Strathprints Institutional Repository

Huang, Kunlan and Qin, Yi and Yang, Yi (2014) Effect of particle size on densification of copper powder during electric-field activated sintering for micro-scale forming. In: Advances in Manufacturing Technology XXVIII. Southampton Solent University, Southampton. ISBN 9780992695842 ,

EFFECT OF PARTICLE SIZE ON DENSIFICATION OF COPPER POWDER DURING ELECTRIC-FIELD ACTIVATED SINTERING FOR MICRO-SCALE FORMING

Kunlan Huang
School of Manufacturing Science and
Engineering, Sichuan University
Chengdu
Sichuan 610065, PR China
kunlan.huang@strath.ac.uk

Yi Yang
School of Manufacturing Science and
Engineering, Sichuan University
Chengdu
Sichuan 610065, PR China
yangyi@scu.edu.cn

Yi Qin
Department of Design, Manufacture and
Engineering Management, Strathclyde University
James Weir Building,75 Montrose Street
Glasgow G1 1XJ, UK
qin.yi@strath.ac.uk

ABSTRACT

A novel Micro-forming technology, called electric-field activated sintering for micro-scale forming (Micro-FAST), was introduced for the forming of micro-components. The effect of particle size on densification is revealed for copper powder being sintered under the influence from electrical field and force-field during forming of micro-components. Three kinds of copper powders of different particle sizes ((i) average particle size of 0.5μm; (ii) average particle size of 30μm and (iii) the mixture powders with 20% weight of 30μm and 80% weight of 0.5μm) with no binder were used for the experiments. The results show that the density of the compact sintered with mixed copper powders is the largest due to more volume of liquid phase was formed in the particle's contacts. The result being in correspondence with the analytical results of computer simulation. The new understanding developed would help to better quality control during the sintering of micro-components.

Keywords: Electric-field activated sintering, Micro-FAST, Particle size

1 INTRODUCTION

During the last 10 years, various micro-metal-forming processes have been studied and used to produce a variety of micro-metal-components. These efforts were highlighted particularly by the EU large-scale integrated project MASMICRO, which conducted research leading to the development of various manufacturing facilities for micro-manufacturing (Qin 2010). The use of micro-components as well as intensive competition on manufacturing cost has led to the requirement for cost-effective production of these components, without much compromise on their final quality. To address this issue, a novel micro-forming technology, named electric-field activated sintering for micro-scale forming (Micro-FAST), was proposed recently by the present authors for the forming of micro-components (Lu et al. 2013; Huang et al. 2013; Lu et al. 2012; Huang et al. 2013):

Micro-FAST is a method which can be used for the forming of micro-components with a variety of material systems, the process is illustrated in Fig.1. For instance, copper gears (shown in Fig.1 bottom right corner) have been fabricated successfully at the micro-scale. Joule heating is the main heat source during the sintering process and thus it has different densification mechanisms compared to those of conventional processes (including FAST or 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 (Huang et al. 2013).

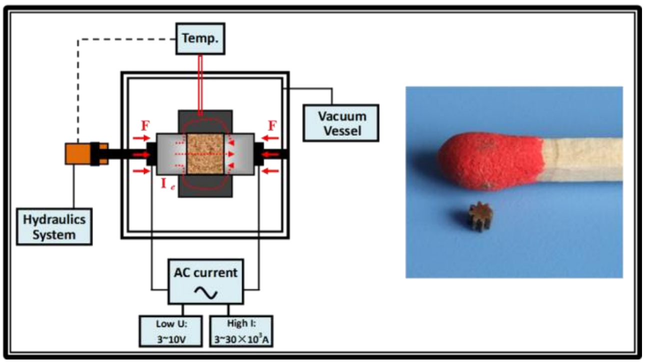

Figure 1: Illustration of the Micro-FAST sintering and forming process.

