1 µm with diamond paste, and, eventually, cleaned with ethanol to remove surface contamination. The relative density of the samples was measured by Archimedes method shortly after removing the sintered specimens from the graphite die. The value obtained for each alloy is an average of five measurements which were 92.4% and 97.3% for microstructured and nanostructured alloy respectively.

2.2. Isothermal oxidation test

Specimen size of 7 mm × 5 mm × 4 mm was cut from the sintered superalloys. The initial weights of the specimen were taken with the aid of an electronic balance (0.01 mg precision, Ohaus). The isothermal oxidation tests were carried out in a muffle furnace (PHOENIX Muffle Furnace) at 1100 °C up to 120 h. Every cycle of the oxidation test entails heating the furnace to the required oxidation temperature, then heating of the sample for the required duration followed by 10 min cooling outside the furnace. The samples were weighed before and after each oxidation cycle. The weight changes of the oxidized specimens were calculated by deducting the initial weight from the weight observed after each oxidation cycles. The kinetic behaviour of the alloys was analyzed after oxidation test.

2.3. Surface characterization methods

The morphology of the oxidized superalloys was investigated by scanning electron microscopy (SEM) (JEOLJSM-7600F SEM) incorporated with an Energy Dispersive X-ray (EDX) detector (Oxford X-Max) to reveal the chemical composition of the oxide layers that evolved during the course of oxidation. The oxidized superalloys were characterized for phases by X-ray diffraction (XRD) using a PANalytical Empyrean model with Cu Ka radiation and analyzed with the aid of Highscore plus software.

Table 1
Elemental composition of the sintered alloys in wt%

Cr	Co	Mo	Al	W	Ta	Ni
17	6.5	1.2	6	4	7.6	Bal

3. Results and discussion

3.1. Mechanical milling of nickel powder

The powders obtained after 10 hr milling operations shows that it does not retain the initial spherical shape of the commercial nickel powder as shown Fig. 2. The milled powder shows a morphology of large agglomerates as a result of successive fracture/welding process taking place during the milling operation. The crystallite size of the powder as determined from XRD peak profile analysis (Fig. 2c) reflects a decrease from 244 nm to 7.88 nm, and the broadening of peaks also indicated the reduction in crystallite size. The mechanical milling of nickel powder has allowed for the development of micron sized agglomerates of powders which are composed of nanocrystallites. The XRD result (Fig. 2c) shows that there was no contamination from the milling tools or the presence of additional phase during milling. In addition, the analysis conducted by EDX (Fig. 2b) on the milled nickel powder confirms that there was no presence of foreign inclusions.

3.2. Isothermal oxidation – Kinetics analysis of sintered nickel based superalloys

The distinct features of nickel based superalloys oxidation are defined by oxidation kinetics and the oxide scale composition. The isothermal oxidation behaviour of the sintered nickel based superalloys, microstructured and nanostructured at 1100 °C in air are shown in Fig. 3. As can be seen from Fig. 3, the process of oxidation can be classified in two stages, comprising the initial and the stationary stage. Rapid mass gain was observed at the initial stage of the alloys which is more rapid in microstructured alloy than in the nanostructured alloy. The oxidation rate of the alloys

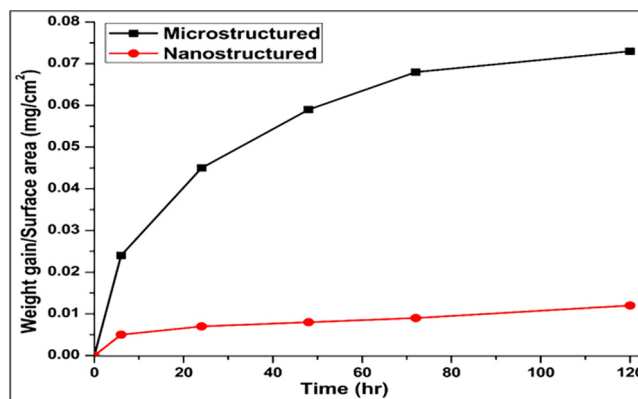


Fig. 3. Weight change/surface area (mg/cm^2) versus time plot for sintered nanostructured and microstructured nickel based superalloy subjected to oxidation at 1100 °C for 120 h.

