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The Fabrication of Stainless Steel Parts for MEMs

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The Fabrication of Stainless Steel Parts for MEMs*

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Abstract

A micro-molding process was used to fabricate parts in the 0.1 to 10 mm size range from a stainless steel nano-powder. The two types of molds used were both produced from parts fabricated using the LIGA process so that they had precise dimensional tolerance and straight sidewalls. Rigid PMMA molds were made by injection molding and flexible silicone rubber molds were made by casting. Mold filling was accomplished by mixing the powder with epoxy to form a putty-like material that was then pressed into the mold cavities and allowed to cure. After pyrolysis of the epoxy, the parts were sintered in forming gas. The densification kinetics were measured *in situ* using a video system. Full densification was achieved after 1 hour at 1350°C. The microstructure of the sintered parts was examined using the SEM. The hardness, dimensional tolerance and surface roughness of the sintered parts were also measured.

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The Fabrication of Stainless Steel Parts for MEMs

Introduction

The two most common techniques for producing parts for MEMs, silicon surface micromachining and LIGA, have limitations. Two limitations of Si micromachining are composition and thickness. The available materials set includes only Si, SiO₂, SiC and diamond. Part thickness is generally in micron range. For the LIGA process, the materials set is limited to materials that can be plated such as pure metal and some simple alloys. Plating is a slow process and generates considerable quantities of hazardous waste. Thus an economical technique that could produce parts of more complex metals as well as ceramics and polymeric materials would complement the existing techniques.

One technique with the potential to fabricate parts for MEMs with precise dimensional tolerance and with a very broad materials set is micro molding. In micro molding, a micro-mold is first fabricated that has cavities that are the shape of the desired parts. The mold cavities are then filled with the material of interest, typically in powdered form. The parts are then released from the mold and sintered to achieve high density and strength. Since it is a batch process, micro molding has the potential to be low cost, as long as the cost of the molds is not excessive. Previous work has focused on either powder injection molding¹⁻⁴, a more complicated process, or on filling molds made by deep x-ray lithography (DXRL)⁵, the same process used to make molds for LIGA, which were therefore relatively expensive due to the cost of synchrotron exposure. In the present work, LIGA was used to make the master from which the molds are made so that precise dimensional tolerance, straight sidewalls and high aspect ratio parts can be produced without the expense of DXRL.

Experimental Procedure

Two types of micro-molds were used. The first type, shown in Figure 1a, was made by injection molding of poly(methyl) methacrylate (PMMA) using a nickel master that had been made by the LIGA process. These molds were rigid and had cavities of either 90 or 170 μm deep. The cavities ranged from several mm to several hundred microns with the smallest features in the 10 μm size range. The same pattern was used for the flexible silicone rubber molds. The silicone rubber resin was cast over the nickel master and allowed to cure (see Figure 1b). Molds with cavities up to 400 μm deep were produced.

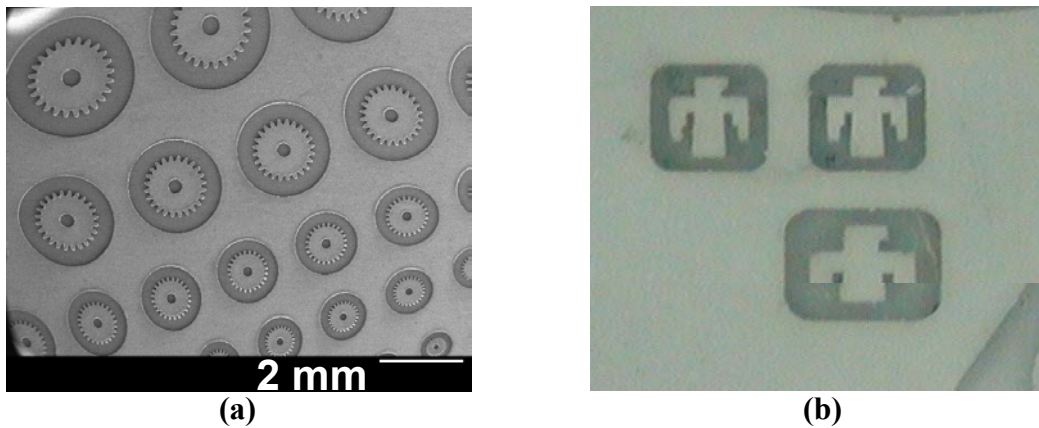


Figure 1. Images of micro-molds. (a) SEM image of PMMA mold and (b) optical image of silicone rubber mold.