Over the last few years, pioneering work has been done by several authors and many interesting experimental results have been achieved. Firstly, Micro-FAST is an efficient process, the entire forming process can be accomplished within a few seconds and the relative densities of the formed micro-components are high. Furthermore, for the sintering parameters, the heating rate, sintering temperature and heating cycle have a significant effect on the densification of the metal powders during the sintering process (Lu et al. 2013; Du et al. 2012). Moreover, with continuous high-pressures being applied and ultra-fast forming time, Micro-FAST occurs without the coarsening of grains during the densification process (Lu et al. 2012). However, the effect of the particle size of starting raw materials on the densification in the presence of an externally applied electrical field remain mostly unexplored.

The purpose of this work was to study the effect of the particle size of copper powder densification in Micro-FAST, which presently has not been studied in previous work. The main objective was to find the microstructure differences among samples sintered by different particle sizes, which help to improve the understanding of the sintering process and hence the quality of the component. The methodology in the research combined both experimental and computer-simulation results to explain the effect of particle size.

2 EXPERIMENTAL PROCEDURES

For the experiment, commercially-pure copper powders (99.9% purity) was obtained in two particle size, (i) with an average particle size of 0.5μm; (ii) with an average particle size of 30μm. (iii) the mixed powders with 20% weight of 30μm and 80% weight of 0.5μm, were used for the experiments. Fig.2 (a), (b) and (c) show scanning electron micrographs of the three kinds of copper powders respectively. The initial powders were loosely agglomerated. Moreover, the atomized powder exhibited a near-perfect spherical shape and a homogeneous size distribution.

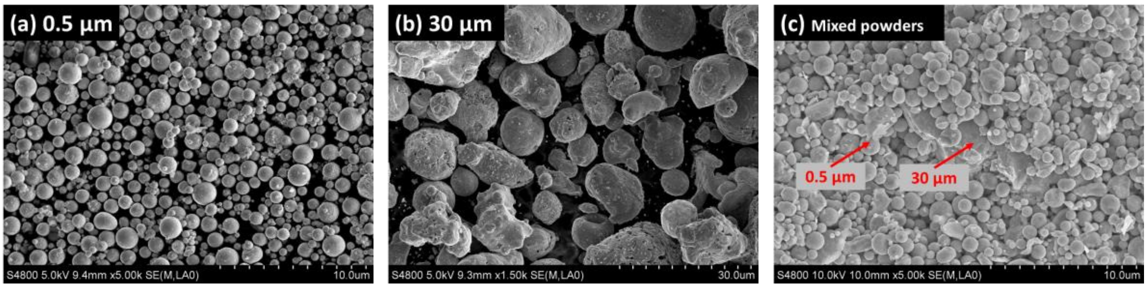

Figure 2: SEM micrograph of copper powder: (a) 0.5μm; (b) 30μm; (c) the mixed powders with 20% weight of 30μm and 80% weight of 0.5μm.

Fig.3 shows the setup of the tool-set used in the experiments with a Gleeble-1500D thermal simulation machine from Dynamic System Inc., USA. The electric field produced by the machine has low voltage and high current (3~10 V and 3,000~30,000 A). The as-received of copper powders consisted of agglomerates, which were sufficient for the fabrication of a micro-gear with a pitch diameter of 1.6 mm. After weighing, the powder was loaded into a die, a model of which is shown in Fig.3. Next, the die filled with copper powder was placed into the Gleeble-1500D machine, and then heated rapidly to a particular sintering temperature at a preset heating rate in a vacuum ($<10^{-4}$ Pa) (a high electric current passes through the die set), and at the same time, a preset pressure was applied to the punch. The processing parameters of the experiments are given in Table1.

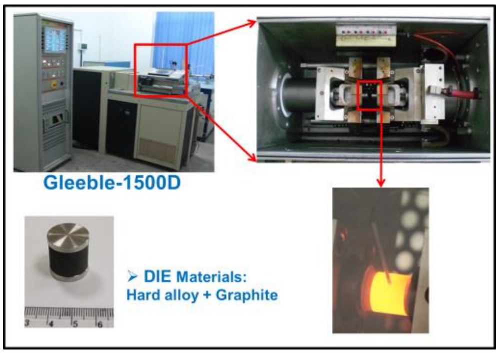

Figure 3: Tool-set used and experiment set-up with the Gleeble-1500D

Table1. The processing parameters and the relative density corresponding to the samples formed.

