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Influence of sintering temperature on microstructural evolution of spark plasma sintered Inconel738LC

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Abstract

This work studied the effect of processing temperature on the microstructure of sintered Inconel 738LC nickel-based superalloy processed via spark plasma sintering technology. The processing parameters used were, 50Mpa pressure, 5minuts holding time and 100 ˚C/min heating rate, with the sintering temperature varied over three different temperatures at 1000, 1100 and 1200 °C. Inconel 738LC powder was mix at the same proportion for all the samples and under the same condition. Test samples were fabricated from the admixed powders and the influence of sintering temperature on the microstructure, porosity and fracture toughness was characterized. Scanning electron microscope (SEM-EDS) was used to investigate the bulk morphology of the sintered alloy, the phases present within the bulk volume of the fabricated samples was investigated, using energy dispersive X-ray diffraction spectroscopy (XRD). From the result analyzed, the increase in sintering temperature lead to a reduction in porosity, decrease in fracture toughness and increase in hardness value. Increase in sintering temperature had positive impact on the sintered alloy, by reducing the level of porosity present in the sintered samples. There is a reduction in the fracture toughness value of Inconel 738LC, this is expected due to the increase in hardness property.

Peer-review under responsibility of the organizing committee of SMPM 2019.

Keywords: Spark plasma sintering; Inconel 738LC; Microstructure; Porosity; Fracture toughness

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1. Introduction

Nickel-based superalloys are material that has appreciable structural properties, even at high temperature, this encourage their usage at a higher fraction of melting temperature which make them, a better and desirable superalloy for high temperature application when compare to cobalt and iron based superalloys. They possess good thermal and mechanical properties, that enable them to perform at a temperature close to their melting temperature [1]. Nickel-based superalloys are used for several engineering applications, especially areas where strength at high temperature is required, such area includes: gas turbines (vanes, turbine inlets nozzle and turbine blades), nuclear reactor, aerospace engine and tooling materials. Among properties poses by the material are: corrosion and oxidation resistance, creep resistance, thermal stability at high temperature and fatigue strength[2]. Furthermore, the alloys are flexible to other forms of strengthening processes such as solid solution strengthening. Hence the phases usually present comprises, the dominating ɣ matrix phase and ɣ' intermetallic phase, its strength is derived from the coherent precipitation of ɣ' phase in the ɣ matrix phase. Some other possible phases are ɣ'' phase, primary carbides and laves phase which mostly depend on the alloying element present. Inconel738LC (IN738LC) posses all the characteristic mentioned, it is used to fabricate component parts of gas turbines. Especially those that are exposed to high temperature service condition. However, to have an IN738LC with good properties, the material composition and processing technique has a huge impact on the microstructure, phases formed, phase distribution and the mechanical properties [3]. The alloy is commonly fabricated in bulk by melting processes which usually have defects such as micro-segregation, pores and low mechanical properties [4]. Therefore, powder metallurgy is a modern-day technology, use for the production and working of metals, including alloy metal powders, by pressing them in the mould in order to consolidate and bring strength to metallic powder through the application of heat energy (sintering). Opportunities presented by this technology have been greatly explored in recent times, this is attributed to rapid temperature rise, low production time, good densification of material and ease of material production for a specific application [5]. Alloy produced through powder metallurgy usually have smaller grain sizes and superior properties to cast alloys [6]. Spark plasma sintering (SPS) is one of the most sorts after technology in powder metallurgy. It is a method that uses a pulsed direct electrical current of low voltage and uniaxial force, for powder consolidation. The process of powder consolidation and densification are done in one-step during operation, this enables materials to attain near full density within a short period. The diffusion process is aided by the application of intense heating and rapid cooling sequentially, as a result, material composition homogeneity, residual stress reduction, grain refinement and even distribution of secondary phase precipitates (for alloy prone to precipitate formation or intermetallic phase formation) [7] are the characteristic of the product formed by SPS. It was reported[8] that sintering temperature has a greater influence on the densification of material when compared to other sintering variables. The aim of this study is to investigate the effect of sintering temperature (1000, 1100 and 1200 °C) on the microstructure evolution and fracture toughness property of IN738LC. This study is part of a comprehensive work on the investigation of sintering temperature on the microstructure evolution, densification, thermal diffusivity and thermogravimetric analysis of IN738LC.

