Hijji, Hasan and Qin, Yi and Huang, Kunlan and Bin Zulkipli, Muhammad and Yang, Song and Zhao, Jie (2016) Forming alumina (Al2O3) by micro-FAST. In: Advances in Manufacturing Technology XXX. Advances in Transdisciplinary Engineering, 3 . IOS Press, Amsterdam, pp. 61-66. ISBN 978161499668 , http://dx.doi.org/10.3233/978-1-61499-668-2-61

Forming Alumina (Al_2O_3) by Micro-FAST

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Abstract. The Alumina (Al_2O_3), also known as Aluminium oxide, has a good thermal conductivity, but it is an electrical insulator. The Alumina is being used widely in the industry. Several research the sintering of Alumina using the conventional hot pressing process or spark plasma sintering (SPS). However, these methods are and have their own limits and disadvantages, such as long process chains and low efficiency with the processes and, rarely developed for the forming of miniature and micro-scale components. In this study conducted in the report. A new process has been used adapted from the electric-current activated sintering techniques (FAST) and it is been combined with micro-forming technology and called the (Micro-FAST). The Alumina powders were loaded directly into the die, followed by electric-sintering under certain pressure. In this paper Ø4.00mm × 4.00 mm and Ø2.00mm × 2.00 mm cylinder solid samples were produced. This experiment was conducted by use of a Gleeble 3800 thermal- mechanical simulator. Several properties of the solid samples, such as relative density, ESM and EDS, were examined, and these showed good results have been obtained.

Keywords. Micro-FAST, Micro-forming, Micro-manufacturing, Sintering, Al_2O_3.

1. Introduction

Alumina (Aluminium Oxide), Al_2O_3 is a major engineering material. It offers a good mechanical properties and electrical properties which leads to a wide range of industrial applications [1]. Recently, an increased the demands for micro- or miniature-components have been taken into account where there is rapid growth in applications in telecommunications and automotive engineering industry. Wide applications are also seen in bio-medical industry, information technology and home-use electronics products. Therefore, the non-traditional manufacturing technologies have been developed to improved productivity and economic effectiveness [2]. One of these technologies is Electric Current Assisted Sintering (ECAS). The ECAS is a general term for a class of consolidation methods which combining external electric field/currents with mechanical pressure for powder sintering. In 1990, significant interests in many researches and as well industrial applications have been identified from many other countries worldwide, especially from Japan, China and USA. Many papers have been published on this field; whilst on the other hand, efforts have been made on tools and machines development for industrial-scale production.

The current divided mainly into two categories: Resistance Sintering (RS) and Electric Discharge Sintering (EDS). The first category is a low-voltage and a high current with a characteristic waveform, such as (direct current (DC), alternate current

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(AC), rectified current (RC), pulsed, etc.). The other category depends on the sudden electrical energy that discharged from a capacitor bank through a column of the powder of the workpiece contained within an electrically non-conducting tube [3].

The SPS is one of the most recognised and widely applied technology, and having been used for forming many types of materials, it has limits and drawbacks in respect of its lesser heating rate and that the pressure is influential on the final result during sintering. According to Lange [4], the densification process using the conventional powder-sintering method involves a coarsening or neck growth, which is a critical mechanism needed in order to achieve densification. This is caused by surface diffusion or evaporation/condensation and that is the reason why conventional powder sintering is taking long time to be completed. In this research, the focus is on the application of FAST (Field-activated Sintering Technology) to the forming of micro-components where FAST shows particular qualities since small volumes of materials are to be heated up, such as ultra-fast heating and cooling rate (and hence, maintaining nano-structures of powder is possible), large plastic deformation of particles to rise density of the parts formed. Using the combination of forming and FAST process for sintering powders, the densification can be achieved more quickly, by deformation and breakage of the powder particles. The main differences between this process with SPS and others are: AC current applied for high heating efficiency; larger heating rate-, holding time- and pressure-dependent densification; and moreover, simplified process setup and control. Encouraging findings have been made using Micro-FAST with metallic materials [5, 6]. However, the feasibility of forming ceramic materials still needs to be investigated further.