To be able to faithfully reproduce features in the 10 μm range using a powder, the particles in the powder must be much finer than the minimum feature size. Commercial stainless steel powders produced for powder metallurgy have average particle sizes of 15 μm or larger and are therefore unsuitable for this application. Therefore, a specially prepared nano-powder of 316L stainless steel was used. This powder was made by a plasma process involving the electro-

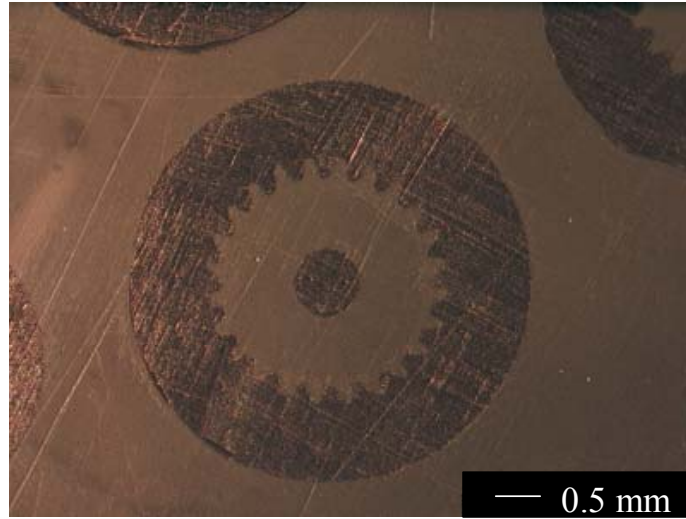
explosion of metal wire (Argonide, Sanford, FL) and had particles in the hundred-nanometer range.

A variety of techniques were investigated for filling the mold cavities with the powder. These techniques can be divided into three groups: dry, semi-dry and wet. The dry techniques included dry pressing and tape embossing. For dry pressing, the powder was first dispersed in a solvent that contained a binder then dried and granulated. The granules were then applied in a layer on top of the PMMA mold and uniaxially pressed at 25 MPa using a sheet of polyurethane between the die ram and the granules to help transfer the pressure into the cavities. The parts were then de-molded by partially dissolving the mold in methylene chloride. A problem with this process was that inter-granule porosity was difficult to eliminate because of the low-pressure limitation of the PMMA mold.

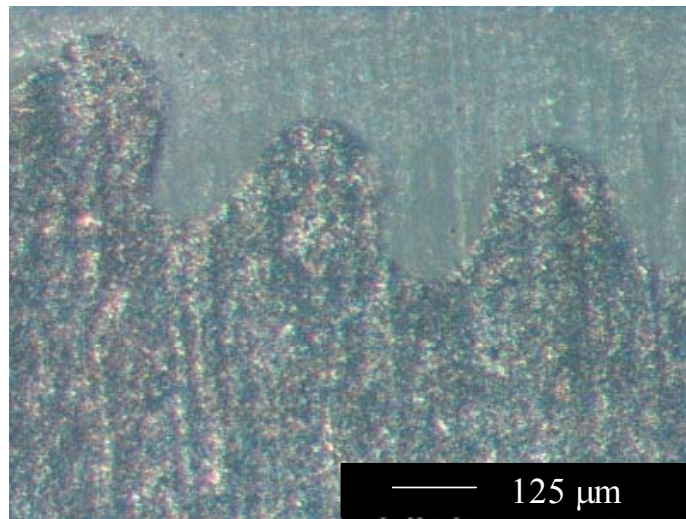
The wet process involved making a dispersion of the powder in a solvent containing dissolved binder and then applying the dispersion to the mold surface and allowing it to dry. One problem with this process was the tendency to entrap tiny air bubbles, especially in small features in the mold. Also, cracking during drying was a problem especially for thicker parts and for nano-sized powders.