enters the stationary stage with increasing oxidation time as the mass gain by the alloy reduces. It can also be observed that nanostructured alloy have a lower oxidation rate when compared to its microstructured counterpart. A total mass gain of $0.073 \text{ mg}/\text{cm}^2$ was recorded for the microstructured alloy compared to a lower total mass gain of $0.012 \text{ mg}/\text{cm}^2$ recorded for nanostructured alloy after 120 hr oxidation time. This can be attributed to the incorporation of milled nanocrystalline nickel powder. In addition, the incorporation of nanocrystalline nickel powder in nanostructured alloy helps to reduce the diffusion of oxygen into the alloy by closing down the pores due to their surface area. This trend is in agreement with the density values of both alloys, at higher densification,

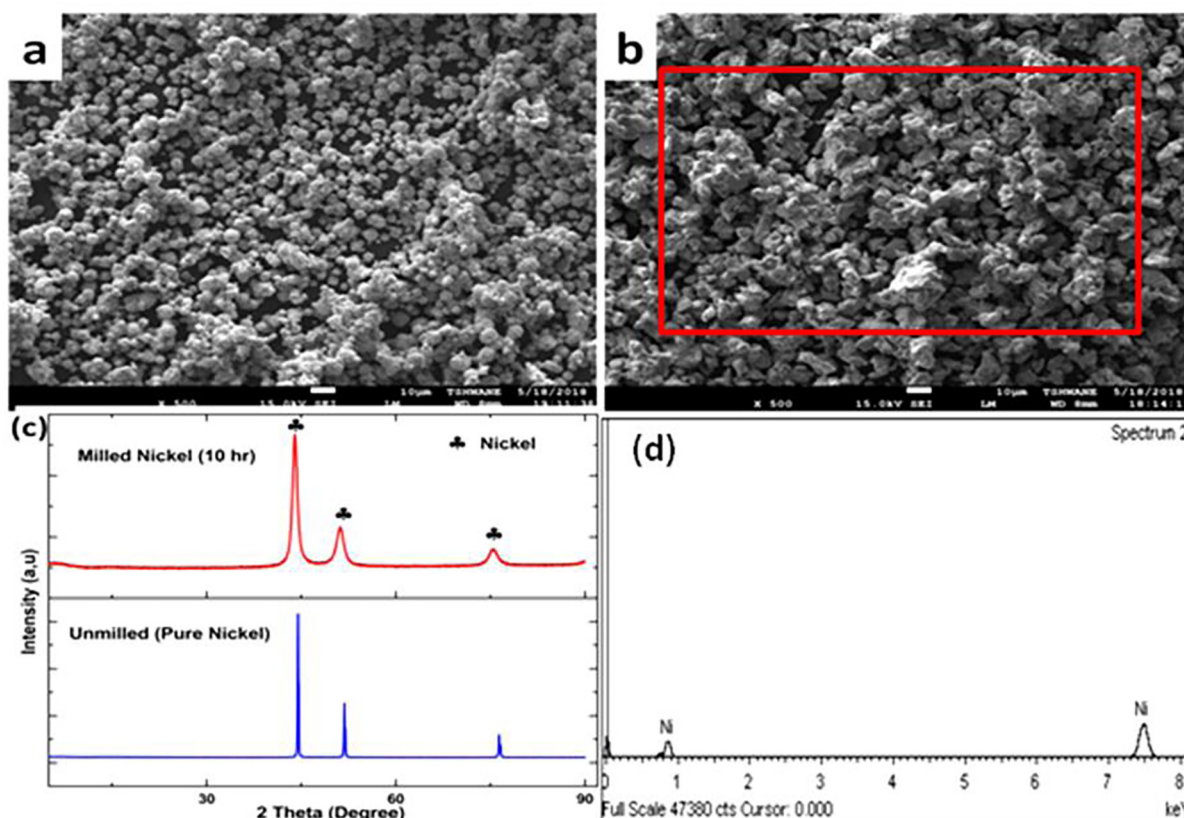


Fig. 2. SEM observation of (a) commercial Nickel powder in the unmilled state, and (b) Nickel powder after 10 hr milling; and (c) XRD patterns of commercial pure (unmilled) nickel and 10 hr milled nickel powders (d) EDX pattern for Nickel powder after 10 hr milling.

