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Influence of Temperature on Microstructure and Mechanical Properties of Ni-40Fe-10Co Alloy Consolidated by Spark Plasma Sintering

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Abstract

The unique features of the Spark Plasma Sintering (SPS) technique were used to investigate the effect of sintering temperature on mechanically milled nickel and iron respectively, with Co additions (Ni40Fe10Co), on the densification and microstructural evolutions. Admixed powders were sintered at various temperatures ranging between 900 and 1100 °C. Material characterizations of powders and sintered compacts was done using scanning electron microscopy (SEM), Optical microscopy and X-ray diffraction (XRD). Milling of Ni for 20hrs was found to be producing larger cold welded clusters, while Fe milling resulted in finer rod-shaped particles. The observed behaviour of Ni during milling was due to its ductility; of which further milling might probably start disintegrating the agglomerates. With an increase in sintering temperature, no substantial detectable new phases were observed. Furthermore, optical images of polished surfaces revealed that with an increasing sintering temperature, the pores coalesce, becomes spherical and thereafter grows. This is attributed to the interdiffusion/phase transformations between the ternary particles upon heating and cooling cycle during sintering. This phenomenon was suspected to be the cause of density and microhardness drop with temperature increase.

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Keywords: Ni based alloys, Spark plasma sintering, Microstructure, Density, Hardness.

1. Introduction

The fast development of modern materials consists in searching for new alloys and improving the already existing ones in order to meet the end-users' requirements [1]. For example, some alloys are intended to be used in high temperature applications where they are required to withstand loading at temperatures near their melting points

while their creep and oxidation resistances are of primary importance. Ni based superalloys have emerged as the material of choice for these applications [2]. These alloys are primarily used in gas turbines, coal conversion plants, chemical process industries and other specialized applications requiring heat and/or corrosion resistance. The alloys are commonly fabricated in bulk by melting processes [3]. However, by the use of mechanical alloying (MA) process, a nanostructured powder product can be produced with low cost [4, 5]. Powder metallurgical alloys give smaller grain sizes and have superior properties to cast alloys [6]. Nanoparticles became a useful way of improving material's performance as compared to their contrary micron-sized particles [7].

Further process development by SPS has even further improved the properties by removal of possible failure sites. SPS process consists of powder pressing under simultaneous flow of current pulses [8, 9]. This leads to increase of the temperature in the local micro-volumes inside the material. The peculiarity of SPS - short sintering time, limitation of grain coarsening, reduced sintering temperature and production of near net shape materials - has made it the technique of choice over other conventional sintering techniques [10]. However, it is of extreme importance to understand the effect of processing parameters if one is to produce fully dense materials. It is reported [11] that the influence of sintering temperature on densification is more pronounced as compared to other sintering variables.

The aim of this paper is to investigate the effect of sintering temperature (900, 1000 and 1100°C) on mechanically milled nickel and iron respectively with cobalt additions (Ni₄₀Fe₁₀Co) on the densification and microstructural evolutions. This study is a part of a comprehensive work in which the independent effect of ternary additions on the microstructure, mechanical and electrochemical properties of Ni-40Fe-10X (X = Al, Co, Cr, Mo, Ta or Ti) based alloys is being investigated.

2. Experimental methods

The as-received, as well as milled, powders were characterized with an X-Ray Diffractometer (model PANalytical EMPYREA). X'Pert high-score plus software was used to investigate the structural changes and phase transformations of powders occurring during mechanical milling. The XRD measurements were carried out with a Cu K α radiation (K α = 1.54056 Å) at an accelerating voltage of 40 kV and a current of 20 mA. Scanning electron microscopy (FESEM, JSM-7600F, Jeol, Japan) with EDAX energy dispersive X-ray spectroscopy (EDS) was used in order to evaluate the morphological changes of certain phases observed in the as-received, as well as nano structured, powder particles. The nickel (Ni), iron (Fe) and cobalt (Co) powders used for this study were supplied by Wear Tech (Pty) Ltd with an initial particle size of 0.5 – 3 and -44 μ m, respectively. The reduction in particle size of powders (Ni and Fe) from micron level to the nano level was carried out using a high-energy planetary ball mill (Model: Retsch, PM 400, Germany) in a stainless steel vial using stainless steel balls of 9 mm Φ . The rotation speed of the planet carrier was set at 300 rpm. The ball mill was loaded with a ball to powder weight ratio (BPR) of 10:1. Ethyl alcohol was used as the medium with an anionic surface active agent to avoid powder agglomeration, severe adhesion to the ball and vial surfaces and oxidation at room temperature [12]. The total duration of Ni and Fe milling was 20 hours and in order to avoid excessive heating, the mill was stopped after every 2 hours for 10 minutes.

