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Injection molding of nickel based 625 superalloy: Sintering, heat treatment, microstructure and mechanical properties

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ABSTRACT

This study concerns determination of optimum sintering and thermal process parameters for Ni-based alloy 625 superalloy formed by the method of powder injection molding (PIM). Samples, formed from the feedstock by mixing the prealloyed 625 powder with a multi-component binding system, are made subject to sintering at different temperatures following the debinding process. Samples that are sintered under such conditions giving way to the highest relative density (3 h at 1300 °C), are aged after they have been subject to solution treated thermal process. Sintered, solution treated and aged samples have been subjected to microstructural analysis and mechanical test. Mechanical tests such as hardness measurement and tensile test as well as microstructural characterization such as X-ray diffraction (XRD), scanning electron microscope (SEM), transmission electron microscope (TEM) and elemental analysis all have shown that the aging thermal process increases strength of the material. However, it is observed that alloy 625 produced by the method of PIM is at such level to compete with the characteristics of cast alloy 625.

1. Introduction

Nickel-based superalloys are an unusual group of metallic materials, showing an extraordinary combination of high temperature strength, toughness and surface stability in corrosive or oxidative environments [1]. Thanks to such superior characteristics of them, they are very important materials for high temperature applications such as aerospace and power generation industries [2]. One of the Ni-based superalloys which are most successfully applied in the engineering applications is alloy 625 [3]. Because of its corrosion strength, high strength and creep strength, alloy 625 is used in the aerospace, chemical power plants and marine applications [4]. Alloy 625 is designed to initially gain high temperature strength by solid solution hardening brought about by Mo and Nb elements in Ni–Cr matrix. However, although designed to initially gain strength by means of solid solution, it is seen that the intermetallic phases and carbides precipitate in it through aging treatments performed in the temperature range of 550–750 °C [4–9].

PIM allows production of parts of complex for in small size close to the net shape from a very wide range of materials such as ceramics, metals and hard metals in medium or high volumes [10,11]. PIM of superalloys have attracted great attention in recent years [12–15]. The reason is that PIM allows overcoming problems that adversely affect part-related characteristics and increase cost of production encountered in the production of the superalloys by traditional methods such as segregation, difficulties of forming and machining [16]. Although a powder metallurgy technique, PIM allows producing parts with complex form in case the traditional mold pressing proves to be inappropriate, and the parts thus produced may have higher densities and, consequently, more superior mechanical characteristics as they have no density gradient [17].

There is a limited number of studies on production of alloy 625 by PIM method [18–20]. These studies have concentrated rather on PIM of alloy 625, but no detailed characterization performed. And the characterizations performed have remained limited to some characteristics of the materials as sintered or solution treated. In the study by Johnson et al. it is reported that more studies are required on optimization of the sintering parameters for alloy 625 formed by the injection molding [18].

It is known that the precipitation hardening plays significant role in the development of mechanical properties of the nickel-based superalloys such as hardness, strength and creeping strength [21]. It is reported that $M_{23}C_6$, MC, M_6C, γ' (gamma prime), γ'' (gamma double prime), δ (delta) and laves phases may precipitate

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in alloy 625 depend on temperature and time [6–9]. Although there are many studies on aging of alloy 625 produced by traditional methods [5–9,22–24], no study was found with respect to aging of alloy 625 produced by PIM.

This study concerns determination of optimum production parameter in producing parts by PIM method that allows economical production of the parts which have more superior characteristics than alloy 625. As mentioned above, it has been reported that alloy 625 gains strength with intermetallic precipitations that may occur by the heat treatment or under service conditions, and there are contradictory explanations in the literature with respect to the temperature and time for their occurrence due to difficulty of the characterization operations of these nano-sized intermetallics that impart strength. For this reason, the parts produced under optimum conditions are kept to different thermal treatment cycles to characterize them. Characterization of samples has been performed both by mechanical tests and XRD, SEM, TEM and elemental analysis. The experimental studies have been conducted to determine how the heat treatment affects the microstructure and mechanical characteristics of this alloy, which is designed to gain strength through the solid solution hardening. However, the data obtained from the characterization have also been compared with forged alloys and cast alloy 625.

2. Experimental procedures

In the experiments, 625 superalloy powder produced by Osprey by gas atomization has been used and the chemical composition of the powder is given in Table 1. Fig. 1 shows the curve obtained from the particle size distribution analysis conducted by use of an equipment, trademark Malvern Mastersizer. From SEM image taken for determination of the particle form given in Fig. 2, it is seen that the powder are spherical in shape. Table 2 shows physical characteristics of alloy 625 powder obtained from the manufacturing firm and used in the experiments.

Feedstock preparation, the first stage of part production by powder injection molding, has been performed in a specially designed mixer by keeping the superalloy powder 60% by volume and binder 40% by volume to a mixing operation under vacuum for half an hour at 170 °C. Multi-component binding system, which constitutes 40% of the feedstock in volume, is consisted of paraffin wax, polypropylene, carnauba wax and stearic acid. Following the mixing operation, the feedstock, cooled down, has been manually granulated and molded in form of tensile samples by using molds produced in an injection equipment with its barrel and nozzle parts heated up to 170 °C under pressure of 12.5 MPa and for a retaining time of 20 s as the standard MPIF-50. Molded tensile samples have been kept subject to a debinding process consisting of two stages, namely, solvent and thermal debinding. The solvent debinding process has been performed by keeping the samples in heptane heated up to 60 °C for 6 h. Thermal debinding, the second stage of the debinding operation, has been performed by thermal cycle shown in Fig. 3b with its conditions as determined according to results of the thermo-gravimetric analysis (TGA) of the components shown in Fig. 3a. As all binding components keeping the powder particles together abandon the materials when the temperature reaches up to 600 °C in the thermal debinding stage, a pre-sintering is assured by reaching up to 900 °C and waiting for 1 h at this temperature to prevent loss of shape of the part in order to keep the forms of the samples till the main sintering performance. Thermal debinding has been performed in the argon atmosphere of high purity.