the pores are further reduced which hinders the diffusion of oxygen into the nanostructured alloy. The oxidation kinetics of both alloys followed a parabolic law. The diffusion of oxygen and metal oxidative species moving within the oxidation layer of the alloy brings about parabolic oxidation (Saber et al., 2017). The curves (see Fig. 4) can be fitted with the aid of a straight line. The straight line represents the parabolic rate constant K_p which have been calculated for high temperature oxidation behavior for both cases (Geddes et al., 2010; Saber et al., 2017), and it is expressed by the following equation:

$$\left(\frac{\Delta m}{A}\right)^2 = K_p t \quad (1)$$

where Δm is the mass change, A is the surface area of the alloy, K_p is the parabolic constant, and t is the oxidation time. The parabolic constant rates with respect to oxidation time are given in Table 2.

The slope of a linear regression-fitted line, indicating $(\Delta m/A)^2$ against t plot is presented in Fig. 4, which shows the parabolic characteristics of oxidation kinetics. According to the oxidation rate constant values in Table 2, the oxidation rate constants of both alloys was at the peak at the initial stage of the oxidation, and then gradually reduces as the oxidation time increases. This can be attributed to adherent oxide formation on the surface of the alloy, as a result of reduced diffusion of oxygen into the alloy. With continuous isothermal oxidation exposure of both alloys, and increasing growth in the oxide formation, spalling of the oxides was observed more significantly on the microstructured alloy at the 48 hr and less in the nanostructured alloy at the 72 hr. The factors leading to the spalled oxides may be attributed to the difference in thermal expansion coefficients between alumina and chromia, which consequently leads to the breakaway of oxides on the surface of the alloy. The differences in the thermal coefficient occurring between oxides on the surface of the alloy may result into shear stress, and consequently leads to the damage of the oxides at the process of cooling (Yu et al., 2018). The results of the oxidation behaviour of the sintered alloys are indeed lower compared to similar nickel based superalloys developed by conventional processing routes. Ni-Cr-Co-W base superalloy was prepared through a double process of vacuum induction melting (VIM) and vacuum arc remelting (VAR) a parabolic constant of $2.945 \text{ mg}^2 \cdot \text{cm}^{-4} \cdot \text{h}^{-1}$ at 1300°C (Zhang et al., 2017). Ni48Cr28W5Co3Mn1Si1.6 was developed using induction furnace and a minimum parabolic constant of $1.68 \times 10^{-1} \text{ mg}^2 \cdot \text{cm}^{-4} \cdot \text{h}^{-1}$ for the superalloy with 0.2% Re addition, and a minimum of $1.76 \times 10^{-1} \text{ mg}^2 \cdot \text{cm}^{-4} \cdot \text{h}^{-1}$ for the superalloy without Re addition was reported (Liu et al., 2016). The oxidation behaviour of two nickel based superalloy developed

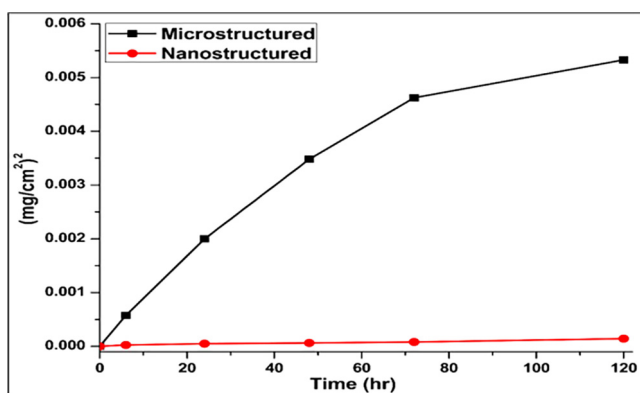


Fig. 4. The parabolic constant rate (k_p), the slope of the curve fitting, $k_p = (\Delta m/A)^2/t$ for the sintered alloy subjected to isothermal oxidation for 120 hr at 1100°C .

Table 2

Oxidation rate constant (K_p) of sintered alloys at the temperature of 1100°C at different time.