Specimen designation	Particle size (µm)	Heating rate (°C /s)	Pressure on the punch (MPa)	Sintering temperature (°C)	Relative density (%)
1#	30	50	75	700	96.63
2#	0.5	50	75	700	99.05
3#	Mixed powders	50	75	700	99.39

After sintering, the relative density of the sintered compacts was measured using the electronic analytical balance TP-214 and the microstructure of the samples were observed under a scanning electron microscope JSM-5900LV, JEOL (Japan).

3 RESULTS AND DISCUSSION

The morphology of a formed sample with particle size of 30µm can be seen in Fig.1, which latter displays a well-shaped profile. This micro-gear with 8 teeth was fabricated to a module of 0.2 and a pitch diameter of 1.6 mm. The calculated relative densities of the sintered samples are given in Table 1. As shown in this table, the particle size is an important parameter to influence the relative density of the samples. It can be noted that the 3# micro-formed sample has a largest relative density of 99.39%.

3.1 Microstructure of the Formed Samples

Comparing the SEM micrographs of the fracture surface of the samples sintered with different particle size are shown in Fig.4, the following was found:

(i) Copper powders from a small to large particle size can be well sintered to micro-sized compacts under the coupled actions from multi-fields (electric field, temperature field and pressure field) within a very short sintering time (e.g. 84 seconds), while with a conventional sintering it could take a couple of hours (Beri et al. 2010). Furthermore, it was found that no coarsening of grains accompanied the process of the densification of the micro-sized compact. Moreover, the fractograph of the fracture section was of a typically dimpled nature, which indicates that the fracture belongs to ductile fracture, and with enlarged dimples and more liquid phase formed. However, there were still some residual holes (indicated by the arrow in the Fig. 4) present in the samples.

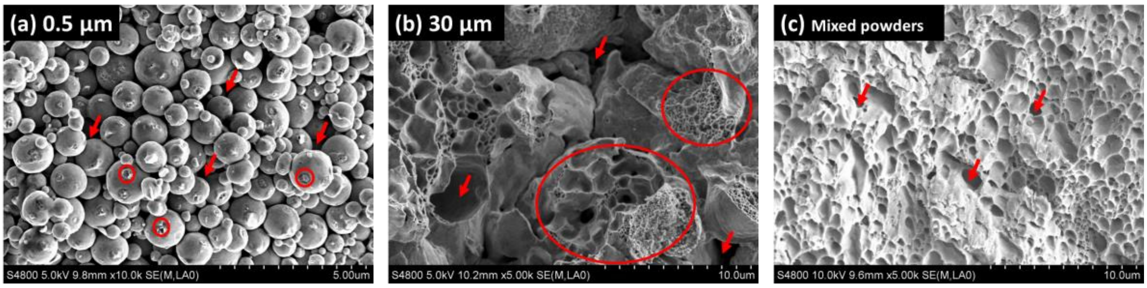

Figure 4: SEM micrographs of the fracture surface of the samples sintering with different particle size: (a) 1#, 0.5µm; (b) 2#, 30µm; (c) 3#, the mixture powders with 20% weight of 30µm and 80% weight of 0.5µm.

(ii) Comparing the Figs. 4(a) and (b), it can be seen that the relative density of the 1# sample was larger than that of 2# sample while the sintering neck area (indicated by the circle in the Fig. 4) was smaller. Due to the small size effect, the 0.5µm particles have a reduced melting temperature compared with 30µm particles. This is because the arrangement of atoms on the free surface and interfaces (such as grain boundaries, phase boundaries, etc.) is very different from the inner complete lattice of powder particles. However, large particles are more prone to fracture at compaction pressure compared to fine particles. The mechanical plastic deformation of particles made a great contribution to the densification of powders during the Micro-FAST sintering process.

(iii) The density of the compact sintered with the mixed copper powders is the largest. Since the interfacial areas between particles are larger, the extent of the densification of the compact can be enhanced greatly. Also, the mixture particles specific surface energy promote the surface melting process due to large surface contact area in the mixture powder system. Due to the high current density and high instant contact temperature, the melting point of small powders may be reached rapidly in the heating period of the sintering procedure. Therefore, small particles may evaporate and melt at relatively low nominal temperature.

