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Citation: KIM, J-S., JIANG, K. and CHANG, I.T., 2006. A net shape process for metallic microcomponent fabrication using Al and Cu micro/nano powders. Journal of Micromechanics and Microengineering, 16 (1)pp. 48-52

Additional Information:

- This article was published in the serial, Journal of Micromechanics and Microengineering [© IOP Publishing Ltd]. The definitive version is available at: http://iopscience.iop.org/0960-1317/16/1/007

Metadata Record: https://dspace.lboro.ac.uk/2134/11226

Version: Accepted for publication

Publisher: © Institute of Physics (IOP)

Please cite the published version.
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A Net Shape Process for Metallic Microcomponents Fabrication Using Al and Cu Micro/Nano Powders

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Abstract

This paper presents a new fabrication process for producing micro Al-based alloy components. The process is based on micro powder injection moulding technology, while the micromoulds are produced using MEMS technology. The process involves (1) fabrication of PDMS micromoulds from SU-8 masters, which are produced using UV photolithography process; (2) making metallic paste by mixing 80 wt% of ultrafine Al (2.5micron in average) powder, 5 wt% Cu nanopowder (less than 60nm), and 15 wt% adhesive binder in about 30ml of acetone; (3) mould filling with metallic paste and demoulding; and (4) sintering of moulded component in Ar atmosphere. The proposed process has been used to sinter Al-Cu powder microcomponents successfully without the help of either high pressure compression or mixing Mg with Al powder. This research proposes a new approach to fabricate 3D micro metallic components to meet the needs in applications where metal, rather than silicon, microcomponents are required.

Keywords: Metallic microcomponent, µMIM, µPIM, SU-8 moulds, Al powder, and Cu nano powder

1. Introduction

Metal injection moulding (MIM) is a manufacturing process for net shape forming of high integrity metal parts [1]. It combines the advantages of producing geometrical complex components inherited from plastic injection moulding and good mechanical properties of high performance metallic alloys. Due to the nature of net shaping, where machining is normally not required, the cost of the production is low, especially in large volume production. Influenced by the rapid progress of microsystems technology, on the other hand, the demands have appeared for precision micro metallic components. Metallic microcomponents generally have better conductivity and mechanical property and perform better in many applications than Si which the MEMS technology is based on. Examples can be found from micro RF components, micro imprinting maters, fuel injection nozzles and micro gears for durable watches. In response to these demands micro metal injection moulding (µMIM) has emerged. µMIM provides an alternative way to semiconductor fabrication process based MEMS technology[2]. Its distinguishing feature from MEMS technology is the capability of producing metallic micro components[3]. When compared micro EDM and micro laser fabrication, µMIM has the advantage of volume production at low costs. The potential of µMIM and related micro powder injection moulding (µPIM) have been recognized and some research work has been carried out. Piotter et al developed the idea of manufacturing micro components in mid 90s and some preliminary results were published in 1997 on manufacturing of ceramic or metal microstructures[4]. Their further research work was published in the following year, where carbonyl iron, aluminum oxide and zirconium oxide powders were used in making microstructures of 260 µm in lateral dimension and 80 µm in the smallest feature [5]. The other interesting work was presented by Shimizu et al [6], in which a micro mould was made using laser ablation and stainless steel powder mixture was injected in the mould. The component
produced from this process has an aspect ratio of 5:1. The research activities in µMIM and µPIM have just started and their full potential has to be explored yet. Although limited powder materials have been used in µMIM and µPIM, most metallic powders which have been successfully sintered in powder metallurgy experiments have the potential to be used in µMIM. Al powder is one of them.