The powders were poured into a graphite die with a diameter of 20 mm and then sintered using the spark plasma sintering system (model HHPD-25 from FCT Germany) at different temperatures in vacuum. 13.5g of powder for each sample was filled in a 20 mm diameter die. Graphite paper was put in between die and powder to avoid direct contact of powder with die and for easy removal of sample after sintering. The powders were sintered at 900, 1000 and 1100 °C with a constant pressure of 50 MPa, followed by a 10 min holding time at maximum temperature. For all the sintering experiments, the heating was from room temperature to the desired temperature at the heating rate of 150°C/min. At the end of the prescribed holding time, applied current was switched off and the specimen was rapidly cooled to room temperature. The sintering temperature was measured by an optical pyrometer which was implanted in the SPS apparatus at 3 mm from the top of the sample surface. Discs of 20 mm diameter and approximately 5 mm in height were produced. After the removal of sintered specimens from the graphite die, the density of each samples were measured using Archimedes method where the method involves distilled water as a wetting liquid. The Vickers microhardness (Hv) at room temperature was used to measure the mechanical properties

by a microhardness tester (Future-tech) at a load (P) of 100 gf (1.0 N) and dwell time of 10 s. The test results were recorded for each sample with the arithmetic mean of 10 successive indentations used.

3. Results and discussion

Fig. 1 shows SEM micrograph of the as-received and 20hrs milled Ni and Fe powders. The as-received Ni and Co particles show agglomerates with irregular shapes while Fe particles have flake-like morphology. It can be seen that mechanically milled powders get flattened and coalesce due to the high impact forces (ball-ball and ball-wall collisions) on ductile Ni and Fe powders that lead to cold welding. It is suggested that at longer milling times, powder particles will begin to disintegrate due to welding and fracture cycles and the brittleness induced as a result of continuous milling and work/strain hardening [4]. From Fig. 1 (a), milled Ni particles are further agglomerated/cold welded with irregular shapes. It can also be observed that milled Fe powder particles (Fig. 1b) were flaky and rodlike in nature and a few fractured particles were also present. These features are indications of the severe plastic deformation, together with fracturing of powder particles, occurring during ball milling.