2. Experimental details

The starting metallic powder for the fabrication of test samples for Inconel 738LC, was a high purity (99.5%) Ni powder, which serve as the matrix powder is supplied by TLS-Technik Gmbh, with a size range from 45 to 90 µm. Other metallic powders were Co and Cr (99.9% purity) in the size range from 3 to 45 µm, supplied by Alfa Aesar. Ta, W and Ti with a particle size less than 45 µm, were supplied by Sigma Aldrich and Al which has a particle size of 25 µm (99.8% purity) was supplied by TLS-Technik Gmbh. All the powders were spherical in shape and had a near to zero contamination at the point of supply. A weighing scale was used to weigh all the elemental powders in accordance with a stoichiometric composition of Inconel 738LC [9], the percentage composition was; 64.58 % Ni, 16 % Cr, 8.3 % Co, 3.4 % Ti, 3.4 % Al, 2.6 % W and 1.7 % Ta. A tubular mixer was used to mix all the powders for a period of 10 hours at 49 rpm speed, after which they were poured into the graphite die for sintering. The sintering was conducted over three different temperature (1000, 1100 and 1200 °C) using 50 Mpa pressure, 100 °C/minute heating rate and 5 minutes holding time, under a controlled argon atmospheric condition. Three test samples were produced in total with a dimension of 40mm diameter by 6 mm thickness (height) and sandblasted to ensure the remover of the graphite coating that was attached to the surface of the sample due to the heating process.

Sintered Inconel 738LC samples were characterized by scanning electron microscope (SEM) (JEOL JSM-7600F SEM) incorporated with an EDS detector with INCA X-Stream2 pulse analyzer software, the phases present were characterized by X-ray diffraction (XRD) machine, a model from PANalytical Empyrean with Cu kα radiation and further simplified the result by analyzing using Highscore plus software. The hardness was done by using Vickers microhardness tester (Future-tech), the percentage porosity was calculated from the percentage density value of the test samples and the fracture toughness was equally characterized by microhardness tester (Falcon 507). Each sample was metallographically prepared before the characterization, grinding and polishing was done (in the order of 600, 800, 1200 grit size silicon carbide papers, while 0.004 mm colloidal silica was used for polishing)

3. Results and discussion

Fig. 1 shows OPM and SEM micrograph of the sintered test samples of IN738LC. There was a web-like pattern with some black patches on the surface of the samples. Pores were not clearly obvious, however, the two close, but different colour (light brown and dark brown) denote different phases formed which was confirmed from SEM micrograph (using EDX) to be chromium rich area, solid solution strengthening dispersion (tungsten) and ɣ' intermetallic phase. The chromium was formed on the interface of ɣ and ɣ', the characteristic that suggests that the material might have good corrosion and oxidation property. Coherent precipitated ɣ' (NI_3(Ti, Al) intermetallic phase is present[10] which may contribute to the improvement of the mechanical property. It was again observed, that the major solid solution strengthening element that precipitated and sprinkling dispersed was tungsten, this is a hard material that might improve the hardness. The SEM micrograph further makes obvious the pores present which were later quantified in percentage. Other solid solution strengthening element such as Ta decomposes in the matrix as it was not revealed by the XRD and EDX analysis.