Because of the problems with the other techniques, a semi-dry process was developed and used for the rest of this work. In this process, epoxy was used as a curable binder so that the mold could be filled when the epoxy was fluid and then de-molding could be done after the epoxy cured and was thus stronger and insoluble. In this process, the powder was first dispersed in methyl ethyl ketone (MEK) using a dispersant (Disperbyk-180, Byk-Chemie, Wallingford, CT) and an ultrasonic horn. The epoxy resin (Shell 815) was then added (1g per 8 g of powder) and the MEK was removed by evaporation, thus producing a homogeneous mixture of the powder and the resin. The curing agent (TETA) was then added (0.125 ml per g of resin) and mixed in using a mortar and pestle until the mixture resembled putty. The mixture was then pressed onto either type of mold. The mixture could be removed from the mold as a sheet with the parts on one face when the epoxy had cured to the leathery stage. If the epoxy was allowed to fully cure in the PMMA molds, the excess could be sanded off, leaving the cavities fully filled as shown in Figure 2. These parts could then be released using methylene chloride.

(a)



(b)



(c)



Figure 2. Optical micrographs of PMMA micro-molds with 90 μm deep cavities filled with stainless steel/epoxy: (a) top surface (b) top surface at higher magnification and (c) bottom surface taken looking through the mold.

Prior to sintering, the epoxy binder had to be removed. To pyrolyze the epoxy without oxidizing the stainless steel, a low pO_2 atmosphere produced by bubbling 3% hydrogen in argon through room temperature water was used.

A video imaging system was used to measure the shrinkage of the molded parts during sintering. The system had a closed tube with a window on one end to allow control of the sintering atmosphere. A strobe illumination system (Control Vision, Idaho Falls, ID) was used to be able to acquire high contrast images at high temperatures.

Sintered parts were characterized using SEM. Part distortion and part-to-part dimensional variation were characterized using optical microscopy. A profilometer was used to measure the average surface roughness. Hardness values were determined using instrumented nanoindentation data collected on a Hysitron Triboscope with a 3-sided diamond Berkovich indenter tip on a polished cross-section.

Results and Discussion

Figure 3 shows the structure of the starting powder stainless steel nano-powder. The particles were spherical and mostly in the 100 to 200 nm size range, although there were occasional particles up to 1 μm in size. In spite of its small particle size, the powder was quite stable with respect to oxidation. Figure 4a shows that at room temperature, oxidation starts after 3 days and reaches about a 1% weight gain after a week. In Figure 4b the weight gain during heating at 10°C/min in air using the thermal gravimetric analysis system (Netzsch, Selb, Germany) is shown. Oxidation starts above 200°C and is nearly complete around 550°C.