Some previous work examined the feasibility of sintering the Alumina powder with several particle sizes and under various processing parameter, such as (sintering temperature, holding time, pressure). Many of the formed Alumina from the previous work reached 99% of the relative density under sintering temperature between 1100 °C and 1550°C and the soaking time was between 3 and 30 min which is long time and for serve the purpose of this project [7].

The material selected for making the die set is usually graphite due to excellent conductivity and it works at high temperatures up to 2500 °C. The low mechanical strength of the graphite material at high temperature is a major issue, a higher forming pressure cannot be applied during the electrical field activated sintering process (FAST) [8, 9].

2. Experiment

Al_2O_3 powder with an average particle size of 0.18 μm and purity of 99.9% was used for experiments. The theoretical bulk density for the used powder is 3.95 g/cm^3. The received powders was sufficient and ready to make up a sample with the size of Φ 4.0mm×4.0mm and Φ 2.0mm×2.0mm (solid cylinders). The weighting of the powder was performed using a precision electric balance according to the calculated value (see Eq. 1) and then it filled into the die.

$$m = \rho \; x \; V \tag{1}$$

Figure 1 shows a graphite cylindrical dies and punches were designed and manufactured for the experiments to form 4.0mm×4.0mm and Φ 2.0mm×2.0mm and examine the formability of the material and to improve the operating parameters. A small hole has been set in the middle section of the die, in order to measure the temperature using the thermocouple.

Figure 1 Graphite Punch and Die set

Unlike the powder metallurgy, in which the powder is usually mixed with binder or additive, but in the Micro-FAST process pure powder is applied directly for powder sintering. The die model is shown in Fig 1 filled with Al_2O_3 powders was then horizontally between two electrodes on the Gleeble 3800 (see Figure 2).

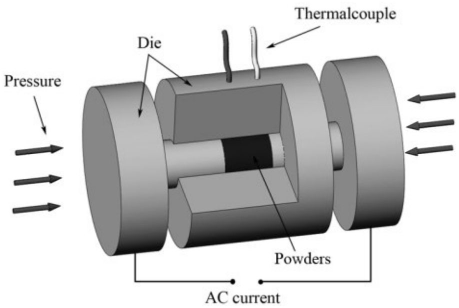

Figure 2 3 Illustration of the basic set up for the Micro-FAST experiment

Figure 3 shows The Gleeble-3800 thermal simulation machine that has been used for the experiment. The machine controls the heating process with a computer-controlled system which is able to pre-set a value of the heating rate, and the accuracy of the temperature control is within ±3 °C. The electric field that has been produced by the machine has low voltage (3~10 Volts) and high current (3000~30000A). The die-set was heated rapidly to a certain temperature at a specific heating rate in a vacuum (<10-4Pa). The high current passes through the die-set and at the same time, a certain pressure was applied onto the punches. As shown in Figure 2, the thermocouple was used for measuring the real-time temperature and giving a feedback to the computer system. The temperature and pressure were maintained for a given period of time, in order to consolidate the powder and gain a high density solid part.

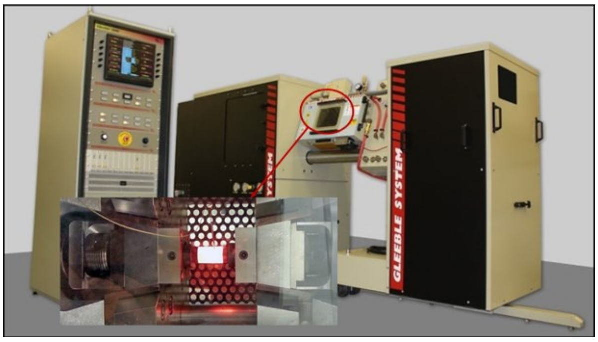

Figure 4 Gleeble 3800 and Position of the die-set with powder inside the machine

At the beginning of the sintering, there was a gap between punches and the die. The gap decreased regularly during the heating time as the temperature increased. During that process it can be clearly noted that the gap gone during the temperature holding period which shows an ideal flow of the electrical current.

