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A new densification mechanism of copper powder sintered under an electrical field

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A new sintering mechanism is revealed for copper powder sintered under the influence of an electrical field and a force field during the formation of microcomponents. Analysis of the microstructure and grain boundary evolution of the sintered samples showed that the disappearance of the interface at contact areas between particles is a continuous process which involves new grain formation and grain refinement during this innovative microsintering process. The densification process is therefore different from what is known in a conventional powder sintering process.

Keywords: Electric-field-activated sintering; Micro-FAST; Copper powder; Grain joining; Grain refinement

Over the last two decades, the development towards miniaturization of products and devices in industries such as electronics, optics and communications has increased the demand for metallic parts manufactured at the microscale. During the last 10 years, various micrometal-forming processes have been studied and used to produce a variety of micrometal components. In particular, such processes have been studied as part of the EU large-scale integrated project MASMICRO, which researched and produced various manufacturing facilities for micromanufacturing [1].

The use of microcomponents as well as intensive competition on manufacturing cost led to the requirement for cost-effective production of these components without much compromise on their final quality. To address this issue, a novel microforming technology, named Micro-FAST (combining microforming and field-activated sintering technology (FAST)), was proposed recently by the present authors for the forming of microcomponents [2–5]: the process is illustrated in Figure 1. Micro-FAST is a method which can be used for the forming of microcomponents with a variety of material systems, e.g. copper gears (shown in the bottom right corner of Fig. 1), have been fabricated successfully at the microscale. Over the last few years, pioneering work has been carried out by several authors and many interesting experimental results have been achieved. First, Micro-FAST is an efficient process: the entire forming process can be accomplished within a few seconds, and the relative densities of the formed microcomponents are high. Furthermore, of the sintering parameters, the heating rate, sintering temperature and heating cycle all have a significant effect on the densification of the metal powders during the sintering process [2,5]. Moreover, with continuous high pressures being applied and the short forming time, Micro-FAST occurs without coarsening of grains during the densification process [4]. Joule heating is the main heat source during the sintering process, and thus the process has different densification mechanisms compared to those of conventional processes (including FAST or spark plasma sintering (SPS)). Lastly, the densification mechanism of metal powder is related to plastic deformation and the interfacial melting of particles. A three-stage sintering model has been established to describe the process of the densification of 316L stainless steel powders when being sintered by Micro-FAST [3]. For the densification process in a traditional powder sintering method, grain growth and neck growth are the two critical mechanisms to achieve densification. Grain growth is caused by coarsening, which is associated with either surface diffusion or evaporation/condensation [6]. Grain coarsening can also be observed in other FAST methods [7,8]. However, the mechanisms of neck formation during the sintering of metal powder without grain growth for enhanced densification in Micro-FAST are virtually unexplored.

The purpose of the present work was to study the mechanism of copper powder densification at the microscale in a Micro-FAST process, based on scanning electron microscopy (SEM), transmission electron microscopy (TEM) and high-resolution TEM (HRTEM) analysis. This kind of work has not been done by other researchers. The main objectives were to establish the geometric model of grain

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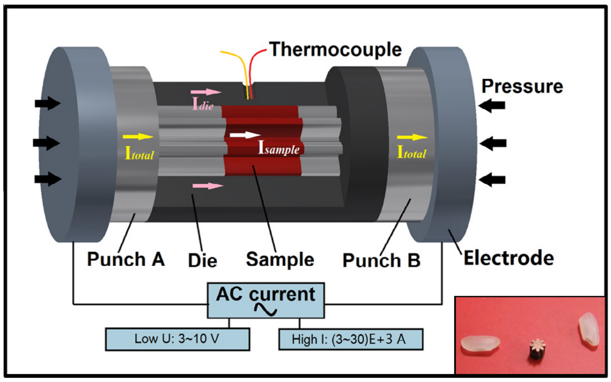

Figure 1. Illustration of the Micro-FAST sintering and forming process.

evolution between the two particle contact areas during this innovative microforming process. The new understanding developed would help to develop a new processing strategy for this kind of powder, as well as providing better quality control during the sintering of microcomponents.

Commercially pure copper powder (99.7% purity, 0.05 wt.% Pb, 0.05 wt.% As, 0.05 wt.% Fe, 0.05 wt.% Sb), with an average particle size of 20 μm, was used for the experiment. The powder was purchased from CNPC Powder (Shanghai, China). A scanning electron micrograph of the initial powder is shown in Figure 2(a). The atomized powder also contain a fraction of fine particles of size <10 μm.