Oxidation duration (hr)	Oxidation rate constant, K_p ($\text{mg}^2/\text{cm}^4 \cdot \text{s}^{-1}$)	
	Microstructured alloy	Nanostructured alloy
6	2.670×10^{-8}	4.2×10^{-10}
24	2.440×10^{-8}	5.0823×10^{-10}
48	2.014×10^{-8}	2.71972×10^{-10}
72	1.784×10^{-8}	3.36611×10^{-10}
120	1.234×10^{-8}	1.64648×10^{-10}

by metal injection molding were investigated. The two alloys are Inconel 713 and MAR-M247 from temperature interval of 800°C to 1100°C . The parabolic constants of the alloys at 1100°C were $5.46 \times 10^{-9} \text{ kg}^2 \text{ m}^{-4} \text{ s}^{-1}$ and $1.95 \times 10^{-8} \text{ kg}^2 \text{ m}^{-4} \text{ s}^{-1}$ for Inconel 713 and MAR-M247 respectively (Albert et al., 2014).

3.3. XRD characterization of the oxide layers

X-ray diffraction (XRD) was used to identify the oxides formed on the surface of the sintered alloys after exposure to isothermal oxidation. The XRD patterns are shown in Fig. 5 which reveals the oxides observed for the alloys after isothermal oxidation in air for 120 hr. The observed results revealed that the adherent and protective oxides formed constitutes that of Ni, Al, and Cr elements and corresponding spinels from them such as NiO , Cr_2O_3 , Al_2O_3 and NiCr_2O_4 respectively. The oxides scales that were developed on the surface were mainly protective oxides except NiO oxide due to its fast growth rate and oftentimes with a porous structure (Liu et al., 2016).

There were pronounced formation of NiCr_2O_4 spinel and Cr_2O_3 as shown by the peak intensity in Fig. 5, which are indication of good oxidation resistance factors. As reported, NiCr_2O_4 offers better oxidation protection at higher temperatures beyond 950°C than NiO , notwithstanding Cr_2O_3 offer more oxidation resistance than NiO and NiCr_2O_4 (Zhang et al., 2017).

3.4. SEM and EDX of oxidized nickel based superalloys

The cross sections of the oxidized sintered alloys after 120 hr were analyzed using SEM as shown in Fig. 6. The distribution of oxide formation within the alloys was homogenous, adherent, and dense to the surface of the sample. As it can be seen from the cross sectional view of the sintered alloys, internal oxidation were more pronounced in the microstructured superalloy

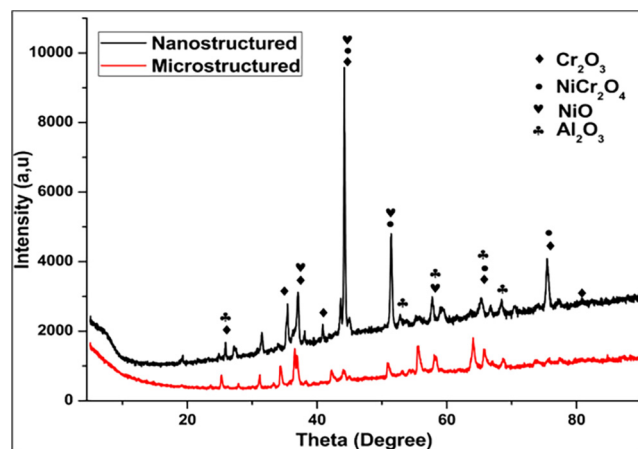


Fig. 5. XRD patterns for sintered microstructured and nanostructured nickel based superalloy oxidized in air at 1100°C for 120 hr.