Components made from Al powder often show exceptional mechanical and antifatigue properties, high thermal and electrical conductivity and good response to a variety of finishing processes, [7] and [8]. However, producing Al alloy microcomponents proves to be challenging. The problems come in two folds. One is that when Al powder meets oxygen, rapid oxidization will take place, resulting in high temperature burning and combustion. For this reason, one of the applications of ultra fine Al powder is for solid rocket boosters [9]. The other problem is that the oxide layer formed with oxygen in low density separates particles and makes sintering difficult. One way widely adopted to break the oxide layer sintering Al powder components is to apply high pressure of 100-400 MPa on the powder compacts before sintering at a temperature between 520-600 °C [10] and [11]. A modified process from compression and sintering method is spark plasma sintering process, [12] and [13], where the compression pressure has been reduced to 23.5 MPa and the temperature is kept at 600°C (873K). Another common approach to assist breaking the oxide layers and to sinter Al powder is to add trace Mg with Al powder [14]. Magnesium, especially at low concentrations, has a disproportionate effect on sintering because it disrupts the passivating Al2O3 layer through the formation of a spinel phase. Magnesium penetrates the sintering compact by solid-state diffusion, and the oxide is reduced at the metal-oxide interface. This facilitates solid-state sintering as well as wetting of the underlying metal by sintering liquids, when these are present. The optimum magnesium concentration is approximately 0.1 to 1.0 wt% [15]. In the compressed Al powder sintering process, pressure is the key for success, while in the Mg additive process, pressure is in presence[14] and Mg is used in addition to improve the property of the compacts. High pressure compression is feasible for conventional powder metallurgy fabrication, as the moulds and dies are made of rigid metals. However, in microsystem technology which is based on semiconductor fabrication, Si and photoresists are the main materials for the moulds and hard micromoulds are hardly available.

The research work presented in this paper is set up to combine µMIM, µPIM, powder sintering technology and MEMS technology together in order to develop a new process of producing 3D metallic microcomponents. In the new approach, high quality soft micromoulds are fabricated using MEMS technology, and a pressure free sintering process is developed for micro Al and nano Cu powder mixture. The process can be used with the soft moulds to produce 3D metallic microcomponents. The new micro moulding process involves the following steps: (1) producing master moulds in SU-8 or Si; (2) producing polydimethylsiloxane (PDMS) soft moulds from the masters; (3) mixing micro Al and nano Cu powders with adhesive binder; (4) filling the moulds with the powder mixture; (5) demoulding to produce the green patterns; and (5) sintering the greens to produce the micro components. Al-Cu alloyed microcomponents have been successfully fabricated in the experiments and images are provided. The new microfabrication process is pressure free, which makes soft moulds usable for the process. In addition, it uses Cu instead of Mg. Fine Mg powder is difficult to keep and since difficult to find. Also, in the Mg assisted processes which can be traced so far, pressure is applied. The wide availability of Cu powder and the pressure free feature imply that the new process is both easier to be implemented and cheaper to be used in industry. It provides a new option to produce metallic microcomponents for wide applications.
2. Mould fabrication

The fabrication process to produce net shape microcomponents using Al alloy powder starts from making master moulds. The master mould is relatively rigid, of high precision and usually fabricated using MEMS technology, such as deep reactive ion etching (DRIE) and X-ray exposure on SU-8 photoresist. The moulds used in [3] are an example of the process of X-ray lithography on SU-8, also referred as LIGA process. In an X-ray lithography, very high exposure energy is applied to ensure deep light penetration and result in a vertical sidewall. This feature enables the SU-8 microcomponents to be built with aspect ratios as high as 75:1 [16] and 100:1[17]. However, the X-ray is generated from a synchrotron and the high costs for using such device make X-ray lithography on SU-8 difficult to commercialize. SU-8 UV lithography has gained much progress in recent years. The ultra-thick SU-8 process (UTSP) developed by Jin et al has been used to produce 40:1 aspect ratio features in 1000 µm thick SU-8 and is adopted in the experiments for producing the master moulds. In producing 1 mm thick microstructure, the process starts by casting SU-8 50 (Microchem, USA) on to a well levelled wafer and baking it at 65°C for 2h, and then at 95°C for 15h. Then the baked SU-8 is exposed under UV light for 2.5 J/Cm². The wafer is baked again at 65°C for 15 minutes and then at 90°C for 25 minutes before fully developed in EC solvent supplied by Chestech, UK. More details can be found in references [18] and [19]. Figure 1 shows a picture of an SU-8 microgear fabricated using the UV lithography following the UTSP process. The gear is 1 mm in height and 2.5 mm in diameter, with two through holes in the middle. The SU-8 master moulds have very smooth surfaces that can be replicated to the negative moulds[20].

