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Powder injection molding of Stellite 6 powder: Sintering, microstructural and mechanical properties

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The purpose of this study was to produce Co-based Stellite 6 superalloy components by using the method of Powder Injection Molding (PIM) and to characterize the microstructural and mechanical properties of the produced components. The experimental studies were started through the formation of feedstock by mixing Stellite 6 powder with a multicomponent binder system. Prepared feedstock was formed by utilizing powder injection molding technique. Then the molded samples were subjected to the solvent and thermal debinding processes. Different sintering cycles were applied to the raw components for the purpose of determining the optimum sintering conditions. The densities of the sintered components were determined in accordance with the Archimedes' principle. The microstructural characterization was performed through scanning electron microscope (SEM) analysis, energy dispersive spectrometry (EDS) analyses, and X-ray diffraction (XRD) analysis. Hardness measurement and tensile test were conducted in order to determine the mechanical properties. The results illustrated that the injection molded Stellite 6 components were composed of fine and equiaxed grains, plenty of carbide precipitates exhibiting homogenous distribution throughout the microstructure formed at the grain boundaries and thus the mechanical properties were considerably high.

1. Introduction

The design and production of high strength materials with good wear and corrosion resistance at high temperatures are very important for the severe service conditions in the automotive, aeronautical, chemical, and petroleum industries [1]. Stellite alloys exhibit an unmatched combination of mechanical and tribological properties with superior corrosion and oxidation resistance at both room and high temperatures [2]. They are among the leading ones of Co based superalloys which are one of the three major superalloy classes [3]. While the applications of Co based superalloys are conventionally intended for nuclear industry mostly [4], the use of Stellite alloys today has spread to different industrial fields such as pulpwood, paper making, petroleum and gas processing, pharmaceutical industry, chemical processing, etc. [5]. One of the leading Stellite alloys having such widespread area of usage is undoubtedly the Stellite 6 that was the first Stellite alloy developed by Elwood Haynes during the early 1900s [6,7].

The properties of Stellite alloys are widely determined by their chemical compositions [8]. While solid solution hardeners such as Cr, W, and Mo contribute to the strength, carbide precipitates are the main strengthening mechanism [8,9]. Type, size, and shapes of carbides affect strongly the properties of Stellite alloys. For instance, larger size carbides generally provide with the acquisition of higher strength [10]. The formation of carbides, having significant effects on the properties, is influenced by the type and amount of alloy elements (particularly carbon), the production method, sintering temperature and cooling rate and also operational parameters [2,3]. In the production of Co based alloys by using casting technique, important precautions or additional heat treatments [13] are required so as to prevent or solve problems [11,12] such as porosity, segregation, coarse grain size, eutectic carbide network between the dendrites, etc. It is difficult to form Co based alloys having high rate of carbon even at high temperatures [14]. While the industrial demands shift the usage area of Stellite 6 alloy to higher stress applications, the production processes could be adapted in such a way to provide the tribomechanical properties needed [15].

PIM is a production method which enables to produce products with high dimensional accuracy in such a way to have excellent, fine grain structure and non-anisotropic mechanical properties [16]. Despite being a powder metallurgy process, PIM enables to obtain higher rate of density than those components produced

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through conventional powder metallurgy (PM) methods and thus more superior mechanical properties. Complex shaped components that are difficult to produce by using conventional production methods such as casting, forging, machining, and die pressing can easily be transformed into products by means of PIM. The high surface quality and the fine grain structure of the components produced with high measurement precision through the PIM technique provide highly superior properties since they exhibit a homogenous distribution throughout the microstructure of the chemical compound [17]. Recently, PIM of superalloy components for various applications has attracted attention [18–21].

There is no information concerning the microstructural and mechanical properties of Stellite 6 materials produced by using PIM in the literature. For this reason, production of the component from Stellite 6 superalloy powder by using PIM technique was performed in this study. It was aimed to both determine the optimum parameters for the production of Stellite 6 alloy by using PIM and to characterize the microstructure and mechanical properties of the produced components. Hence, injection molded Stellite 6 green components were kept to different sintering cycles to characterize them. Characterization of samples was conducted by SEM, XRD, elemental analyses and mechanical tests. The experimental studies have been carried out to determine the microstructure, and mechanical properties of this alloy. Furthermore, the data obtained from the characterization were compared with properties of Stellite 6 alloy produced by using different methods.

