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A Fabrication Process of Composite Micro Components using Super Fine Stainless Steel and Ceramic Nano Powders

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1. Introduction

Fabrication of micro components from different monolithic materials such as ceramics, metals, and polymers have been discussed before [Jun et al., 2008; Mohamed et al., 2008; Bong-Kee & Tai, 2008], but hardly any research has been reported in fabrication of composite microparts [Ping et al., 2008; Hany & Kyle, 2009; Su-Jin et al., 2008]. This chapter introduces a novel approach to fabricate stainless steel ceramic composite micro machine components. The two types of composites to be introduced are stainless steel-alumina and stainless steel-titania. Three types of powders were tested, including 5 µm stainless steel, 400 nm alumina and 320 nm titania. Four different compositions prepared for each composite type are 2.5, 5, 7.5 and 10% weight of ceramic. Characterization of composite micro components in terms of slurry preparation process, sintering conditions, shape retention, density, linear shrinkage and micro hardness is reported in detail. The fabrication process is divided into three stages. In the first stage, SU-8 master moulds and their negative replica soft moulds are produced using softlithography process. The second stage includes preparation of composite slurries, filling soft micro moulds and obtaining composite green micro components. In the third stage, the composite green micro components are de-bound and sintered in vacuum and nitrogen-hydrogen mixture (90% nitrogen and 10% hydrogen) respectively at 1350 °C. Afterwards, the properties of sintered micro components are studied in detail.

2. Experiments

2.1 Preparing SU-8 and soft moulds

SU-8 2075 [MicroChem, USA] was used for fabricating 1mm thick master moulds of micro gears, pistons and linkage rods. The SU-8 fabrication procedure are as follows: (i) casting SU-8 resist onto 4 inch Silicon wafer and soft baking at 65C° for 2 hours followed by 95C° for 34 hours; (ii) exposing the coated wafer in Canon PLA-501 FA UV-mask aligner; (iii) post

exposure baking at 65C° for 5 minutes followed by 95C° for 15 minutes; (iv) developing the wafer in EC solvent. After SU-8 master moulds were produced, they were replicated into PDMS soft moulds. Figure 1 shows high quality SU-8 master moulds and their negative soft moulds replicated from the master moulds. Details of the fabrication of SU-8 and replication of soft mould can be found in [Mohamed et al., 2008].



Fig. 1. SEM images of (a) SU-8 master moulds and (b) negative replicas soft moulds.

2.2 Preparing composite slurry and obtaining green components

5 μm 316-L Stainless steel powder with composition of 65.5%Fe, 18.5%Cr, 11.6%Ni, 2.3%Mo, 1.4% Mn and other minor elements (Sandvik Osprey UK) is used as the matrix material. The alumina powder used in this research is α -alumina (≤ 400 nm) with 99.9% metal base, supplied by Alfa Aesar UK. The titania powder has an average particle size of less than 320 nm, supplied by Huntsman England, UK. Duramax B-000 in combination with B-007 and D-3005 were used both as a binder and as a dispersant in the process. Duramax B1000 and B1007 are aqueous emulsion used as a binder in the ceramic fabrications [Mohamed et al., 2008; Dou et al., 2003; Zhigang et al., 2007]. D-3005 is an ammonium salt of acrylic homopolymer which was used for dispersing various types of powders. Different composite compositions were prepared containing 2.5, 5, 7.5 and 10% alumina by weight. In order to prepare composite slurries, a new approach is used to homogenize the ceramic nano particles into stainless steel micro powders as follows: (i) dispersing both stainless steel and ceramic powders in two separated specimen tubes by mixing dispersant, distilled water and powder together, (ii) mixing the two dispersed slurries together, adding the binder and mixing well, (iii) filling the soft mould and obtaining the green parts. Figures 2 (a) and (b) show the composite green micro components based on alumina and titania nano particles, respectively, which retain the same quality of the SU-8 master moulds.

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Fig. 2. SEM images of the composite green micro components based on: (a) 10% alumina and (b) 10% titania.