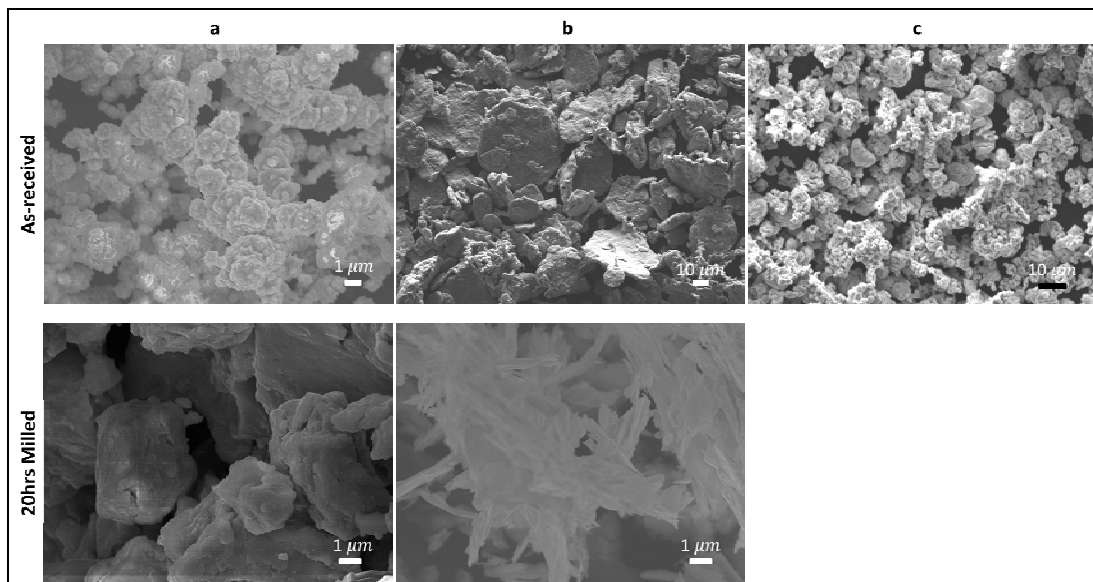


Fig. 1: SEM micrograph of the as-received and 20-h-milled (a) Nickel, (b) Iron and (c) Cobalt powder.

Fig. 2 shows the XRD patterns of Ni-40Fe-10Co alloys sintered at different temperatures. The Figure shows three characteristic diffraction peaks of Ni, Fe and Co. A comparison of the XRD patterns shows an increase in the intensity of peaks followed by a slight shift of a peak to a higher angle with increasing sintering temperature from 900°C to 1000°C. These changes at 1000°C are an indication of a possible crystallization of some amorphous phases, which were formed during ball milling of Ni or Fe. However, with further temperature increase to 1100°C, peak intensity reductions were seen. Further analysis of the XRD patterns shows that there is no visible appearance of any extra peak in the sintered samples with increasing temperature. It shows that sintering temperature did not initiate any appreciable phase transformation in the material.

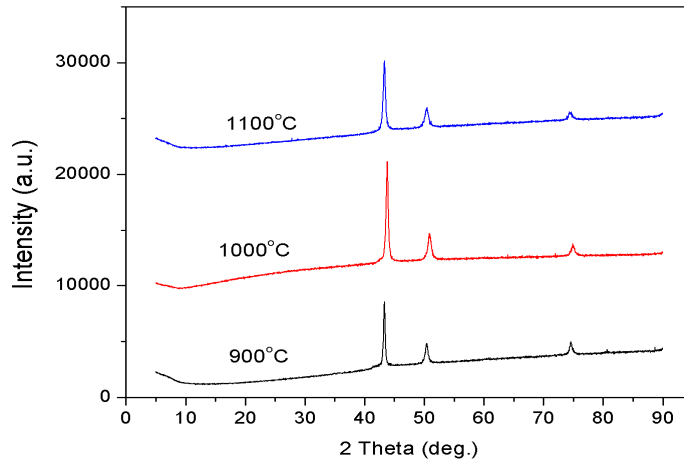


Figure 2: XRD patterns of Ni-40Fe-10Co alloys sintered at different temperatures.

Fig. 3 displays the optical surface microstructures of the Ni-40Fe-10Co alloy sintered at the temperature of 900, 1000 and 1100 °C. Optical analyses of polished surfaces reveal pores for the samples sintered at all temperatures. The pores are seen to be becoming more spherical and coarsening with increasing sintering temperature. Fig. 3 (a) shows a surface of sintered sample at 900 °C. At this temperature, the resulting microstructure still shows characteristics from original structure and the pores are of interconnected nature around the grain boundaries. With increasing sintering temperature, the observed pores coalesce and becomes more spherical (Fig. 3b-c). The larger amount of voids with increasing temperature is probably related to the interdiffusion/phase transformations of the particles during prolonged sintering.