2. Materials and method

Fig. 1 illustrates the SEM image of the prealloyed Stellite 6 superalloy powder used in the experiments. It was seen in the SEM images that Stellite 6 powders had a spherical form. Fig. 2 shows the curve obtained from the particle size distribution analysis conducted by using Malvern Mastersizer instrument. While Table 1 illustrates the chemical composition of the Stellite 6 powder, Table 2 illustrates its some physical properties. The experimental studies were started by preparing the feedstock. The feedstock was obtained by mixing Stellite 6 powder through a multicomponent binder system. The mixing process was performed in a specially designed mixer under vacuum and at 170 °C for half an hour. The feedstock included 62.5 vol% Stellite 6 powder and 37.5 vol% binder. The multicomponent binder system consisted of 69 wt% paraffin wax (PW), 20 wt% polypropylene (PP), 10 wt% carnauba wax (CW), and 1 wt% stearic acid (SA). Rheological behavior of the Stellite 6 feedstocks was measured by using a rotational viscometer Physica MCR51 (Anton Paar, Austria) at shear rates from 10 to 1000 s^{-1} at 130–170 °C. The value of viscosity is given based on the shear stress divided by the shear rate.

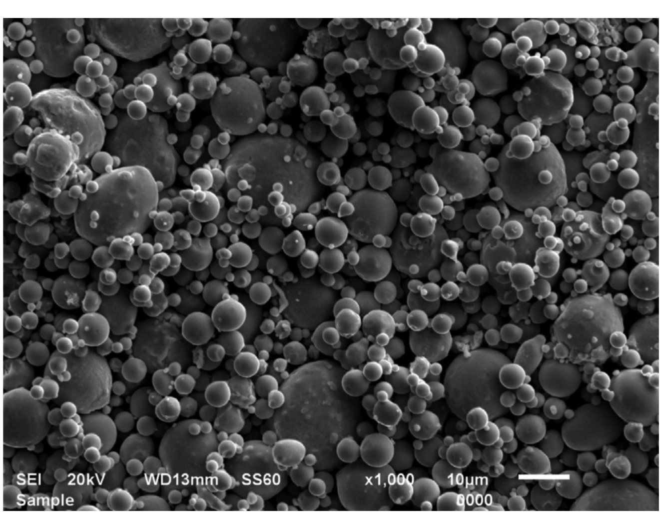

Fig. 1. SEM image of the Stellite 6 powder.

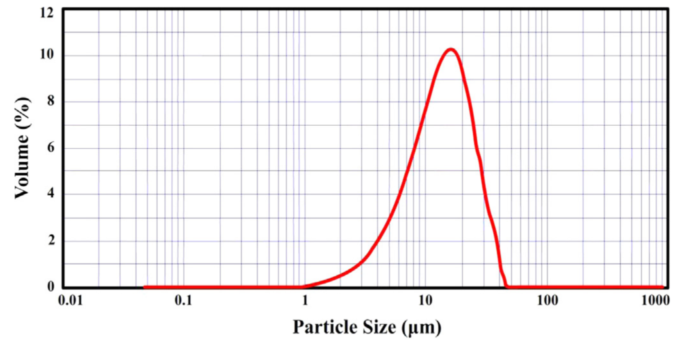

Fig. 2. Particle size distribution of Stellite 6 powder.

Table 1
Chemical composition of Stellite 6 powder.

Weight (%)										
Co	**Cr**	**W**	**Ni**	**Mn**	**C**	**Si**	**Fe**	**Mo**	**P**	**S**
58.438	30.3	4.5	2.0	1.17	1.15	1.02	0.86	0.55	0.008	0.004

Table 2
Some characteristics of Stellite 6 powder.

Properties	
Producer	Osprey
Production method	Gas atomization
Particle shape	Spherical
Particle size (μm)	
D_{10}	5.3
D_{50}	13.8
D_{90}	27

After the feedstock was granulated, it was molded in the form of tensile specimen at 12.5 MPa and for a holding time of 20 s. in the injection device. The temperature of the injection device's barrel and nozzle sections was adjusted to 170 °C during molding. The tensile specimens molded were subjected to a two-stage debinding process, as solvent and thermal. Being heated to 60 °C in the solvent debinding process, the samples were kept for 6 h in the heptane. The thermal debinding operation was implemented by using Al_2O_3 substrate in a high purity argon atmosphere based on the thermal cycle in Fig. 3.

Differential scanning calorimetry (DSC) test was performed in order to determine the sintering behaviors of the debinded samples. The DSC analysis was carried out by heating the Stellite 6 powder at heating rate of 10 °C/min until 1400 °C and at 100 ml/min flow rate under high purity Ar gas atmosphere in a Setaram Labsys Evo device. Al_2O_3 was used as a reference material. In the light of the data from the DSC, the samples were sintered with holding time of one hour within the temperature range of 1200–1300 °C. The sintering processes were carried out by using Al_2O_3 substrate under high vacuum in Protherm tube furnace. 10 °C/min fixed heating and cooling rate was used in the sintering process. Fig. 4 illustrates the images of the samples taken after the molding, debinding, and sintering stages.

The densities of the sintered samples were measured according to the Archimedes' principle by using Precisa XB 320M precision balance. The XRD analyses were carried out in the Rigaku Ultima IV X-Ray Diffractometer device by using Cu X-ray tube ($\lambda = 1{,}5405$)