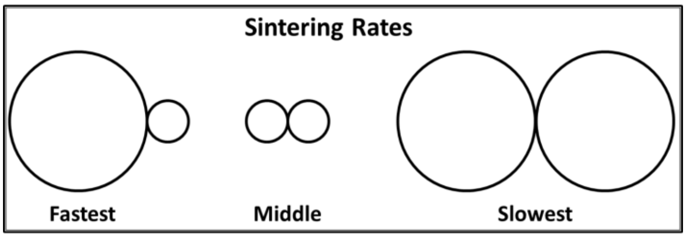

Figure 5: Relative sintering rates of pair arrangements (Darcovich et al. 2003).

K. Darcovich et al. (2003) formulated the various sintering rates which arise for the three pairing available with two different sized particles. The results of Darcovich's study was shown in Fig.5, which is clear evidence of the mixed copper powders can achieve the largest relative density under the same sintering parameters comparing to the other particle size.

3.2 FEM Simulation Sintering Process

To establish the current density and temperature transmission model during the sintering process in Micro-FAST, COMSOL Multiphysics software was used due to its capability to solve coupled multiphysics phenomena simultaneously (Wang et al. 2011).

The simulation result of the electric and temperature field distribution of the model with two copper spherical powders during the sintering process is shown in Fig.6. The current density and sintering temperature were different between sintering neck and non-contact area, the sintering temperature of which were about 1045.10 °C and 745.07°C, respectively. Therefore, welding joins can obviously be seen between adjacent particles in samples. The liquid phase occurs at an interface due to the contact resistance. The presence of a liquid phase resulting in fast densification of the compacts at a low sintering temperature.

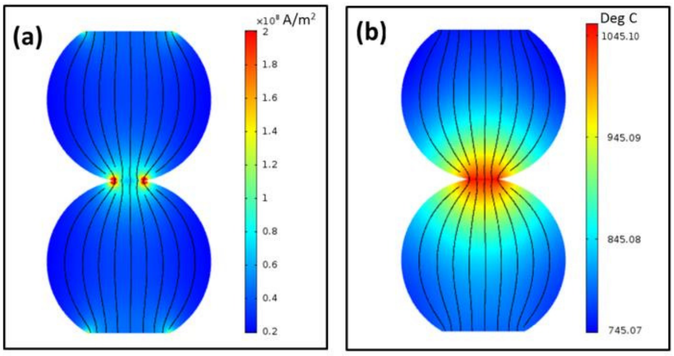

Figure 6: Electrical and temperature field distribution of the model filled with copper powders: (a) electrical field distribution; (b) temperature field distribution.

3.3 Effect of Particle Size on the Local Overheating

Joule heating is the main heat source during the heating process and occurs without generating sparks, compared to SPS (Zhou et al. 2003). When an AC current passes through a compact, Joule heat will be generated at an interface due to the contact resistance. Fig.7 illustrates the distribution of the current in the compacts. According to the Joule-Lenz's law, the heat generated by the current passing through the compact is:

$$Q = I^2 Rt \tag{1}$$

Q is the generated heat when current passes through the compact; R is the resistance of particles and t is the time of duration of electrical flow. In the process of sintering, the change of Joule heat is affected by the interfacial contact resistance and current flow through the particles. As observed, the greater the temperature and electric current density, the greater the Joule heat.

As the surface area of the particles increases, the resistance of the particles' interface becomes greater, namely, the contact surface among particles can gain a local high-temperature in the temperature-rise period. A certain amount of liquid phase is formed when the local temperature reaches the Cu liquidus temperature (1083 °C), which leads to the melting of the contact surface between particles (Olmos et al. 2009).