3. De-binding and sintering

The composite green parts were de-bound in nitrogen atmosphere and then sintered in vacuum and forming gas of 90% nitrogen and 10% hydrogen in separate experiments at 1350 °C.

4. Results and discussion

After sintering, the composite micro components sintered in forming gas and vacuum were inspected under SEM and shown in Figure 3 (a) and (b), respectively. It is found that all micro features are retained after sintering. The geometric quality of the sintered composite micro components based on titania is the same as that based on alumina. Generally, ceramic inclusions are distributed homogenously throughout the stainless steel matrix after sintering. Analysis were carried out on the effects of composite compositions on both green and sintered densities (based on stainless steel, 8g/ml), linear shrinkage (based on micro gear diameter and SU-8 is used as reference) and micro hardness (Vickers) of sintered composite micro components and the analytical plots are provided in Figures 4 (a), (b), (c) and (d). As expected, the green and sintered densities decrease with the increase of ceramic content. Because the density of the alumina is lower than that of the stainless steel, increasing its content decreases the overall composite density. The linear shrinkage curves follow the same trend as the sintering density curve, in which the greater the ceramic contents are, the lower the linear shrinkage and vice versa. Moreover, the more the ceramic content increases, the higher the hardness of different ceramics in different sintering atmospheres. It is also clear that, for given composite composition and sintering atmosphere, the hardness of the micro composites based on alumina powder is greater than

that based on titania one. This happens due to the greater hardness of pure alumina than that of pure titania. Furthermore, for a given composite composition and ceramic type, the hardness of the composites sintered in nitrogen/hydrogen mixture atmosphere is significantly greater than those sintered in vacuum. The hardness based on nitrogen/hydrogen mixture atmosphere may increase due to one of the following reasons: (i) because nitrogen/hydrogen mixture contains 90% of nitrogen, it may form nitride precipitating into the grain boundaries and harden the stainless steel micro components [Monnapas et al., 2008]; and (ii) it may be dissolved into the grains and promotes solid solution hardening [Abenojar et al., 2003].



Fig. 3. SEM images of stainless steel ceramic composite micro components: (a) based on10% alumina and sintered in nitrogen/hydrogen mixture, and (b) based on10% titania and sintered in vacuum.

5. Conclusions

This chapter proposes a novel approach to fabricate stainless steel composite micro components based on experiments. In the research, composite micro components were successfully fabricated from stainless steel alumina and stainless steel titania with different compositions. The fabrication process was investigated in detail and characterizations of composite micro components were studied in terms of composite preparation process, green and sintering density, linear shrinkage, and micro hardness. The following conclusions can be drawn from the research:

- 1. Increase the ceramic content decreases both density and linear shrinkage, but it increases the hardness.
- 2. For a given composite composition, using nitrogen/hydrogen mixture and vacuum atmospheres produces nearly the same density and linear shrinkage, while the hardness increases significantly by using nitrogen/hydrogen mixture atmosphere.
- 3. For a given composite composition, the sintered density and linear shrinkage of stainless steel-titania composite is greater than stainless steel-alumina, while the hardness of the later is greater than the former one.



Fig. 4. Effect of ceramic inclusion on: (a) composite green density, (b) composite sintered density, (c) composite linear shrinkage and (d) composite Vickers hardness.

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Advances in Nanocomposites - Synthesis, Characterization and Industrial Applications was conceived as a comprehensive reference volume on various aspects of functional nanocomposites for engineering technologies. The term functional nanocomposites signifies a wide area of polymer/material science and engineering, involving the design, synthesis and study of nanocomposites of increasing structural sophistication and complexity useful for a wide range of chemical, physicochemical and biological/biomedical processes. "Emerging technologies" are also broadly understood to include new technological developments, beginning at the forefront of conventional industrial practices and extending into anticipated and speculative industries of the future. The scope of the present book on nanocomposites and applications extends far beyond emerging technologies. This book presents 40 chapters organized in four parts systematically providing a wealth of new ideas in design, synthesis and study of sophisticated nanocomposite structures.

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