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Influence of spark plasma sintering temperature on the densification and micro-hardness behaviour of Ni-Cr-Al alloy

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Abstract. Sintering temperature is essential towards attaining desired densification and formation of phases which in turn influences the microstructure and properties of a material. In this study, the densification and microhardness behaviour of Ni-Cr-Al alloy prepared by spark plasma sintering (SPS) at different sintering temperatures were investigated. After sintering operation, the density, hardness, phase analysis, and microstructural evolution were investigated using the Archimede's principle, hardness tester, X-ray diffraction (XRD), and scanning electron microscopy (SEM) respectively. The nickel based alloy was sintered at temperatures of 600, 750, 950 and 1100 °C. The results indicated that the densification, microstructure, and hardness values were influenced by changes in the sintering temperature. The relative density increased from 73.89 % at 600 °C to 99.89 % at 1100 °C, while the hardness value was enhanced from 131.9 ± 2.8 HV to 404 ± 1.2 HV respectively.

1. Introduction

Spark plasma sintering (SPS), otherwise referenced as the field assisted sintering technique (FAST) has been recognized for efficient consolidation of materials ranging from metallic alloys, composites, ceramics, functionally graded materials [1] and polymers [2]. The consolidation to near theoretical density is enhanced by spark discharge which is formed between the voids of elemental particles. The spark discharge effect is notable for the purification of the particles' surface from potential oxides and impurities present and its activation which promotes the generation of joule heating. The joule heating effect on the surface of the particles which initiates liquid state particle surfaces results into necking of one particle to another and subsequently aid densification with applied pressure [1, 3]. Shorter sintering times [4], easy control of sintering parameters, inhibiting little or no grain growth [5] and the possibility to consolidate constituent powders at a temperature relatively below melting point [6] forms part of the significant benefits of the spark plasma sintering process when compared to other processing routes. In addition, conventional processing routes have been reported to induce defects such as microsegregation of alloying elements [7, 8], inclusion of low melt inclusion which can



compromise the structural integrity of engineering materials in service and subsequently lead to catastrophic failure. One of the materials which have gained attention over the years are nickel and its alloys owing to their superior ability to retain their strength at elevated temperatures, resistance to oxidation and corrosion among others. They find application in aggressive environments such as aerospace engines, thermal plants, marine [9] and other elevated temperature demanding environments. For instance, Ni-Cr-Al system accounts for the most critical ternary system for nickel based alloys, utilized for elevated temperature application and has attracted research attention [10].

However, defects which arise during conventional fabrication of these alloys have plagued their excellent performance in service and thus necessitate the production of these alloys via novel approach in order to meet increasing performance and efficiency demands. This investigation forms part of research work conducted on the development of nickel-based superalloy via spark plasma sintering technique in which some have been reported [4, 6, 11].

2. Material and methodology

The features of the elemental powders utilized for this research are presented in Table 1. The nickel powder used in this study was further subjected to mechanical milling process through high energy ball milling process (PM400) and a nanocrystalline size of 7.867 nm after 10 hr milling duration at 350 rpm, 10:1 ball to powder weight ratio (BPR), was attained. The mechanical milling of the nickel powder was carried out in a stainless steel milling vial and balls with dimensions of 250 ml and 2 mm diameter respectively. Ethanol was used the process control agent (PCA). The detail report on the milling experiment has been reported elsewhere [11].

Table 1: Alloy elemental constituent's details

Element	Particle size (μm)	Purity (%)	Supplier
Al	25	99.8	TLS Technik GmbH & Co.
Cr	10	99.2	FloMaster Metal Powder
Ni	4-7	99.8	Goodfellow metals

The milled nickel powder after drying was blended with chromium (Cr) and aluminium (Al) in their as received state according to Ni-17Cr-10Al composition by wt.% and mixed thoroughly in a Turbula mixer for 8 h in order to attain homogenized alloy powder prior to sintering process. Mixed starting powder (containing constituent elements) was emptied into a graphite die which have 20 mm internal diameter, lined with graphite foil to ensure lubrication and easy removal after sintering.

The schematic diagram which illustrates the graphite die and sample assembly within SPS process is shown in Figure 1. The mixed powder was sintered at different sintering temperatures (600, 750, 950 and 1100), at 50 MPa sintering pressure, 10 min holding time and 100 °C/min heating rate in vacuum using HHPD-25 SPS model (FCT Germany).