The experiments were conducted with variation of different key parameters, such as pressure, heating rate, maximum temperature and holding time. The Al_2O_3 material has a very high melting-point and low electric conductivity. Therefore, the sintered temperature that has been set for the experiment was between 1200ºC and 1300 ºC with a heating rate of 50ºC/s.

3. Results and discussion

The formed samples were examined carefully using the following methods: the sample geometry morphological measurement, calculating the relative density by the Archimedean approach, and microstructural observation under SEM. All experiment of Al_2O_3powders have been successfully formed into solid cylindrical components. The results show that the highest relative density of 92.68% has been and It can be predicted that the Al_2O_3 powders sintering process can be improved by optimising the process parameters, such as by increasing the sintering temperature and holding time. Table 1 shows the tested materials and its processing parameters and relative density.

Table 1 Details of the process parameters for the Al_2O_3

Specimen designation	Sintering temperature (°C)	Heating Rate (ºC/s)	Sample size (mm)	Sintering cycle time (s)	Pressure (Mpa)	Holding Time (s)	Sample relative density
#1	1200	50	4x4	417	75	240	86.50 %
#2	1200	50	4x4	297	75	120	88.50 %
#3	1200	50	2x2	537	75	360	86.08 %
#4	1300	50	2x2	429	125	240	87.09 %
#5	1300	50	2x2	429	75	240	92.68 %

The results also show a presence of carbon at the centre and edge of the samples. This is due to a drawback when using graphite die and punches where the carbon at the punch and die wall can penetrate into the sample during the sintering process. As a result, it is difficult to achieve a formed sample that is 100% free of contamination by other elements such as carbon when graphite dies and punches are used.

Figure 4 shows The morphology of a formed sample #5, and it is very good and strong sample and it not easy to be break. The dies were designed to have a samples with dimension Φ 4.0mm×4.0mm and Φ 2.0mm×2.0mm, and the samples formed were found to have similar dimensions. Moreover, the microstructure of the formed sample #5 in Figure 4 shows a good surface, but there were some small pores. And no obvious large pores were observed.

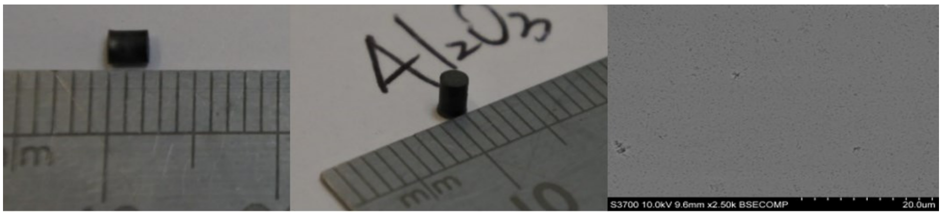

Figure 5 . A formed sample (solid cylinder) with a size of Φ 2.0mm×2.0mm and its SEM micrograph

4. Conclusion

In this paper, Micro-FAST showed the capability of the rapid forming of micro-components with Al_2O_3 material. The process can be optimised by improving sintering time and sintering parameters. Al_2O_3 material can be sintered successfully at low sintering temperature and sintering time. The optimum parameters for the Al_2O_3 sample with a 92.68% relative density were: a pressure of 75 MPa, a heating temperature of 1300 °C, a heating rate of 50 °C/s and a holding time of 240 s.

This efficient process has the potential to save time compared to the conventional process. Future work will be focussed on the investigation of influential sintering parameters in order to optimise the process and to increase its repeatability, to ensure greater quality of the parts to be produced

Acknowledgements

The authors would like to acknowledge the funding support from European Commission through FP7 FOF Micro-FAST Project (GA No. 608720)). The authors would specially thank Prof. Jianguo Lin and his colleagues in Imperial College London for providing the Gleeble machine for conducting the experiment and for their kind support through the experiment. Thank Micro-FAST project partners for supplying the powder materials for the experiments as well

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