M3-015 Sintering of Stainless Steel Nanopowders for Micro-component Part Applications

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ABSTRACT

Micro components have potential market in new millennium. Micro metal injection molding is one of manufacturing technique to produce micro parts. STS 316 nanopowders (average diameter of 100 nm and spherical shape) was mixed with thermoplastic binder to compose the feedstock which have to be injected in to mold. Debinding and sintering process was carry out to increase mechanical and physical properties. Utilization of nanopowders to construct micro component will produce more detail structure and will giving extraordinary properties due to very fine structure. Several problems were occurred during sintering stainless steel 316 nanopowders. First is difficulties on removing oxide in sintered part, second; the existing secondary phase, third: necking growth of nanopowders is very fast which make surface are rapidly closed. It will prevent gas release and produce porosity. Sintering parameter should be controlled to optimize the potential of nanopowders.

Keywords: Sintering, Stainless steel nanopowders, Micro-component

1. Introduction

Micro-systems and related products will have increasing applications and potential huge markets in the new millennium. Micro-system is seen one of the key and fastest growing sector of technology, and researcher and companies have been focusing their attention on miniaturizing metal component that have application in micro-systems technologies(MST).

The definition of micro-component is structures which have a weight in the range of few grams, features less than 1000 µm (common rage is 100 – 500 µm) and exhibiting dimensional tolerance in the micro meter range. Micro metal injection molding (µMIM) and hot embossing has been developed in recent years [1,3]. The hot embossing process is becoming a dominant fabrication method for nano/microstructures when low cost and high throughput are desired [4-12]. It is require submicron particle in order to achieve micron scale.

Sintering is thermal treatment to bond the particle in to coherent, solid mass. The bonds between particles grow by the motion of individual atoms via either solid-state or liquid phase events. Sintering temperature usually occurs close to melting temperature. The nanopowders have a tendency lower sintering temperature than micro powder. For example Fe nanopowder can be sintered at 1000°C. The Fe micropowder (5-10μm) sintering temperature is 1200°C under hydrogen atmosphere [13]

Nanopowders are very fine particle with grain size less than 100 nm. Due to the size and surface effect made it have novel mechanical, electrical and optical characteristics. The nanopowder gave some advantages: a) provide to smaller structural details, higher aspect ratio and better shape retention of microstructure [1,2] b) Give fairly isotropic behavior c) better surface finish.

In this work we observed sintering behavior of stainless steel nanopowders as one of processing stage in the manufacturing process of nanopowders to fabricate micro component by using Micro metal injection molding.

2. Materials and Experimental method

2.1. Materials

STS 316 nanopowders with an average diameter of 100 nm and spherical shape were selected in this experiment as shown in fig. 1. The STS 316 nanopowders and binder system were mixed in a *Brabender plastograph* mixer for 1 hour at 170 °C to produce feedstock. It is raw material for Micro metal injection molding process. The mixing speed was set at 100 rpm. The binder system which utilized in this experiment is consists of multiple components of polymer such as waxes, ethylene vinyl acetate (EVA) and polypropylene (PP). Feedstock with 52% powder loading was molded in sintering specimen.

2.2. Debinding and Sintering

Debinding and sintering program can be seen on fig. 2. Debinding process was performed at 300°C for 2 hours, and 550°C for 2 hours to remove the binder material before sintered. Sintering was applied on the alumina tube furnace under hydrogen atmosphere with various temperatures, 900, 1000, 1100, 1200, and 1300°C.

