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Binder system for STS 316 nanopowder feedstocks in micro-metal injection molding

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Abstract

The stainless steel (STS) 316 nanopowder (average particle size of 100 nm) were mixed with thermoplastic binder system to produce feedstock for micro-metal injection molding (μMIM) by using lost mold method. The combination of paraffin waxes, bee's waxes, carnauba waxes, ethylene vinyl acetate (EVA), polypropylene (PP) and stearic acid were chosen as binder systems with various formulations adopted to attain optimum feedstock properties. Three different kinds of binder systems of EVA-based, PP-based, and wax-based binder system were deeply investigated. It is found that the wax-based binder system performs the lowest viscosity and heat capacity as well as greater pseudo-plasticity than other binder systems. The feedstocks were injected into SU-8 micromold which was fabricated using photolithography technique. The feedstock which was mixed with wax-based binder system performed the smallest deformation. This result inferred that wax-based binder system is appropriate as binder materials for very fine metal powder applied in μMIM. In this paper we also demonstrated the application of nanopowder on sintering process and showed that the sintered part using nanopowder provides less surface roughness than one produced using micron-sized metal powder.

Keywords: Binder system; Micro-metal injection molding; Stainless steel 316 nanopowder

1. Introduction

Recently micro-metal injection molding technique was selected to produce micro-component due to its versatility on manufacturing geometrically complex component. For micro-parts fabricated by MIM, surface roughness is important for two reasons. First, the tolerances of micro-parts are decreasing toward the range of surface roughness. Second, applications of micro-parts have strict requirements on surface roughness, because surface roughness affects friction and wear at the interface. A very fine metal powder was successfully fabricated to achieve good surface roughness. The small sizes of the metal powder are required appropriate binder system. A very fine metal powder promotes higher viscosity behavior of the feedstock than bigger powder sizes.

Binder system selection for very fine powder is important to achieve low viscosity of the feedstock. It is desired to complete filling during injection stages. The binder system in injection molding consists of major binder, minor binder, and various processing aids such as surface modifier and plasticizer [4]. Usually, the major binder, which controls the general properties of the mixture and the green bodies, are first selected. The selection of binder system for STS 316 nanopowder was observed in this work.

2. Experimental setup

2.1. Mixing

The spherical shapes STS 316 nanopowder with the average diameter of 100 nm and were selected in this experiment to produce small parts. The STS 316 nanopowder and the binder system were mixed in *Brabender plastograph* mixer for 1 h at 170 °C. Mixing speed was kept constantly at 60 rpm. Multi-components of binder system were used in this experiment. Table 1 shows the binder systems compositions and feedstock identification of each binder. During mixing the nanopowder had to be fed below 100 °C in order to prevent burning. Mixing torque was recorded during mixing, since it represents the viscosity value.

2.2. Micromold

The SU-8 2100 which manufactured by Microchem was utilized to fabricate micromold by UV photolithography process. The micromold consisted of two layers of photoresist (PR). There was PMMA (positive PR) layer in the bottom

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Table 1
Binder composition

	EVA-based binder system (%)	PP-based binder system (%)	Wax-based binder system (%)
Paraffin wax	30	30	25
Carnauba wax	10	10	20
Bees wax	10	10	20
Ethylene vinyl acetate (EVA)	45	–	25
Polypropylene (PP)	–	45	5
Stearic acid	5	5	5
Feedstock identification	FSA	FSB	FSC

side to prevent sticky between feedstock and glass substrate. Second layer was SU-8 (negative PR) as a mold body.

Glass substrate was ultrasonically cleaned by piranha solution (H_2SO_4:H_2O_2 = 3:1), followed by cleaning with de-ionized water, then heated at 200 °C for 30 min in heating oven. About 0.5 ml PMMA was dropped to glass substrate and spun at 1000 rpm followed by baking at 170 °C for 30 min and cooled slowly in oven. About 0.5 ml SU-8 was dropped on the top of PMMA layer and was spun at 1000 rpm during 30 s. Two steps soft bake were used to remove SU-8 solvents at 65 °C for 10 min, it also made SU-8 re-flow and made flat surface. SU-8 then transferred into oven and heated until 95 °C for 90 min and then cooled slowly. SU-8 exposed in near UV light (λ = 350 nm) with exposing dose of 1000 mj/cm^2. Post-exposed bake was applied at 65 °C for 10 min and 95 °C for 30 min and cooled slowly. Specimen was immersed in an EBR-PG to remove covered area for 18 min to obtain micro-cavities. The obtained hole cavities were 400 μm in diameter and the depth of the micro-cavities was around 200 μm.