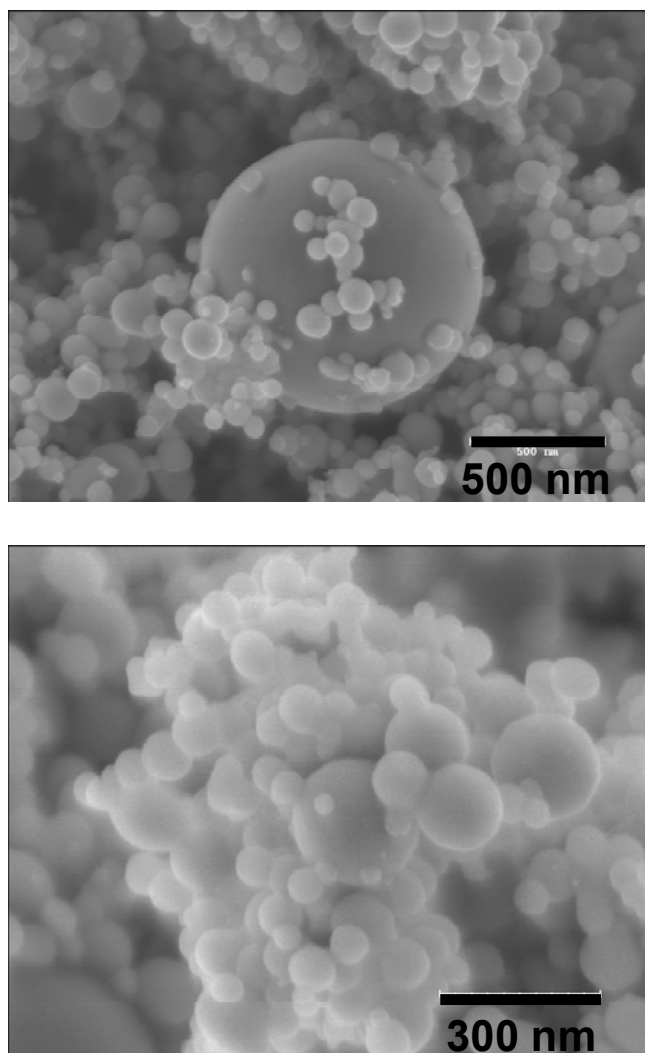


Figure 3. SEM images of Argonide stainless steel powder.