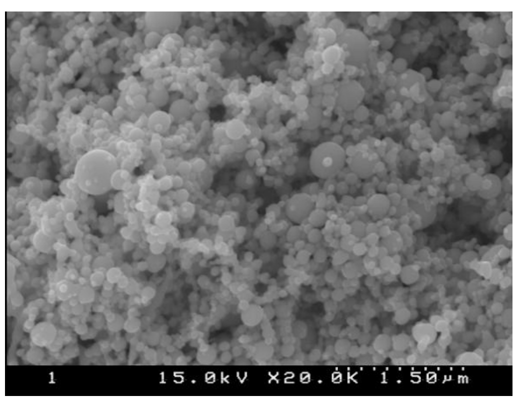

Fig.1. 316 Stainless Nanopowderss

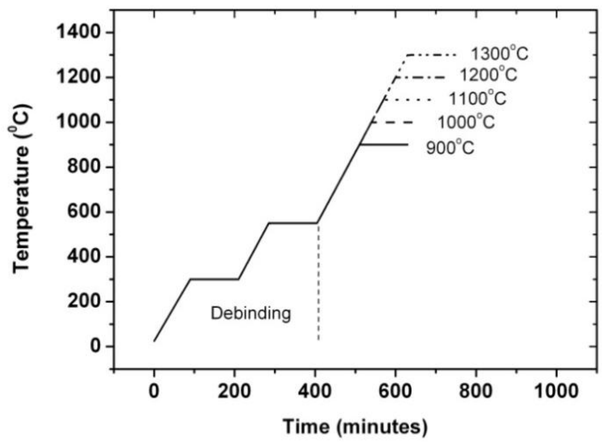

Fig. 2. Sintering program with constant heating rate 3.3°C/min.

3. Result and Discussion

The lower necking formation and growth of nanopowders is due to higher surface area. Secondary phase was observed under Scanning Electron Microscope (SEM) as globular particle which is show in fig. 3. It was found above 1000°C. This particle was reducing the ductility of sintered part with poor bonding strength with austenite matrix. This particle sizes was increase and was joined each other with sintering temperature. It shows that secondary particle has higher melting point than STS 316.