In order to determine sintering behaviors of the samples formed by PIM method and debinded, the differential scanning calorimetry (DSC) and dilatometric tests have been conducted. DSC analysis has been performed by heating the sample up to 1350 °C at a heating rate of 10 °C/min by Setaram DSC-131 equipment in Ar gas atmosphere of high purity with flow rate 100 ml/min. Dilatometer analysis, allowing determination of expansion or shrinkage which occurs in the material depend on change of temperature, has been performed by heating the sample up to 1313 °C at a heating rate of 10 °C/min under H_2 atmosphere by use of Unitherm Model 1161H equipment followed by cooling down to the room temperature.

Samples have been sintered, which lasted 1 through 3 h in the temperature range of 1260–1300 °C as determined according to the results obtained from DSC and dilatometer tests. The sintering of samples has been performed under high vacuum in Protherm tube furnace by using Al_2O_3 as base. In the sintering operations, constant heating and cooling rate of 10 °C/min has been used. Fig. 4 shows images of the tensile samples produced from alloy 625 powder after molding and after sintering. It is seen that the samples for tensile test have been shrunken considerably.

Density of the sintered samples was measures by Precisa XB 320M precise balance according to the Archimedes principles and, after the metallographic preparations such as grinding and polishing, they were etched by use of the calling solution and visions of them were taken by the optical microscope, Nikon (LP-1200-Elipse).

Table 1
Chemical composition of the alloy 625 powder.

Composition in weight percent (wt.%)										
Ni	Cr	Fe	Nb	Mo	Al	Ti	Co	C	Si	Mn
64.04	20.9	2.6	3.2	8.4	0.01	0.01	0.01	0.029	0.31	0.39

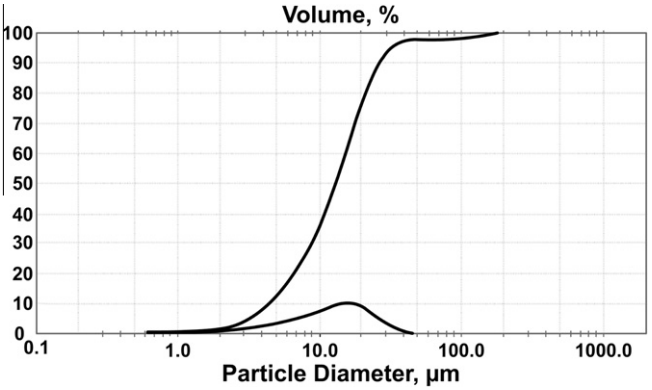

Fig. 1. Particle size distribution of alloy 625 powder.

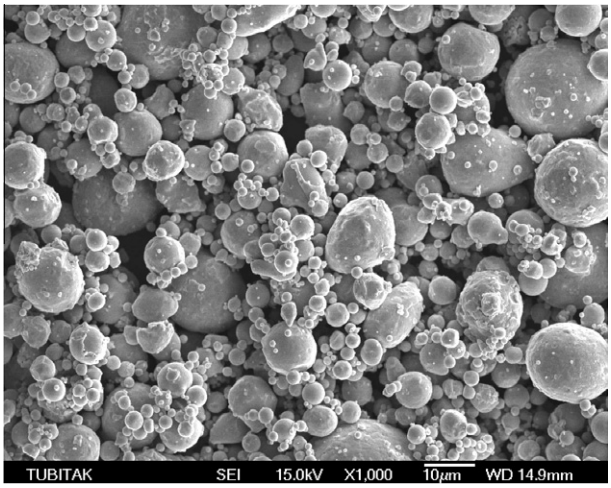

Fig. 2. SEM image of alloy 625 powder.

Table 2
Physical characteristics of alloy 625 powder.

Producer	Sandvik Osprey Ltd.
Production method	Gas atomization
Particle shape	Spherical
Tap density (g cm^{-3})	5.3
Pycnometer density (g cm^{-3})	8.42
Particle size distribution (µm)	
D_{10}	3.7
D_{50}	11.1
D_{90}	26.7

It was understood that the sintered tensile samples that have given the highest density were kept at 1150 °C for 2 h after being done some experiment series as specified in the literature for alloy 625 [25] and solution treated and cooled down in the water. The solution treated samples were aged at 745 °C for different times. Sintered, solution treated and aged samples were measured for microhardness by means of Vickers scale using 100 g load for 10 s via Shimadzu equipment.

Initial powder, sintered samples and thermally treated samples were kept subject to XRD analysis on the equipment, Rigaku XRD-6000, by use of Cu X-ray tube (λ = 1.5405) at a scan rate of 0.02/0.4 degree/s. After both sintered samples and samples kept subject to thermal operation are kept to tensile tests at a constant rate