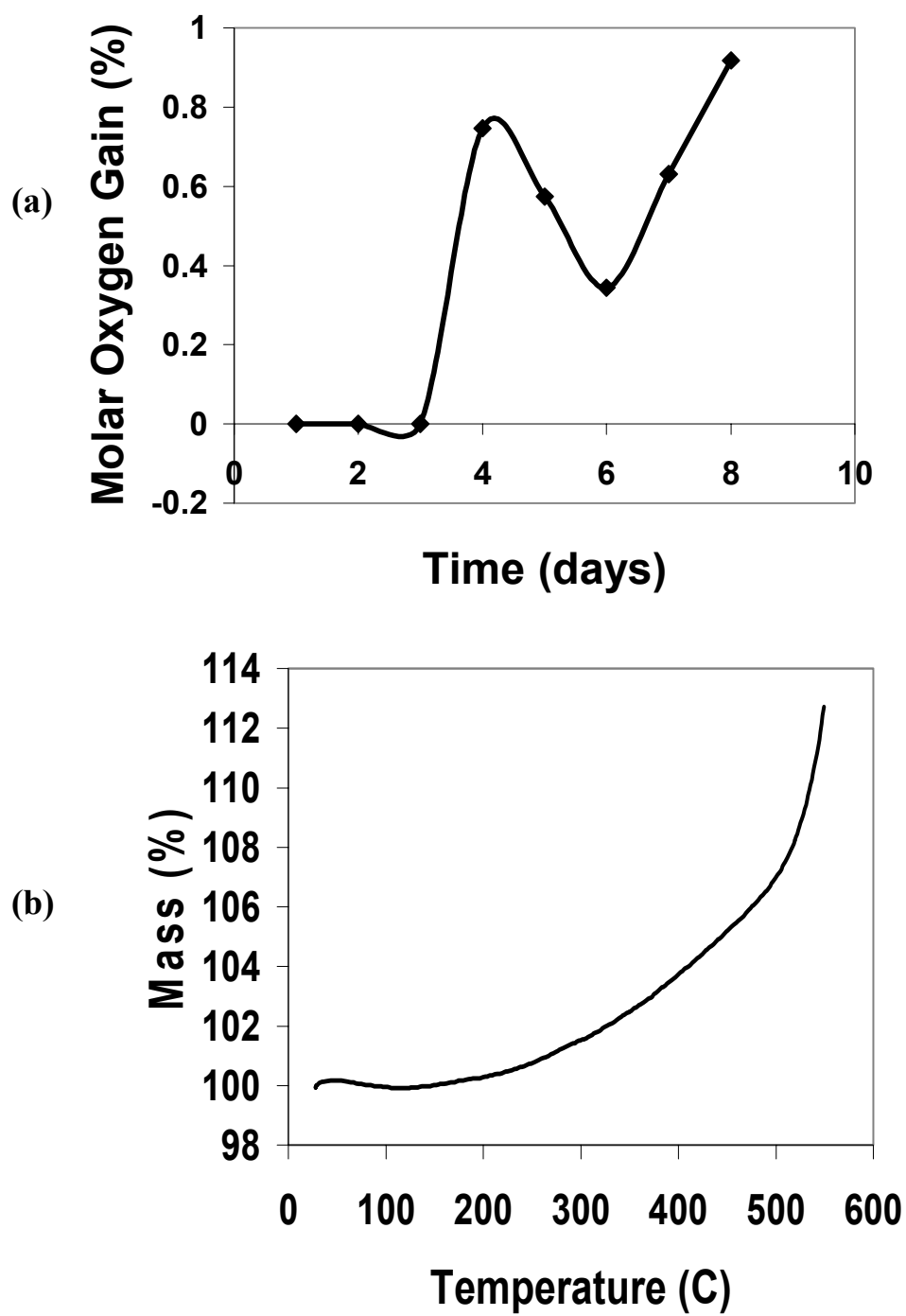


Figure 4. Oxidation of SS nano-powder in air at room temperature (a) in during heating (b).

The linear shrinkage measured *in situ* using the video system is shown in Figure 5. Shrinkage commenced around 1050°C and reached 19% at the start of the soak at 1350°C. An additional 2% shrinkage occurred after an hour at 1350°C. From this amount of shrinkage, a green density of 49% can be calculated, assuming theoretical density was achieved, as is shown below. The low green density is the main reason that the sintering temperature required for densification was higher than expected for a stainless steel with particles this small. Figure 6 is an optical image of a sintered part from a PMMA mold that was held for 1 hr at 1350°C.