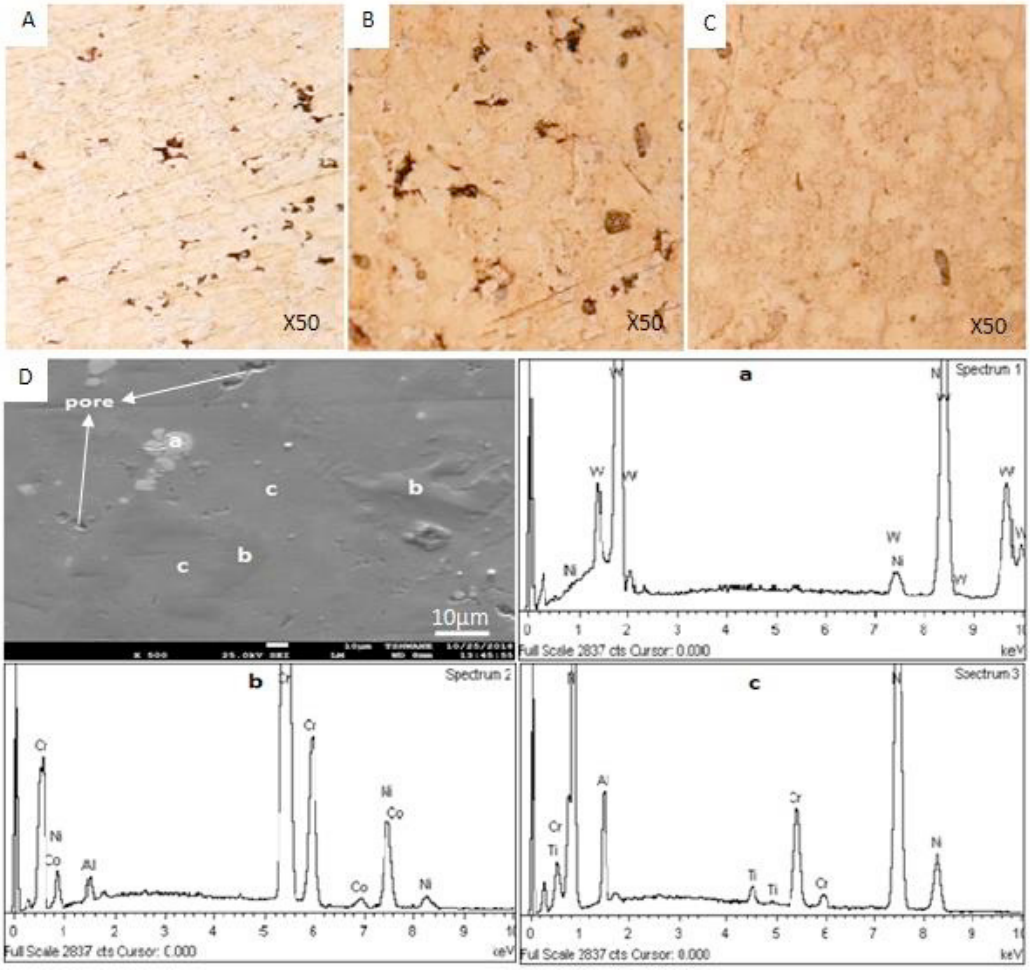

Fig. 1. OPM microstructure of IN738LC sintered at (A) 1000°C, (B) 1100°C, (C) 1200°C and D is the SEM microstructure of 1200°C sintered sample, incorporated with EDX

Fig.2. shows the XRD patterns of IN738LC superalloy sintered at different temperature, peaks formation was identified, the angle 2θ, where the peaks were located was observed. The peaks show the phases formed, the phases intensity and elemental decomposition with respect to sintering temperature. Intermetallics (ɣ') phase dominated, with Cr_2 and Ta also evident, the Ta element decomposes into the matrix as the sintering temperature increases. The intermetallic phase was observed at 45^0, 52^0 and 77^0 Angle 2θ which was in line with a similar investigation conducted by l. Zhu [11]. The ɣ and ɣ' phase overlap, this is not strange as it was a characteristic of superalloys. Chromium precipitation was observed at an angle 2θ of 41^0, signifying that the sintered sample might have good corrosion property.

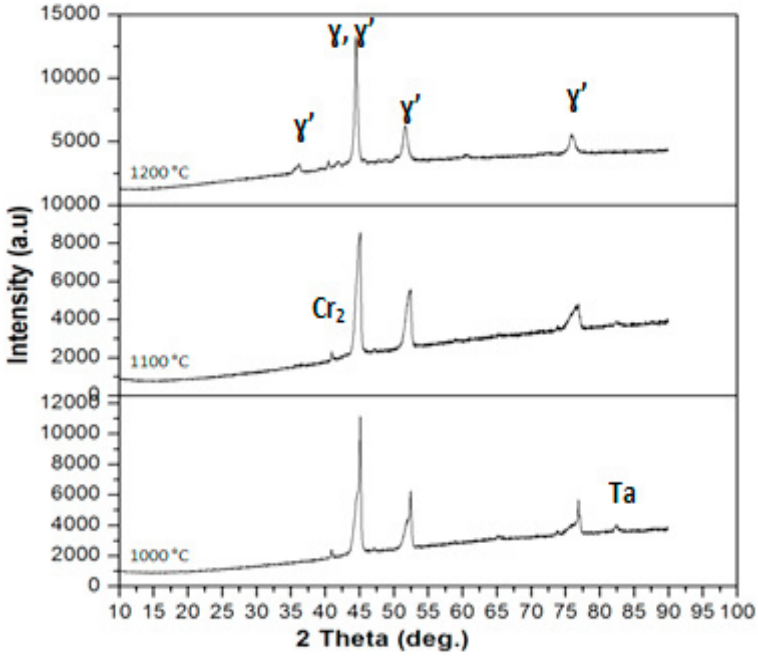

Fig. 2. XRD patterns of IN738LC sintered at different temperatures.