The microgear was used as the master mould and a negative soft mould was produced from it. A widely adopted soft moulding technique is using elastomer PDMS to pattern the micrometer and sub-micrometer sized structures. The PDMS slurry was prepared by mixing the PDMS precursor with curing agent (Dow Corning Corp. Sylgard 184) in a weight ratio of 10:1 and leaving it for 30 min to allow the trapped air to escape. The mixture was then poured on the SU-8 master mould template and placed in a vacuum condition until all residual bubbles had been removed. Afterwards, it was cured at 65°C for 4 hours according to the recommended schedule by Dow Corning[21]. After cooling to room temperature, the cured PDMS was peeled off from the SU-8 master mould template. Figure 2 shows the PDMS negative moulds produced from the SU-8 master moulds. Further details about the PDMS moulds can be found in [21] and [22].

3. Powder premixing and mould filling
The metallic powders used in the experiments are micron sized Al powder, supplied by Alpoco, UK, and nano sized Cu powder, supplied by Shenzhen Junye Nano Material Co., Ltd, China. Table 1 shows the particle sizes, purity, shapes and sources of the powders. Figure 3a is the Scanning Electron Microscope (SEM) image of the Al powder with a magnification of 4828 times. An SEM works using backscattered electrons to generate the image of the sample. It can provide an image resolution down to 0.5 \( \mu \)m and is suitable for showing microparticles or clusters. Fig 3b is the Transmission Electron Microscope (TEM) image of the Cu powder with a magnification of 241,000 times. A TEM works using transmitted electrons going through a sample as thin as 0.5 \( \mu \)m to generate the image of the sample structures. With a resolution down to 0.5 nm, TEM can reveal the finest details of internal structure and metal grains.

<table>
<thead>
<tr>
<th>Powder</th>
<th>Mean size</th>
<th>Purity</th>
<th>Shape</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>2.5 micrometer</td>
<td>99.9%</td>
<td>Spherical</td>
<td>Alpoco, UK</td>
</tr>
<tr>
<td>Cu</td>
<td>69.4 nanometre</td>
<td>99.9%</td>
<td>Spherical</td>
<td>Shenzhen Junye, China</td>
</tr>
</tbody>
</table>

Fig. 3. (a)SEM image of Al powder, and (b) TEM images of Cu powder.

The metallic powder premix was made of 80wt% micron-sized Al powder, 5 wt% nano-sized Cu powder and 15wt% binder. The powders and binder were mixed with about 30ml of acetone to form slurry. The mixing process was carried out for 2 hours using a magnetic stirrer (Hanna Instruments HI180H/D). When the adhesive binder is mixed with the powders in acetone, it is diluted and its adhesion is lost temporarily. When the mixture is dry, the adhesive binder regains its bonding characteristics so the moulded components can maintain their patterns. No additional debinding process was required, as the binder evaporates in-situ during the sintering process in the furnace.

Then the cavity of patterned PDMS mould was filled up with the premix. The following methods were experimented in the mould filling process: (1) pouring the powder premix on to the moulds under gravity; (2) filling the cavity by centrifugal force; (3) filling with mechanical vibration produced from an ultrasonic cleaner and (4) immersing PDMS mould into the mixture. Extensive experiments have been carried out to fill the moulds using the four methods and the results are shown in Fig. 4. It is found that methods (1) and (3) tends to leave some holes unfilled in the moulds, referring to Figs 4a and 4c. It may be caused both by trapping some air bubbles in the PDMS and by uneven force applied to the moulds. The presence of such unfilled volume leads to great porosity level and causes poor shape retention of the moulded component. Figure 4b shows the green components moulded using method (2). The density of the components in this case is better than those in Fig 4a and 4c, but the shapes of the components tend to have a
distortion. Figure 4d shows the microgears formed using immersing method, i.e. method (4). The results are obviously better than the other three moulding methods. In immersing method, the powder is mixed with three times more acetone than in the other three methods and the mixture becomes slurry. The PDMS mould is immersed into the slurry with pattern surface upwards and the thin powder mixture fills in the mould while air bubbles escape from the mould through the thin slurry. Acetone will evaporate gradually, leaving the powder mixture dry in the mould. Then the PDMS mould was carefully removed to achieve the powder components with a good shape retention and low porosity level, as shown in Fig. 4d.