After sintering, 20 mm diameter and 5 mm thickness samples were obtained, followed by sand blasting to remove the remains of graphite foil on the surface of the alloy. This was followed by density evaluation by Archimedes principle; an average of 5 readings for each sintered alloy was recorded. Thereafter, the samples were then prepared for microstructural examination by polishing following standard procedure and using a solution which contains 50 ml of distilled water, 10 g of CuSO_4 , few drops of H_2SO_4 and 50 ml of HCl as etchant.

The characterization of phases formed in the nickel-based alloys was done by utilizing a PANalytical Empyrean model X-ray diffraction (XRD), with $\text{Cu K}\alpha$ radiation. The diffractographs of the sintered alloys obtained over a range of 2θ between 5 and 90°, were studied through the use of Highscore plus software. The microstructural evolutions due to increasing sintering temperature of the alloy were characterized by JOEL JSM-7600F field emission scanning electron microscope furnished with an energy-dispersive X-ray spectroscopy (EDS) detector.

The hardness value of the sintered alloy was evaluated at room temperature by utilizing Future-tech micro-hardness tester at a test load of 1000 gf for a dwell time of 10 s. The average of 10 indentations was recorded for hardness value.

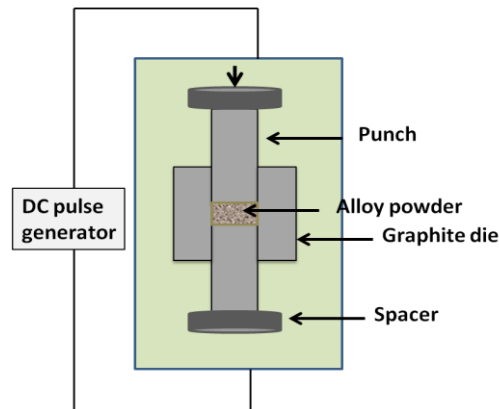


Figure 1. Schematic diagram illustrating SPS assembly of sample and graphite die.

3. Results and discussion

3.1. Densification and hardness behaviour

Figure 2 shows the densification and hardness behaviour of sintered Ni-Cr-Al alloy at increasing sintering temperatures. The relative displacements which occur between the upper punch and the lower punch, within the graphite wall due to the consolidation effect of different sintering temperatures on the powder is presented in Figure 2(a). It can be seen that the displacement increases with corresponding increase in the sintering temperature, while at sintering temperature of 950 °C and 1100 °C attains highest displacement at 3.74 mm and 3.76 mm respectively. This is a typical representation of the densification mechanism of the alloy powder at different sintering temperatures. The relative displacement experienced by the alloy at different sintering temperatures clearly indicates the reduction in the height of powder sample during sintering with respect to time. Similar account on densification analysis has been given in the literature to better understand the consolidation of powders with respect to sintering parameters [12, 13].

Similar behaviour was observed for the relative density and hardness, which agrees with corresponding increase in the sintering temperature, as shown in Figure 2(b). The relative density trend with respect to increasing sintering temperature in this study is in agreement with a study conducted by Zhou *et al.* [14].

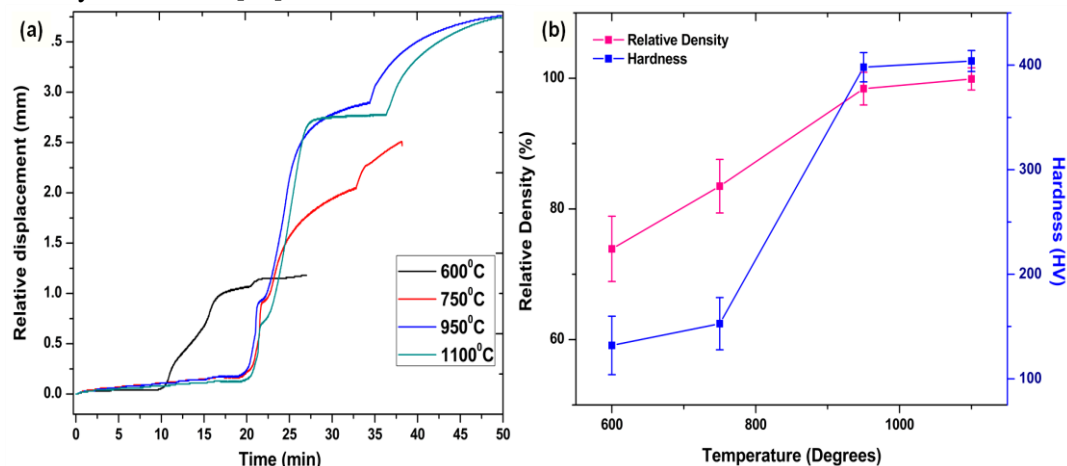


Figure 2. Densification features of sintered Ni-Cr-Al alloy (a) relative displacement against time and (b) comparison of relative density with hardness at different sintering temperatures.