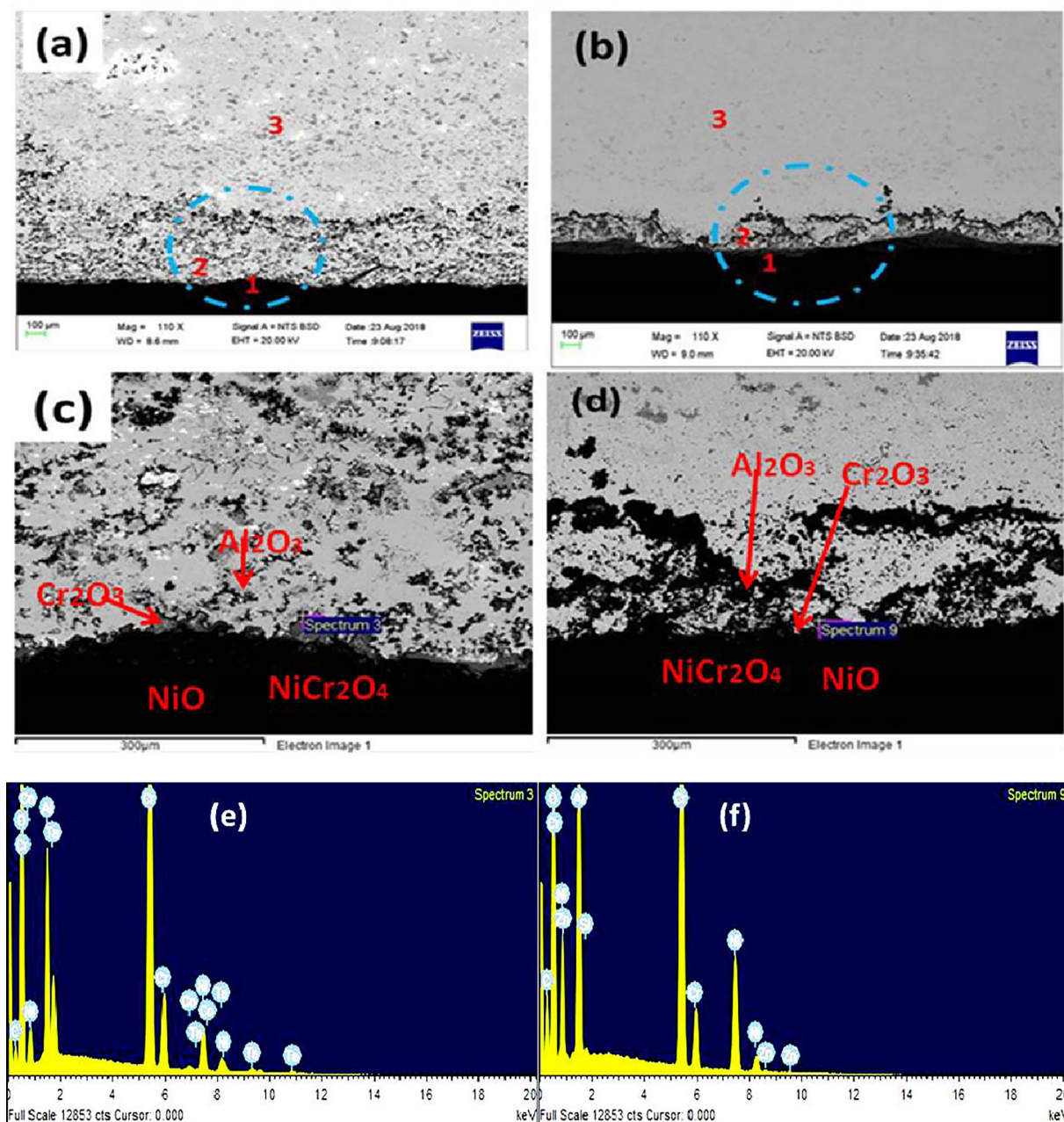


Fig. 6. SEM for sintered nickel based superalloy (a) microstructured, and (b) nanostructured, subjected to isothermal oxidation in air at 1100 °C after 120 hr. (c and d) high magnification of the region in blue scribble in Fig. 6a and b respectively, and (e and f) EDX spot analysis of the oxide layer in Fig. 6c and d respectively.

(Fig. 6a) when compared to the nanostructured superalloy (Fig. 6b). This may probably be as a result of high influx of the diffusion of oxygen into the microstructured alloy than nanostructured alloy. Figs. 6e, f, 7 and 8 shows the EDX analysis of the oxidized sintered superalloys at three different spots in the microstructures, which comprise the oxide layer, internal oxidation zone, and the base metal representing 1, 2, and 3 respectively. The EDX results, as shown in Fig. 6(e, f) revealed that the major oxidation transport elements consist of Ni, Cr, and Al. The oxide layers were mainly NiO, Cr₂O₃, NiCr₂O₄, and Al₂O₃ with their corresponding elemental nominal composition across the points. These were the oxides detected by the XRD analysis. The EDX analysis across the points reveals the variations of the oxidative elements evolved during the oxidation test.

The concentration of the oxidative elements in wt % decreased moderately from the base metal and increased towards the surface

of the metal. Ni and Cr were found to be the dominant diffusion elements during the course of the oxidation. In Fig. 7, the EDX results indicated that the concentration of Ni diffused outwardly from the base metal (with a reduced concentration of 11.01 wt%) region to the internal oxidation layer with an increase of 46.03 wt%. Concurrently, the diffusion of Cr element was steady with its resultant depletion from the base metal and it attained a maximum of 63.35 wt% at the oxide layer. In Fig. 8, the concentration of Ni and Cr as detected by EDX were 61.07 wt% and 15.02 wt% at the base metal region respectively. It was observed from the EDX results, that the concentration of Ni begins to decrease further away from the base metal, and towards the oxide layer which have a minimum of 6.32 wt%.