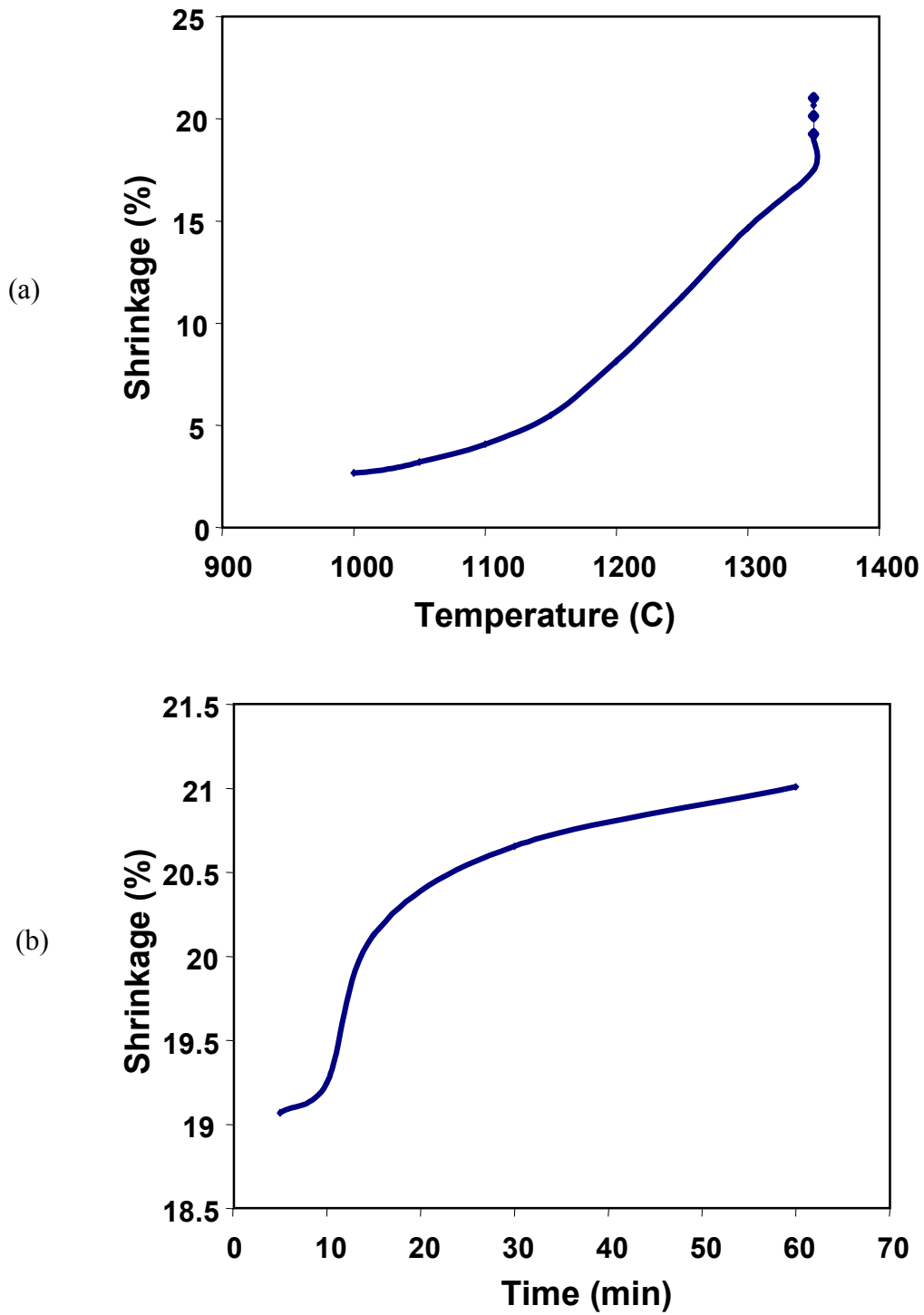


Figure 5. The shrinkage of a stainless steel micropart heated in 3% hydrogen. (a) during the 10°C/min ramp and (b) during the hold at 1350°C.

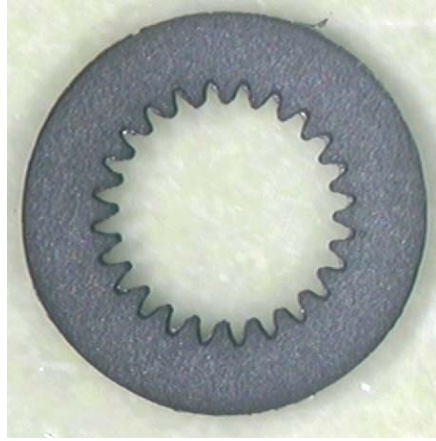
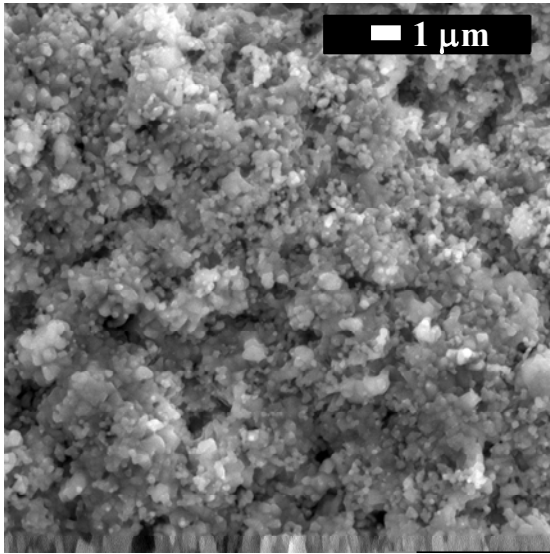
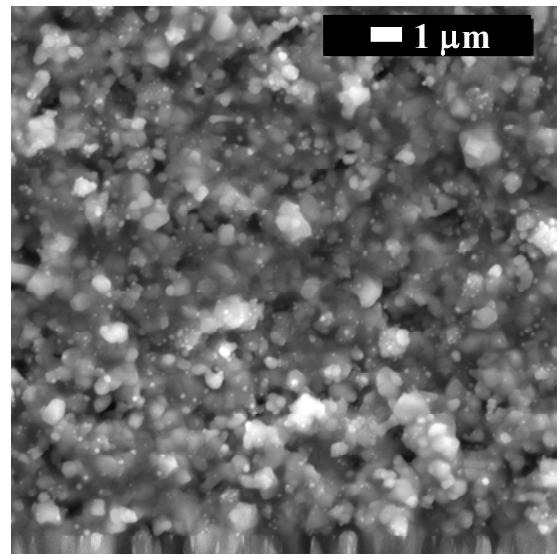


Figure 6. Optical micrograph of sintered stainless steel micro-molded part from a PMMA mold.