It can also be seen that the hardness value increases as the densification increases (Figure 2b), this can be attributed to the effect of reduced porosity, increased bonding of constituent particles as sintering temperature increases. The lowest hardness value, 131.9 ± 2.8 HV attained at sintering temperature of 600°C can be attributed to its poor densification while the highest hardness value of 404 ± 1.2 HV was obtained at 1100°C .

3.2. Phase analysis and microstructural behaviour of sintered alloys.

The XRD patterns of the sintered Ni-Cr-Al alloy according to sintering temperatures (600°C , 750°C , 950°C and 1100°C) are shown in Figure 3. There were no foreign inclusions or elements detected, which indicates that spark plasma sintering technique aids the fabrication of materials towards attaining intrinsic properties. As shown in the XRD patterns, the occurrence of phases, as the sintering temperature increases. Ni_3Al phase occurred only at 1100°C , Ni_2Al_3 phase evolved at 950°C , NiAl and Ni_2Al_3 evolved at 750°C and NiAl evolved at 600°C . According to Mitra [15] and Dey [16], these are the phases responsible for the strengthening of nickel based superalloys. The increase in hardness value can be ascribed to the occurrence of these phases in the alloy, in accordance to the densification of the alloy at different sintering temperature.

Figure 4 shows the microstructural evolution of Ni-Cr-Al alloy obtained at different sintering temperatures. As shown in Figure 4(a), the initiation of necking formation of the constituent particles of the alloy is evident which clearly reflects that the alloy is yet to attain full densification at 600°C . At increased sintering temperature, 750°C (Figure 4b), more bonding between the particles exist and consequently improves the densification. This is in agreement with the relative density results, as discussed in Section 3.1.

However, at higher sintering temperature (950°C , 1100°C), finer microstructures were attained (see Figure 4c-d) as a result of effective bonding of particles, reduction of porosity, which in turn enhances the hardness value as discussed above. Elemental spot analysis was conducted on the alloy consolidated at 950°C and 1100°C , the dominant peaks in the analysis were mainly Ni and Al. This is in confirmation to the XRD analysis which reveals the presence of strengthening phase which is nickel aluminides. Similar report relating to the impact of sintering temperature on density and its associated properties have been reported by [4, 17, 18].

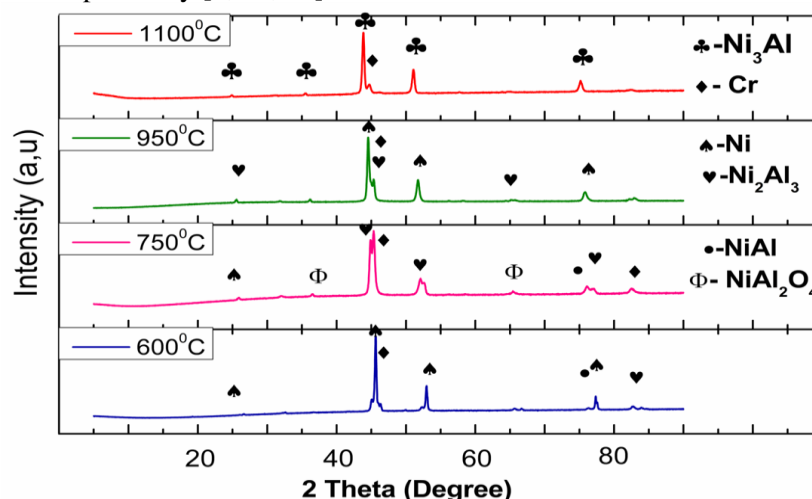


Figure 3. XRD pattern of Ni-Cr-Al alloy fabricated at different sintering temperatures.