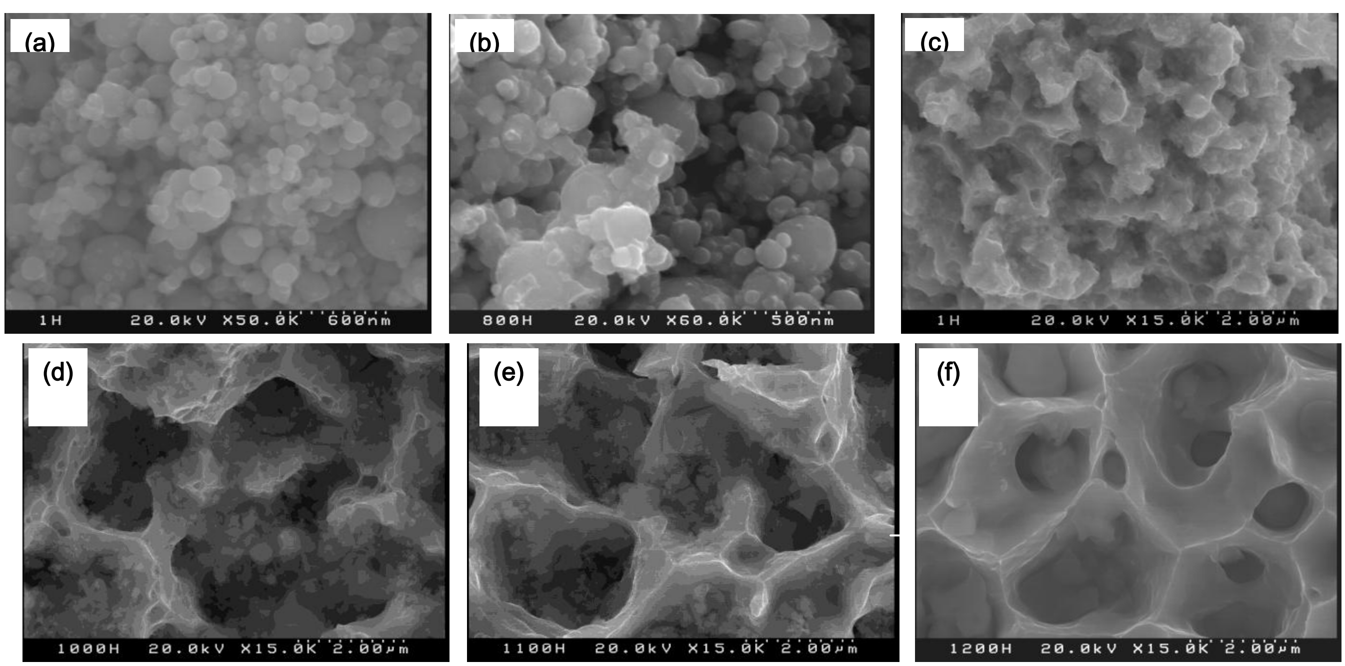

Fig.3. Fractography of sintered specimen (a) 700, (b) 800, (c) 900, (c) 1000, (d) 1100, (e) 1200, (f)1300°C