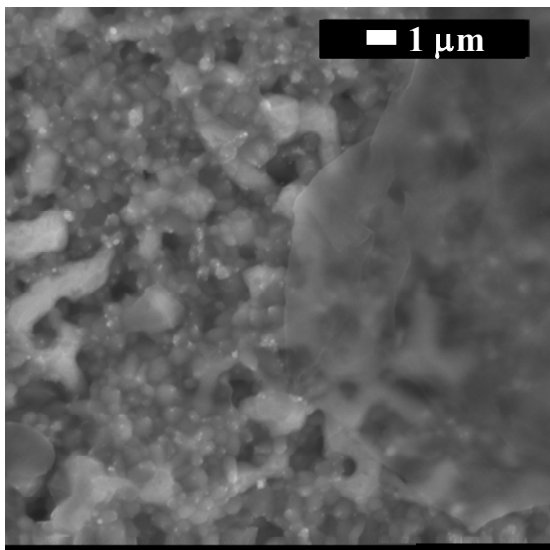
The results of the SEM examination of the microstructure of sintered parts showed that at 1300°C, the particles were still rounded with an average size only slightly larger than the starting particles size (see Figure 7). By 1350°C, the particles were well sintered together with only closed porosity and a 0.5 μm average grain size. Further densification with only slight grain growth occurred after 20 min at 1350°C so that nearly theoretical density was reached with the average grain size less than 0.6 μm . Further heating at 1350°C resulted in rapid grain growth so that the average grain size increased to 9 μm after 1 hr.



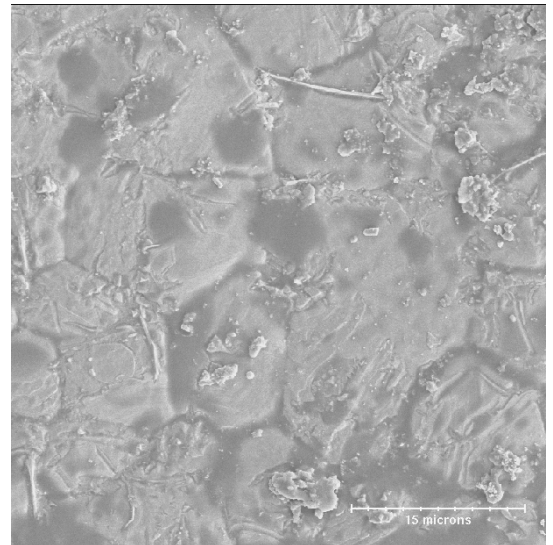
(a)



(b)



(c)



(d)

Figure 7. SEM micrographs of sintered stainless steel micro-molded parts: (a) 1300°C, 0 min, (b) 1350°C, 0 min, (c) 1350°C, 20 min and (d) 1350°C, 1 hr.

The nano-hardness of a part sintered at 1350C for 1 hr was 5 GPa, in line with expectations. The average as-sintered surface roughness (1350°C/1hr) = 300 nm, which is much less than the grain size. The average single part dimensional distortion = 0.45% and the average part-to-part dimensional variation = 2.3%.

Conclusions

Stainless steel parts with features in the 0.01 mm range were fabricated by micro molding of a nano-powder using molds replicated from LIGA-produced masters. Epoxy was used as a curable binder to mold the parts and was removed by pyrolysis in forming gas. Parts were sintered in hydrogen to high density at 1350°C with an average grain size of 0.6 μm . Sintered parts had expected hardness values and reasonably smooth surfaces and small distortion. More work needs to be done to improve part-to-part dimensional variation.

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