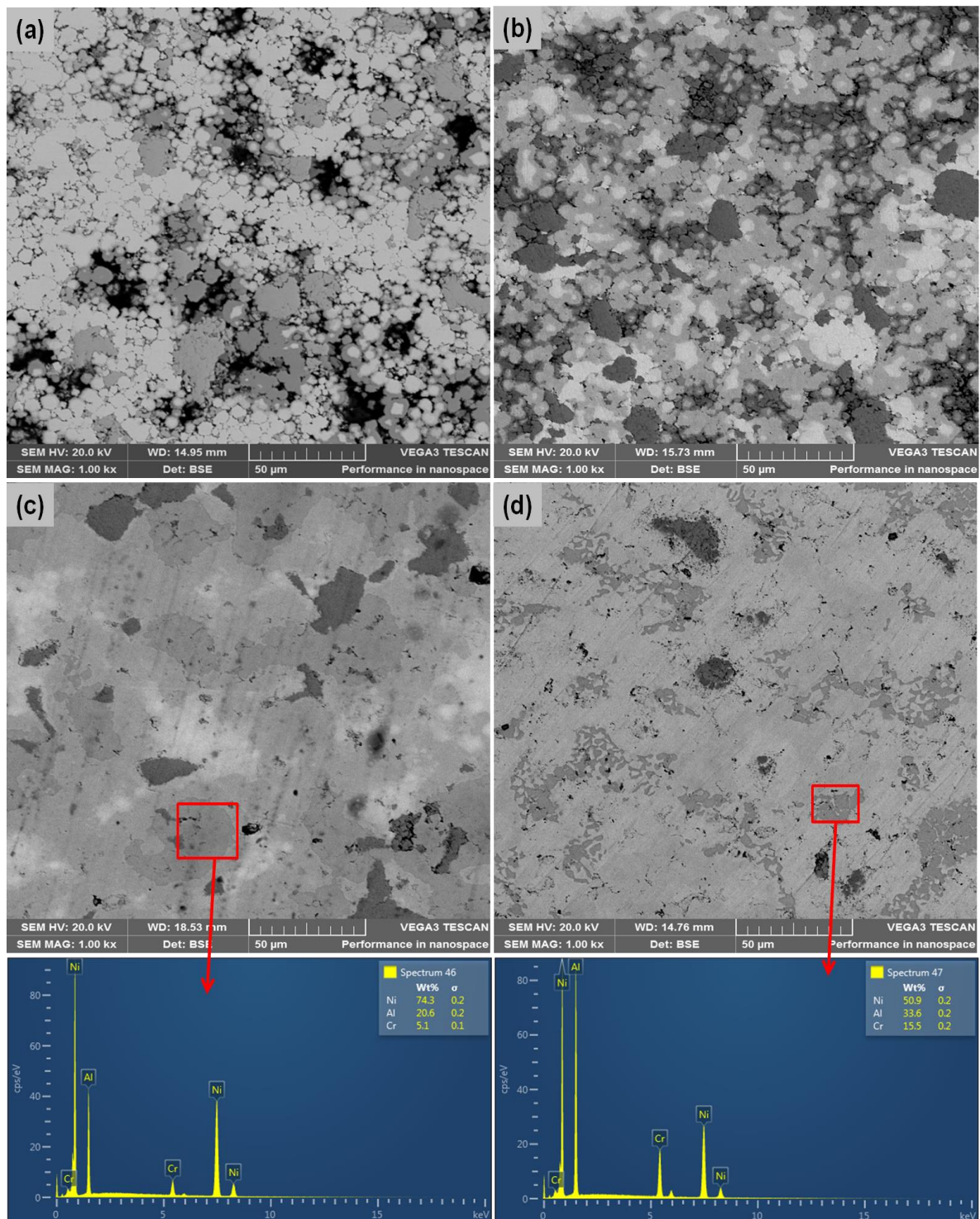


Figure 4. SEM images of sintered Ni-Cr-Al alloy at (a) 600 °C, (b) 750 °C, (c) 950 °C with EDS analysis and (d) 1100 °C with EDS analysis.

4. Conclusion

In this research, an investigation on the role of sintering temperature on the densification and micro-hardness on Ni-Cr-Al alloy was conducted. The following key conclusions were deduced;

1. The relative density and hardness of the Ni-Cr-Al alloy increases due to increasing sintering temperature.

2. The sintering temperature also influences the microstructure of the alloys, more bonding of the particles and finer microstructure were obtained at higher sintering temperature which in turn enhances the hardness value.
3. The formation nickel aluminide phases, which are the strengthening phases in nickel based alloys, were detected. Hence, this production technique is efficient towards the fabrication of advanced material void of defects which can impair its structural integrity while in service.

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