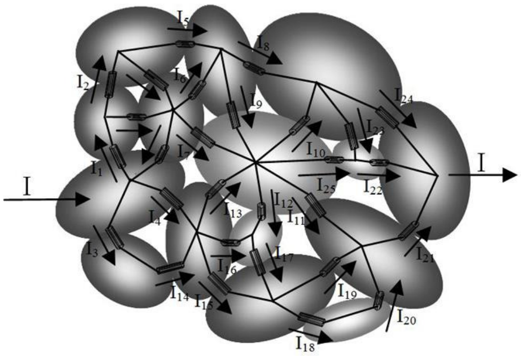

Figure 7: Illustration of the current distribution in a powdered compact

4 CONLUSIONS

From the work completed in this study the following conclusions can be drawn:

Three kinds of copper powders of different particle sizes ((i) average particle size of 0.5μm; (ii) average particle size of 30μm and (iii) the mixture powders with 20% weight of 30μm and 80% weight of 0.5μm) with no binder were used for the experiments. The results show that the density of the sample made from mixed copper powders can reach 99.39% when they were sintered at a relatively low sintering temperature (700 °C), higher heating rate (50 °C/s) and pressure 75MPa. Upon the experiment results and numerical implementation of a model designed to demonstrate the effects of local particle size distribution effects in the copper structure, it can be concluded that compared to an uniform size powder system, a structure with a broad range of powder sizes more easily achieve a good densification.

ACKNOWLEDGMENTS

This work was primarily funded by the National Nature Science Foundation of China (no.51275322) and the UK Royal Society / China NSFC International Exchanges Programme (no.51311130134) on the collaborative development of a new micro-manufacturing process. We also acknowledge Dr. J. Chen (the State Key Laboratory of Coal Mine Disaster Dynamics and Control, Chongqing University, Chongqing 400030, China) for allowing our use of the COMSOL code.

REFERENCES

Beri, N., S. Maheshwari, C. Sharma, and A. Kumar. 2010. Technological advancement in electrical discharge machining with powder metallurgy processed electrodes: A review. Materials And Manufacturing Processes 25 (10):1186-1197.

Du, A., Y. Yang, Y. Qin, and G. Yang. 2012. Effects of Heating Rate and Sintering Temperature on 316 L Stainless Steel Powders Sintered Under Multiphysical Field Coupling. Materials And Manufacturing Processes 28 (1):66-71.

Demirskyi, D., H. Borodianska, S. Grasso, Y. Sakka, and O. Vasylkiv. 2011. Microstructure evolution during field-assisted sintering of zirconia spheres. Scripta Materialia 65 (8):683-686.

Darcovich, K., L. Béra, and K. Shinagawa. 2003. Particle size distribution effects in an FEM model of sintering porous ceramics. Materials Science and Engineering: A 341 (1-2):247-255.

Huang, K., Y. Yang, Y. Qin, and G. Yang. 2013. Densification behavior of copper powder during the coupled multi-physics fields-activated microforming. The International Journal of Advanced Manufacturing Technology 69 (9-12):2651-2657.

Huang, K., Y. Yang, Y. Qin, and G. Yang. 2013. 316 L Stainless Steel Powder Densification during the Coupled Multi-Fields Activated Micro-Forming. Materials And Manufacturing Processes 28 (2):183-188.

Lu D., Y. Yang, Y. Qin. 2012. Effect of particle size and sintering temperature on densification during coupled multifield-activated microforming. Journal Of Materials Research 27 (20):2579-2586.

Lu D., Y. Yang, Y. Qin, and G. Yang. 2013. Forming Microgears by Micro-FAST Technology. Journal of Microelectromechanical Systems, 22 (3):708-715.

Olmos, L., C. L. Martin, and D. Bouvard. 2009. Sintering of mixtures of powders: Experiments and modelling. Powder Technology 190 (1-2):134-140.

Qin Y. 2010. Micro-manufacturing engineering and technology, book, Elsevier, Oxford.

Wang, X., H. Yue, G. Liu, and Z. Zhao. 2011. The Application of COMSOL Multiphysics in Direct Current Method Forward Modeling. Procedia Earth and Planetary Science: 266-272.

Zhou, Y., K. Hirao, Y. Yamauchi, and S. Kanzaki. 2003. Effects of heating rate and particle size on pulse electric current sintering of alumina. Scripta Materialia 48 (12):1631-1636.