![Figure 4d](image)

Fig. 4 Patterned moulded components prepared from various mould filling methods: (a)~(d) correspond to filling methods (1)~(4) respectively.

4. Demoulding and sintering

Once the premix was dry, the green component was achieved by peeling off the soft PDMS mould. Next, the moulded component was placed inside a furnace (Carbolite 2416-tube furnace) filled with Ar gas and heated to 600°C at 5°C/min. From previous studies, a 6 hours sintering time at 600°C was sufficient to densify this moulded microcomponent. Detailed studies of the sintering behaviour of the moulded Al components can be obtained from the literature[12]. The sample was held for 6 hours at 600°C and taken out after the furnace was cooled down to room temperature.

5. Result and density analysis

Figure 5 shows a sintered Al-5wt%Cu microgear and its gear-teeth. It can be seen that a good shape retention of the microcomponent has been achieved after sintering. Figure 6 shows an SEM image of the microstructure of the component.
The density of the Al-Cu microcomponents has been investigated. The analytical approach adopted is to measure the mass and the volume of the components and the density can be obtained by mass/volume. The component was weighed both in air and in distilled water at 10°C and the density can be calculated. The theory used is based on Archimedes’ principle and the equipment used is Density Determination Kit GX-13 from A&D Co Ltd. Table 2 lists density of the microcomponents, both before and after the sintering process. It can be observed that the sintered components have a density of 96% of the theoretical calculation.

<table>
<thead>
<tr>
<th>Component</th>
<th>Density</th>
<th>Percentage of theoretic density of bulk Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Green component</td>
<td>2.033 g/cm³</td>
<td>75.3%</td>
</tr>
<tr>
<td>Sintered component</td>
<td>2.593 g/cm³</td>
<td>96%</td>
</tr>
</tbody>
</table>

6. Conclusions

A new fabrication process is presented for producing Al-Cu alloy microcomponents from micro Al powder and nano Cu powder through sintering. The technology is based on µPIM but extended with the help of MEMS technology. The process involves (1) fabrication of micro master moulds in SU-8 using UV lithographical process and PDMS negative moulds are made from the SU-8 masters; (2) preparation of metallic paste by mixing 80 wt% of ultrafine Al (2.5micron in average) powder, 5 wt% Cu nanopowder (less than 60nm), and 15 wt% adhesive binder in about 30ml acetone; (3) filling the moulds with the prepared metallic powder paste and peeling off the PDMS moulds when the powder patterns are solid; and (4) sintering moulded component in Ar atmosphere. Al-Cu microcomponents have been successfully produced following this process. The density of the components have been studied and it is found the components have 96% of the theoretical density, which seems satisfactory.

The new process has proven that ultra fine micro sized Al powder can be sintered into microcomponents when a very small portion (about 5 wt%) of Cu nano powder is properly mixed with it. Further more, this sintering process can happen without additional high pressure. The proposed process indicates a new way to sinter Al powder, which is different from the well known high pressure Al powder sintering method and Mg assisted Al sintering process.

The use of SU-8 masters plays an important role in the fabrication of the microcomponents. The SU-8 masters of up to 1 mm thick are fabricated following the UTSP process. The geometry quality of the SU-8 components is as good as the Si microcomponents fabricated using DRIE
process, but are deeper and smoother on the sidewall than Si components. With these features, when SU-8 components are used as moulds, they can be employed not only for micropowder injection moulding but also in microelectroforming, microceramic sintering and microplastic injection moulding. In addition, the use of soft PDMS moulds makes the demoulding easier in avoiding the damage of the green patterns by the moulds. The investigation has also achieved progress in identifying the best mould filling method. After repeated experiments, immersing method stands out. It basically avoids the forming of air bubbles and increases the density and mechanical strength of the components.

The proposed process shows a new way of fabricating three-dimensional Al-based alloy components. With some modifications, the method could be used for producing metallic microcomponents using other micro and nano metal powders.

7. References