This indicates that the outward diffusion of Ni in the nanostructured alloy was low when compared to microstructured alloy. Similar behaviour was observed for Al content with more content at

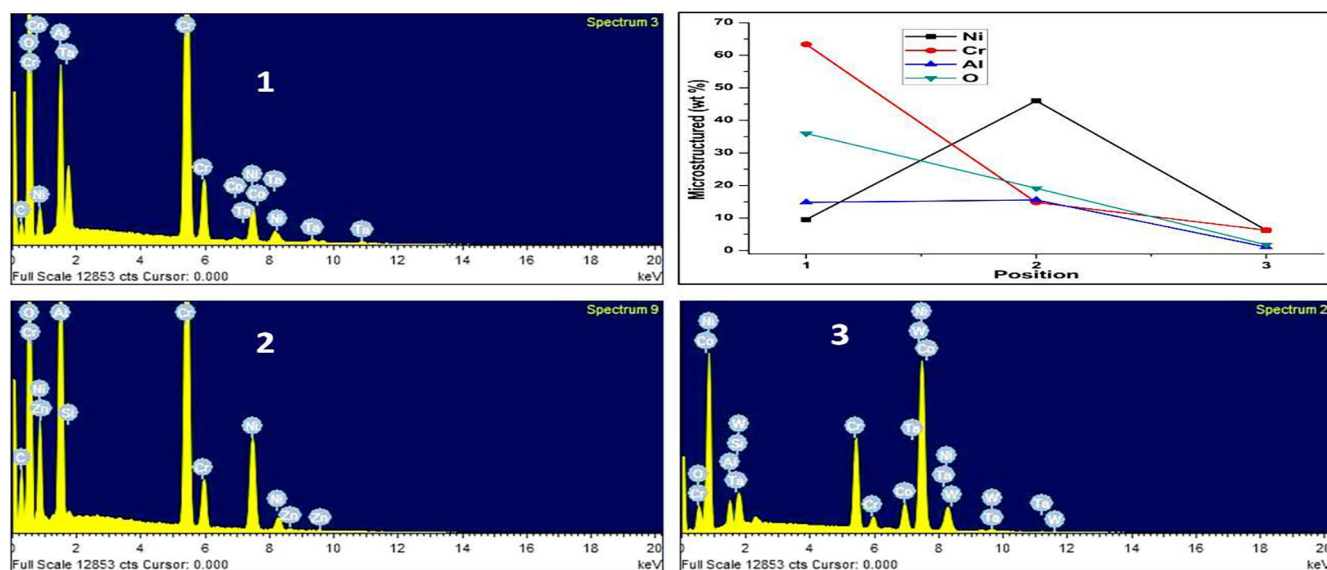


Fig. 7. EDX analysis for microstructured nickel based superalloy with respect to regions labelled in Fig. 6a.