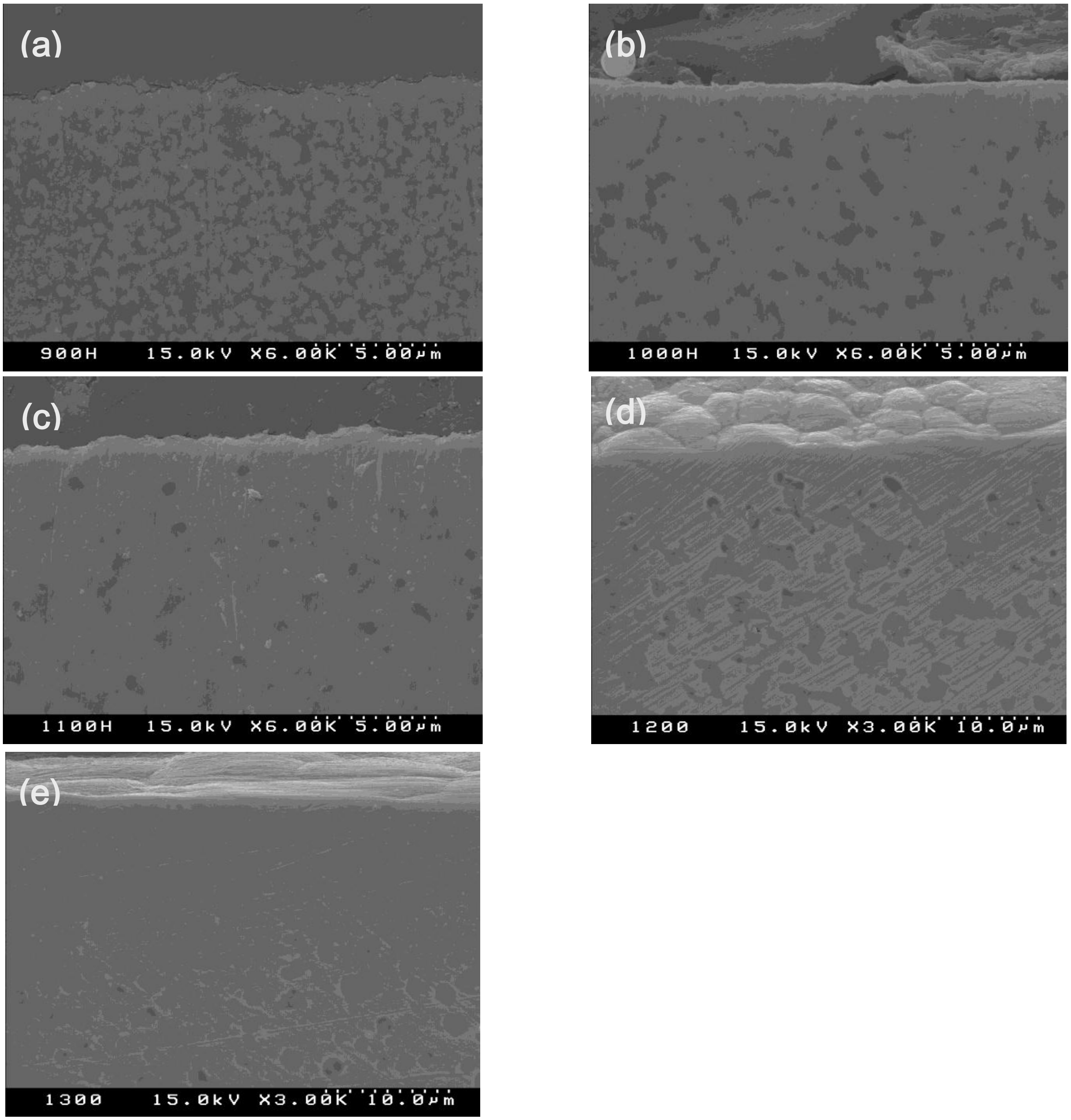

Fig.4. Cross section in surface area of sintered part,(a) 900, (b) 1000, (c)1100, (d) 1200, (e)1300,

In the first stage, reductive atmosphere react with top surface of the sintering specimen. The second stage is necking and growth of each nanopowders, due to the very fine size necking and growth rapidly proceed and form full dens layer. Full dens layer occur on the top surface prohibit atmosphere contact to oxides. Slow heating rate was applied to reduce necking and growth and formation of full dens layer. It will give chance for reductive atmosphere react with oxide. Slow heating rate produce higher densification.

The existing of oxides on the sintered part derived from the oxide layer on the nanopowders and oxidation during thermal debinding and sintering. It is require strong reduction atmosphere during sintering to eliminate the oxides.

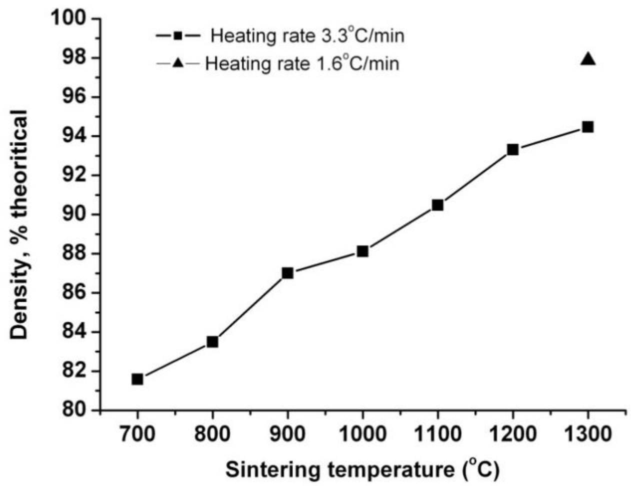

Fig.5. Sintering temperature vs density

Sintering density of nanopowders is increase with temperature. With this sintering system full theoretical density cannot achieve. It is due to the existing of the oxides with low density and the formation of full dense layer on the surface of sintered part as shown in fig.4. Low densification doe to existing of oxides phase, reduce densification rate. The oxides has high melting point therefore need higher temperature to produce necking on the oxides. For this reason controlled sintering atmosphere should be used. Reductive atmosphere was used to react with oxides layer on nanopowders.

Secondary phase was observed under Scanning Electron Microscope (SEM) as globular particle. It was found above 1000°C. This particle was reducing the ductility of sintered part with poor bonding strength with austenite matrix. This particle sizes was increase and was joined each other with sintering temperature. It shows that secondary particle has higher melting point than STS 316.

Secondary phase composition was observed with Electron Disperse Spectroscopy (EDS) show in Table 1.Two kind of secondary phase was found, “Chrome rich phase” and oxides. This result was support with XRD pattern that reveal FCC crystal structure which correspond to austenite, BCC structure which correspond to “Chrome rich phase” and ferro-chrome oxides as shown in fig.6.