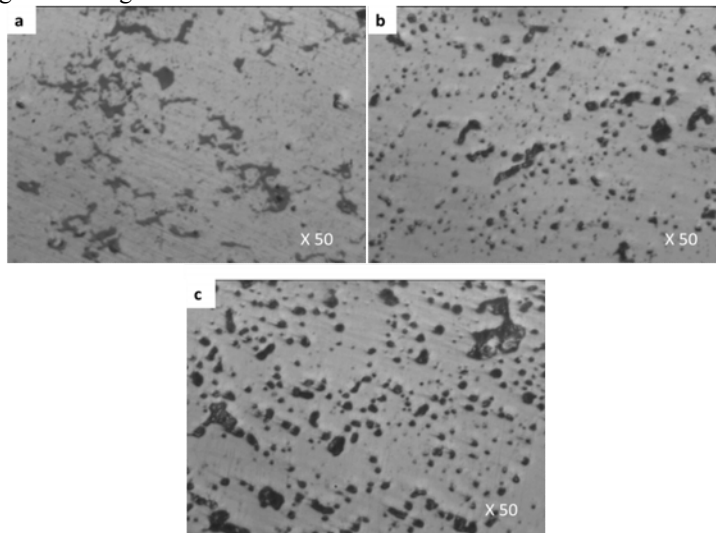


Fig. 3: Optical images showing the effect of sintering temperature on the Ni-40Fe-10Co alloy microstructure,

(a) 900 °C, (b) 1000 °C, (c) 1100 °C.

Relative density and microhardness measurements of Ni-40Fe-10Co alloys sintered at different temperatures are presented in Fig. 4. The samples were polished to a mirror finish before the density and hardness measurements, in order to remove the surface graphite. From Fig. 4 (a), it was seen that an increase in sintering temperature drastically

reduces densification. This can be attributed to the modifications of pore shapes and sizes, seen earlier in Fig. 3, with increasing temperature. The highest relative density reported is 91.4%, with 84.5% being the lowest. The measured microhardness was observed to be near-uniform throughout each sintered sample (low standard deviation), indicating uniform densification. Fig. 4 (b) shows that temperature increase also reduces alloy microhardness with 313.4 Hv being the highest and 211.96 Hv reported as the lowest. In addition to the seen occurrence of pores coarsening, grain growth is also expected to occur as sintering temperature increases and the two are suspected to be the cause of alloy microhardness reductions.

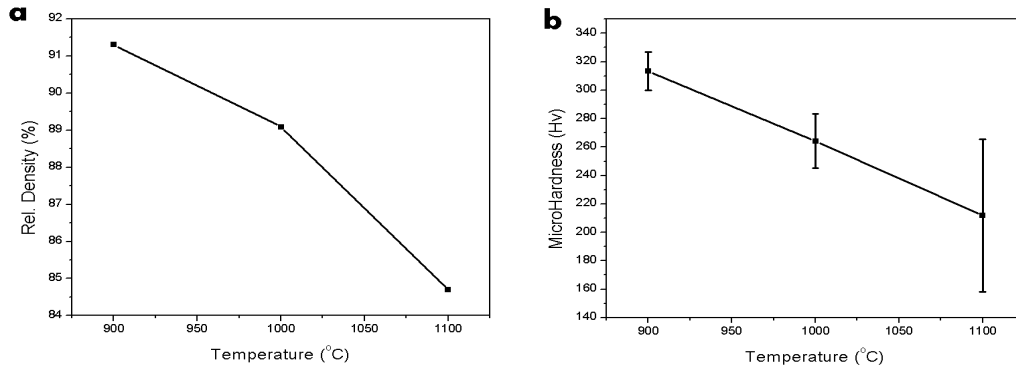


Fig. 4: Analysis of Ni₄₀Fe₁₀Co samples sintered by SPS for range of temperatures 900 to 1100°C, (a) Relative density, and (b) Microhardness.

4. Conclusions

A dense Ni-40Fe-10Co sample has successfully been consolidated through spark plasma sintering and the influences of sintering temperature on microstructure, densification and microhardness were investigated. Mechanical milling of Ni for 20hrs gives clusters of cold welded agglomerates that are larger in size compared to as-received Ni material. This behaviour is normally associated with ductile materials. In contrast, Fe particles' mean size was seen to be reducing after 20hrs of milling. The milled Fe powder particles are flaky and rod-like in shape. Sintering temperature was found to be of significance on alloy densification, and thus an increase in temperature from 900 °C to 1100 °C reduces alloy density. However, change in sintering temperature did not initiate any appreciable phase transformation in the material. Although the experimental sintering parameters used had not given near full densified alloy, increasing sintering temperature was found to be drastically reducing density of the alloy. Microhardness of an alloy was also observed to be dropping with increasing sintering temperature.

Acknowledgements

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