2.3. Feedstock characterization

The melting temperatures of feedstock were measured on *TA instrument* Differential Scanning Calorimeter (DSC) from 0 to 300 °C with heating rate 10 °C/min in nitrogen atmosphere. The thermal degradation of the binder materials and the SU-8 micromold were analyzed on TA instrument Thermo Gravimetric Analyzer (TGA) at 30–800 °C, with heating rate of 10 °C/min under inert atmosphere. Ares Rheometer was employed to examine the viscous-elastic behavior of different binder system at 150 °C.

2.4. Injection system

The injection process to fill the micromold with feedstock was illustrated in Fig. 1. The *Carver* uniaxial pressing was utilized to inject the feedstock entering the cavities. The feedstock was injected into the cavity at 150 °C with injection pressure of 6 MPa. The NMP (1-methyl-2-pyrrolidon) was used to dissolve the SU-8 mold in order to obtain green part.

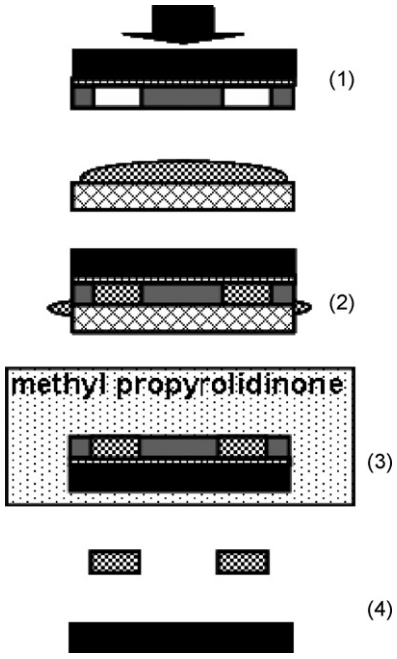

Fig. 1. Injection process.

3. Experimental results and discussion

3.1. Mixing

The mixing torque of three kinds of binder composition was examined by *Brabender plastograph* as shown in Table 2. The mixing torque of FSA at 50% powder loading was highest, and then followed by FSC and FSB. It indicated that FSA more difficult to be mixed than FSB and FSC. The homogeneity of the feedstock occurred at low mixing torque, since the binder material and powder easy to be mobilized. From the torque and rheological analysis, higher torque indicates higher viscosity.

3.2. Feedstock characterization

3.2.1. Thermal properties

A Thermo Gravimetric Analyzer (TGA) was utilized to examine the thermal degradation of the feedstock FSC and the SU-8. Since the constituent of three kind of binder system are similar, it assumed that thermal degradation behavior is also similar. The sample was heated from room temperature to 800 °C, with a heating rate of 10 °C/min. The difference of degradation rate shown in TGA curve (Fig. 3), it was due to the binder materials containing multi-component of waxes, EVA and PP. The binder materials started to degrade at 200 °C and ended completely at 500 °C. The waxes constituents were evaporated at range temperature 200–300 °C. The EVA and PP were evaporated above 300 °C, and increase rapidly above 400 °C. Therefore we suggested thermal treatment of this binder composition such at the mixing stage and the injection temperature must be applied below 200 °C. Thermal debinding temperature should be higher than 500 °C in order to remove all binder components.

Fig. 2(b) shows the DSC curve of the feedstock. The FSA performed two endothermic peaks in DSC curve which indicated the melting point. The first peak at 56.79 °C is melting point of wax components, and second peak at 86 °C

Table 2
Feedstock properties

	FSA	FSB	FSC
Average mixing torque (N m)	22.12	4.67	5.21
n (flow behavior index)	0.15	0.27	0.12
A (constant, Pa)	5.5×10^5	1.8×10^5	3.5×10^5
Viscous-elastic properties	$G' > G''$	$G'' > G'$ (below 10 rad/s), $G' > G''$ (above 10 rad/s)	$G' > G''$
Melting temperature (°C)	56–86	56–149	56–149