Fig.3. show (a) the hardness/toughness value against the sintering temperature and (b) show the hardness/porosity against the sintering temperature. It was observed that the increase in sintering temperature leads to a reduction in porosity and an increase in hardness value, the fracture toughness reduces with increase in hardness value. This signifies that powder consolidation increase with increase in sintering temperature, also the variation in the particle sizes encourage interstice strengthening as the sintering temperature increases. This leads to the reduction in porosity and improvement in the sintered IN738LC fracture toughness.

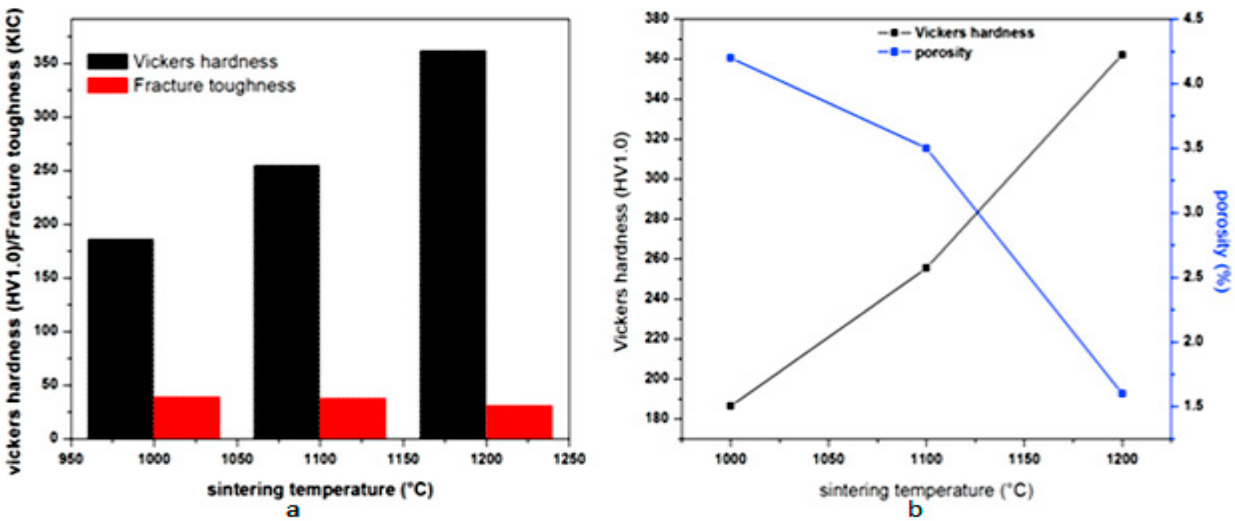

Fig. 3. Analysis of IN738LC samples sintered by SPS for a temperature range from 1000 to 1200°C, (a) Microhardness/fracture toughness against sintering temperature and (b) porosity/microhardness against sintering temperature.

4. Conclusions

IN738LC has been successfully consolidated using SPS technology, the influence of sintering temperature on the microstructure, microhardness, fracture toughness and porosity have been analysed. Formation of ɣ matrix phase, ɣ' intermetallic phase and formation of solid solution strengthening element was observed in the microstructure and XRD analyses. Increase in hardness value, reduction in porosity and fracture toughness was observed with increase in sintering temperature. The sintering temperature was found to have great influence on powder consolidation which helps to reduce porosity formation. There was neither formation of external phase nor agglomeration in the microstructure. With these observations, SPS technology among other powder metallurgical processes is a sustainable method for consolidating metal powders, it is a clean energy fabrication method with a fast production rate and possibility of reproducibility, it also minimize material wastage and it is a cost-effective fabrication method.

Acknowledgments

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