A stepwise experiment was designed to study the grain evolution and the process being carried out in a vacuum ($<10^{-4}$ Pa). In the Micro-FAST process, the as-received powder consisted of agglomerates which were suitable for making up a microgear with a pitch diameter of 1.6 mm. After weighing, the loose powder was loaded into a die. The die was then placed into a Gleeble-1500D machine, and heated rapidly to a particular sintering temperature at a preset heating rate (a high electric current passes through the die set). At the same time, a preset pressure was applied to the punch. The detailed technological parameters of the experiments are given in Table 1.

After sintering, the microstructure of the samples was observed under a JSM-5900LV scanning electron microscope (JEOL, Japan), and TEM and HRTEM observations were performed using a JEOL JEM-2010F microscope operating at 200 kV.

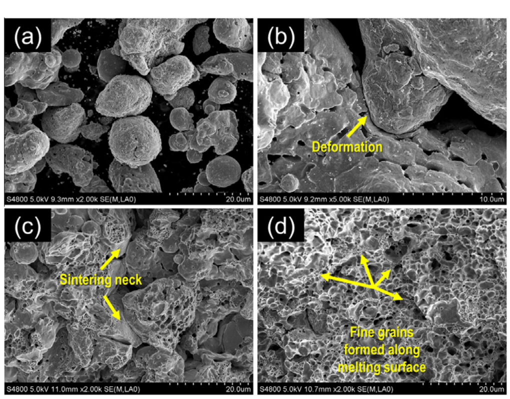

Figure 2. SEM micrographs of the fracture surface of the samples: (a) the initial copper powder; (b) 1#, sintering at 200 °C; (c) 2#, sintering at 600 °C; (d) 3#, sintering at 600 °C with five heating cycles.

The SEM micrographs of the fracture surface of the samples sintered at different levels are shown in Figure 2. Comparing the micrographs in Figure 2, it can be seen that, at different sintering levels, the contact state between particles developed from "point contact" to "lineal contact" and then "surface contact". Lu et al. [2] have reported that if the temperatures at the contact areas between particles are high, the yield stresses of the material at the contact areas and the viscosity of the compact will decrease. Therefore, the deformation rate of the material at a contact area will be greater when the temperature is high. Similarly, the distances between particles will decrease under high pressure and the contact areas will become larger. The microsized pores within the powder compact will be filled with neighboring powder particles due to bulk and plastic flow. This is largely due to the effect of electroplasticity [9]: it was found that an electric field of ~1 kV cm^{-1} could significantly reduce the plastic and fracture stresses of metals at room temperature. It was subsequently confirmed that the electric field also lowered the brittle-to-ductile transition temperature, reduced the flow stress and increased the elongation at elevated temperatures.

A further important observation is that the formed microgear had good mechanical properties. As shown in Figure 2(c) and (d), the fractography of the fracture section was typically of a dimpled nature, which indicates that the fracture belongs to ductile fracture, with enlarged dimples and more liquid phase formed [10]. The welded joints between particles observed in Figure 2(c) are clear evidence of the formation of the liquid phase. This figure also shows that, provided that a significant amount of heat is generated at the interface of the particles, the melting point of fine powder particles is likely to be achieved at those interfaces that cause the generation of a liquid phase at local areas. Once the liquid phase is formed, it will lead to the melting of the contact surface between particles [11]. At the same time, with the aid of the pressure applied to the compact, the liquid phase flows into the vicinal pores due to viscous flow and capillary force [12], which results in the disappearance of the interface between particles and fast densification of the compact (see Fig. 2(d)).

Moreover, upon investigation of the sizes of grains inside the particle, it is interesting to note that no coarsening of grains accompanied the process of the densification of the microsized compact. However, as shown in Figure 2(d), when the formed microparts have nearly obtain full relative density (99.23%), a quantity of fine grains is formed along the particles' edge, especially at the interface melting areas between particles.

The ABEQUS FE code was used for an electrical-heat analysis. A simulated current density distribution in the two assumed copper powders during the sintering is shown in Figure 3(a). It can be seen that there was an electric current concentration in the neck area of the sintering. This could be a main cause of the particles' deformation to an equilibrium size, and the melting of interfaces occurred at this area. This is also evident in the sample shown in Figure 3(b), where formation of the liquid phase and fine grains between two particles are clearly seen. Furthermore, twin boundary migration (as shown in Fig. 3(c)) is observed, and this can become a significant electroplasticity mode. This investigation strongly suggests that the twin grain growth plays a deterministic role in the structural refinement [13].

We conclude from these observations that the applied AC field (low voltage: 3–10 V; high electric current: 3–30 × 10^3 A) leads to welding of the powders/grains that are in contact within a very short time (a few seconds) in