The existing of “Chrome rich phase” is due to high temperature exposure for certain time that make Cr atoms diffusion. Lower sintering temperature is required to prevent this phase formation. The existing of ferro-chrome oxides on the sintered part derived from the oxide layer on the nanopowders and oxidation during thermal debinding and sintering. It is require strong reduction atmosphere during sintering to eliminate the oxides.

Table 1. EDS analysis

Element	Atomic%	Atomic%	Atomic%
Spot	**1**	**2**	**3**
O K	37.13	21.46	0.00
Cr K	28.22	37.14	44.57
Mn K	13.27	16.89	17.89
Fe K	19.27	22.36	35.49
Ni K	2.12	2.16	2.06

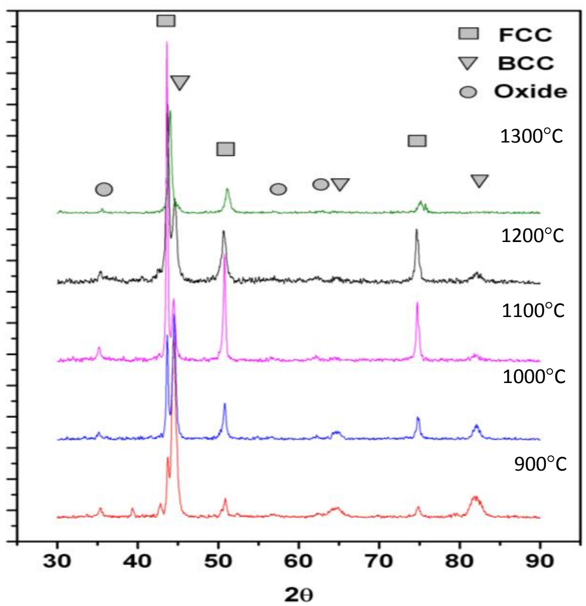

Fig.6. ERD analysis of secondary phase at sintered.

The mail problem of sintering nanopowders is removing oxide particle in sintered part since nonopowder have surface area over 100 time compare with micron size powder. If we assume that oxide layer thickness in nanopowder is 5 nm, it is need a lot of effort to eliminate by increasing sintering temperature. In other hand high sintering temperature will promote formation of secondary phase in stainless steel. Therefore oxide formation in nanopowder should be minimized. Heating and cooling rate on micro part sintering process should be control since the small size is sensitive with temperature shifting.

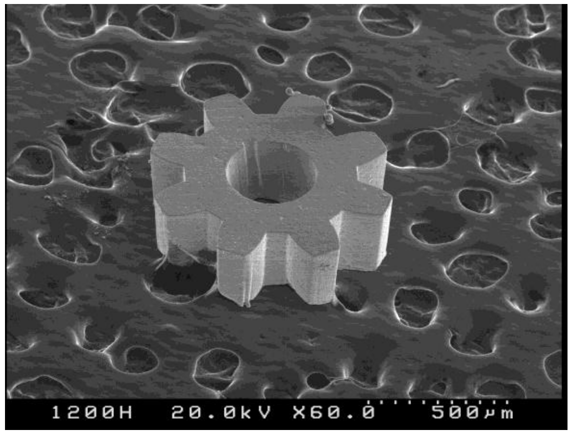

Fig.7. Micro gear specimen with outside diameter 1mm

4. Conclusion

Low densification doe to existing of oxides phase, which reduce densification rate. The oxides has high melting point therefore need higher temperature to produce necking on the oxides. For this reason controlled sintering atmosphere should be used. Reductive atmosphere was used to react with oxides layer on nanopowders. Higher sintering temperature is giving higher densification. In other hand higher sintering temperature will increase grain size, since nanopowders has high surface area that increase atomic movement.

Acknowledgements

The author would like to thank the Center for Nanostructures Material Technology for awarding a research grant 05K1501-00110 under '21 st Century Frontier R&D Program' from the Ministry of Science and Technology, Korea.

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