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The effect of nano-sized stainless steel powder addition on mechanical and physical properties of micropowder injection molded part



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ABSTRACT

Micropowder injection molding (µPIM) is a new technology that has potential in the mass production of microcomponents. A bulk material of nanoparticles possesses completely different properties from those of large-sized particles. The main objective of this study is to study the effects of nano-sized powder addition on the µPIM process of powder-polymer mixtures for the fabrication of miniature parts. The binder systems consist of polyethylene glycol (PEG), polymethyl methacrylate (PMMA), and stearic acid (SA) with different powder loading blended with powders. The results indicate that increasing the nanopowder content to 30 wt.% increased the powder loading and decreased the injection and sintering temperatures. The sintered parts had densities of 96% of the theoretical value. High physical and mechanical properties of the sintered specimen were achieved with the 30 wt.% nano-sized powder sintered at 1200 °C at a heating rate of 5 °C/min under vacuum atmosphere. A significant reduction of the surface roughness of the sintered parts with only micropowder ($S_a = 1.002 \mu$ m). Using nanopowders, the hardness also increased from 182 HV to 221 HV with a linear shrinkage of approximately 9%, which is less than that of the micropowders (18%).

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1. Introduction

Micropowder injection molding (µPIM) is a new technology that has potential in the mass production of metal and ceramic microcomponents because the conventional process of metal injection molding is not suitable for the fabrication of microstructures with a high aspect ratio [1,2]. The µPIM process consists of four main steps: mixing, injection molding, debinding, and sintering. In the first step, namely, the mixing process, powder is mixed with a binder at a selected volume ratio aimed at forming the ceramic or metal. The feedstock resulted from the mixing step is molded in order to produce a "green" compact. When molding is finalized, the binder keeps the particles in place. In the debinding stage, the compact is processed to nearly full density by partially removing the binder component. In the final step, sintering is done aimed at producing the desired mechanical traits. [3–6]. In the µPIM process, components fall into one or more of the following three classes [7,8]: (1) micro-parts: sections that are of a maximum size below 10 mm with features in the micron range; (2) microstructured parts: sections with dimensions between several millimeters and several centimeters with three-dimensional (3D) microstructures located on one or more surface areas; and (3) micro-precision parts: sections of unlimited size but with tolerances in the micron range or smaller.

One main rule for µPIM is that the minimum feature sizes must be at least ten times that of the mean particle size. If smaller features are required for fabrication, then finer powders such as nano-sized powders must be used [9,10]. A bulk material of nanoparticles possesses completely different properties from that of large-sized particles because of the large surface-to-volume ratio. Small particles provide some advantages: (a) a higher aspect ratio, smaller structural details, and better microstructural shape retention; (b) isotropic behavior; (c) better surface finish; (d) strong adhesion; and (e) excellent wear resistance. Keeping the grain size small in relation to the dimension of the smallest microfeature is necessary. Consequently, a shift in raw materials from the approved sub-micrometer range to the nano-sized scale must occur. However, the advantages of using nano-powders in the PIM process include the increase in the comparative density at a low sintering temperature, the decrease in the sintering temperature, the reduction of the grain size of sintered bodies, the increase of the hardness value, and the improvement of the surface properties [9,11,12]. Increasing the packing density of the starting powders is also one of the effective routes to achieve a high



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sintered density and dimensional precision. However, fine powders are also likely to present difficulties in attaining a high packing density due to particle agglomeration [7]. Using a bimodal powder system can help to overcome this problem. For two spherical powders with a large size difference but the same theoretical density, the corresponding weight fraction of large particles for maximum packing is less than 30 wt.% of the smaller particles. The expected fractional packing density would be 0.87 or 87% [13]. Therefore, in this work, a maximum of 30 wt.% nanopowder has been added to the micropowders. The primary objective of this work is to study the effects of nano-sized powder additions to binder-powder mixtures on the fabricated microcomponents using micropowder injection molding.

2. Experimental details

2.1. Materials

(a) Powder

The metal powder used in this study was stainless steel 316L (SS316L). In this study, the two sizes of SS316L powders, with average sizes of 150 nm and 5 um reported by supplier, were purchased from EPSON ATMIX Co., Ltd. (Japan) and Hongwu Nanometer Co., Ltd. (China). Particle size and distribution of metal powders were measured by Malvern Mastersizer 2000 for micro-sized powders and Malvern Zeta Sizer for nano-sized powders. The pycnometer density of the powders was measured using a Quantachrome ultrapycnometer and also tap density of powders was evaluated using the Hall flowmeter. Field-emission scanning electron microscopy (FESEM) was used to determine the physical geometries and the elemental contents of the SS316L powders, as shown in Fig. 1. The tap and pycnometer density of the powders were 4.5 and 8.055 g/cm³ for micropowders. The tap and pycnometer density of the nanopowders were 1.67 and 8.49 g/cm^{3,} respectively. As Fig. 1 shows, the average diameter of the micro and nano-sized SS316L powders was 7.5 µm and 401 nm, with almost spherical and spherical shape, respectively. There is a difference between supplier technical specification report and our analysis in size of nano-sized powders since nano-sized powders tend to agglomerate due to its high surface area. Therefore, it must be deagglomerate before being mixed with binder using milling and heating of powders.

(b) Binder

To avoid using hexane or heptane when the binder extraction is performed, efforts have been centered on finding new types of binders that would be removed by a solvent such as water, which is environmentally favored [14–18]. Therefore, the binder system selected was based on 73% PEG, 25% polymethyl methacrylate (PMMA) and 2% stearic acid (SA). To facilitate lubrication and blending, stearic acid was used as an activator surface or surfactant. The PEG (supplied by Essex, UK) used in the present study has an average molecular weight of 3500. PMMA was purchased by Alfa Aesar, UK, and SA was supplied by Systerm-Chempur, Malaysia. The evaporation temperature was determined from thermogravimetric analysis (STA 449 F3 NETZSCH), and the melting point was obtained via differential scanning calorimetry (DSC) (DSC 1 Mettler Toledo).

2.2. Mixing step

A Fritsch Pul-verisette-6 planetary ball mill with a ball-topowder weight ratio of 4:1 was used to pre-mix the nano and micropowders. With the milling medium and balls in ethanol, the powder was sealed to prevent oxidation. Then, the mixture was ball milled for 3 h at 100 rpm. Then, to evaporate the ethanol, the mixed powders were dried in a vacuum oven at 100 °C for 24 h. Before mixing of the binder and powder in the µPIM process, the ratio of the binder to powder must be measured as a critical powder loading concentration (CPVC). The CPVC was determined using the oil absorption modified technique (ASTM: D-281-12) [16,19]. In this modified method, the powder is mixed with an oil, which is added at a constant rate, the change torque is recorded. CPVC determined by the volume at which the maximum torque occurs [19]. After achieving a good nano-micropowder mixture and determining the CPVC, to create the feedstock (Binder-powder mixture), the mixture powders, PMMA and SA, were mixed using



Fig. 1. SEM images of SS 316L: (ai) micro-size and (bi) nano-sized powder with particle size distribution of (aii) micro-size and (bii) nano-sized powder.

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