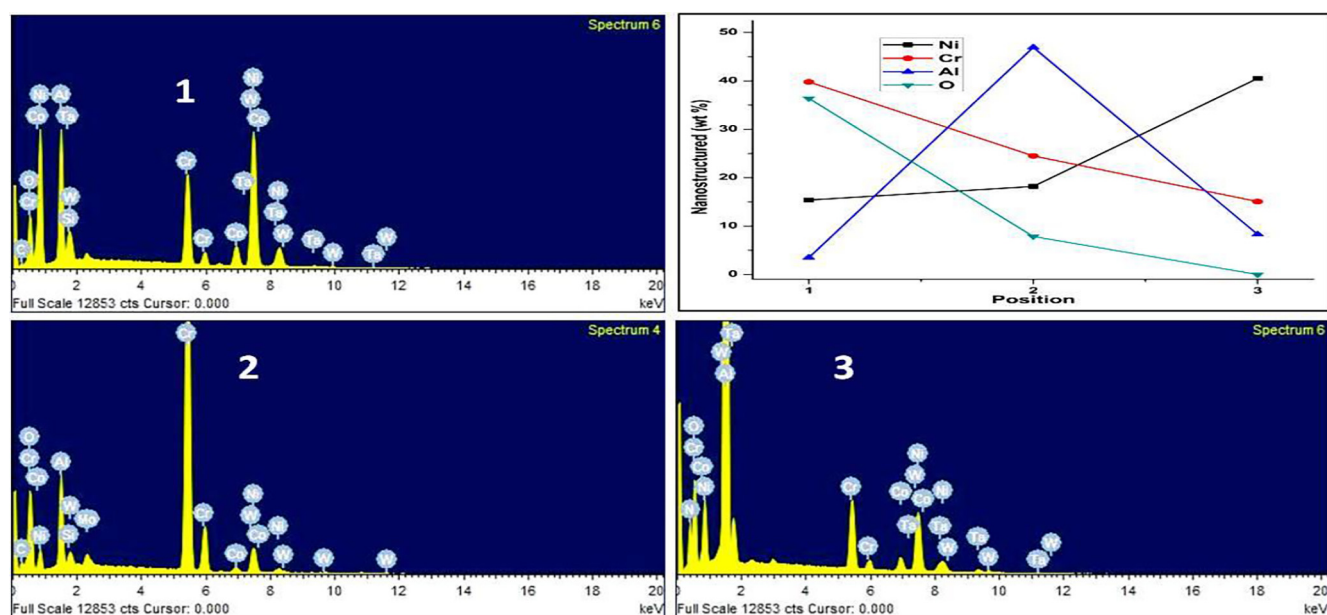


Fig. 8. EDX analysis for nanostructured nickel based superalloy with respect to regions labelled in Fig. 6b.

the outer layer and the internal region for the microstructured alloy while more Al content were retained at the base and internal region in the nanostructured alloy.

However, the concentration of Cr increases to a maximum of 39.92 wt% at the oxide layer which is twice the concentration of the nominal value. It can be said that Cr element diffusion from the base metal to the internal oxidation layer increases in both alloy steadily, while the diffusion of Ni was inhibited in the nanostructured alloy and a rapid diffusion was experienced in the microstructured alloy outwardly. The enhanced oxidation resistance of the nanostructured alloy may be attributed to the reduced oxidation rate of NiO and the formation of a continuous Al_2O_3 internal layer. The Al_2O_3 in the internal layer was not continuous in the microstructured alloy (Fig. 6a). The diffusion of both Ni and Cr to the surface of both alloys indicates the presence of oxides such as Cr_2O_3 , NiO, and NiCr_2O_4 as revealed by the XRD results. In

addition to the enhanced oxidation resistance of the nanostructured nickel based superalloy, a thin layer of NiAl_2O_4 was formed which also inhibit the formation of transient oxides such as NiO (Yun et al., 2017). It was reported that when IN617 alloy is exposed to isothermal oxidation at 900 °C for 1000 h, a good thermal oxidation is observed which comprises of oxides such as NiO, Cr_2O_3 and NiCr_2O_4 (Jang et al., 2011). It was concluded that protective oxides such as NiO, Cr_2O_3 and NiCr_2O_4 were developed which yielded better oxidation resistance of the cyclic oxidation of Inconel 617 (Saber et al., 2017). The development of NiCr_2O_4 spinels on the surface of the alloys enhances the oxidation resistance, as these spinels phases are characterized by smaller diffusion coefficients of cations and anions than their base oxides (Chatterjee et al., 2001; Saber et al., 2017).

Overall, the oxidation kinetics, XRD and SEM-EDX investigations are in agreement with the oxidation resistance and the adhe-

sion of the oxide layer, and it is influenced by the processing adopted in this investigation and the duration of exposure time.

4. Conclusion

The isothermal oxidation behavior of spark plasma sintered nickel based superalloys was studied in air at 1100 °C. The following conclusions were drawn from oxidation test and microstructural analyses.

1. The oxidation process of the nickel based superalloys obeys parabolic rate with parabolic constant (K_p) reducing as oxidation time increases for both structured nickel based superalloy.
2. Protective oxides scales such as, Cr_2O_3 , Al_2O_3 , and NiCr_2O_4 were formed which are responsible for enhanced oxidation resistance at elevated temperature.
3. The outward diffusion of nickel was reduced in the nanostructured nickel based superalloy as it reduces rapid growth rate of NiO.

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