Fabrication of micro components with MSZ material using electrical-field activated powder sintering technology

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Abstract. The electrical filed Assisted Sintering Technology (FAST) process uses low voltage and high current, pressure-assisted sintering and synthesis technique, which has been used recently in materials processing. This method can be used to densify materials and create compounds, and it is similar to hot pressing, but the mechanism of the heating and powder densification are different. In this paper an innovative methodology has been adapted from the FAST process that can decrease the volume of the components into micro scale and called (Micro-FAST). This process is a rapid powder consolidation technology and shows the possibility to produce solid parts from powder material. Using Magnesia-Stabilized Zirconia (MSZ) powder material, several processing parameters have been investigated, such as pressure, heating rate, heating temperature and holding time, which helped to gain optimum results. In this paper Ø4.00mm × 4.00 mm and Ø2.00mm × 2.00 mm cylinder solid samples were shaped. The SEM and EDS have been conducted and the relative density has been examined and the results showed a very good fabricated sample with 99.83% relative density.

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

1. Introduction

Zirconia (ZrO2) is one of the most promising materials for a new generation of ceramics Zirconia stabilized by metal oxide has shown an outstanding structural stability which includes a very good resistance to corrosion under heat, aqueous solutions, high thermal conductivity, and irradiation stability. Moreover, Zirconia can be stabilized down to room temperature by being doped with Magnesia (MgO). The Magnesia-Stabilized Zirconia (MSZ) have excellent mechanical properties at high temperatures and are relatively inert to hostile environments. They have good impact and thermal shock resistance and are used in refractory applications. The MSZ is been used widley in the industry , such as structural ceramics, dentistry, wear parts, ceramics bearings, etc… [1].

Due to revolution of the industry, the demands now for making micro- or miniature-components increased where there is fast growth in applications in several sectors, such as automotive industry, telecommunications, bio-medical industry, information technology and home-use electronics products. Therefore, the non-

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traditional manufacturing technologies have been developed to improved productivity and economic effectiveness [2]. The Electric Current Assisted Sintering (ECAS) one of the promising technology that has been used widely these days. The ECAS technology is a general term for a class of consolidation methods which combining external electric field/currents with mechanical pressure for powder sintering. [3, 4]. ECAS have been achieved rarely until 1990 where significant research interests and industrial applications have been identified from many countries, especially Japan, China and USA. Many papers have been published and efforts have been made on tools and machines development for industrial-scale production for this technology. Several technologies, such as, Pulsed Electric Current Sintering (PECS), and Spark Plasma Sintering (SPS) and Plasma Assisted Sintering (PAS) have been developed, introduced and registered as patent. The SPS is being one of the most recognised and widely used technology for sintering many types of materials. The heating current divided mainly into two categories. The first one has a low-voltage and a high current with a characteristic waveform and it called Resistance Sintering (RS), The second one 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 [5].

Comparing with conventional sintering methods, such as pressure sintering or hot pressing, the ECAS process has many advantages, including a faster heating rate, a lower sintering temperature, a shorter holding time, the consolidation of difficult-to-sinter-powders, the elimination of the need of sintering aids, no need of cold compaction, less sensitivity to the characteristics of the initial powders, and marked comparative improvements in the properties of the consolidated materials.

Lange [6] claims that the densification process using the conventional powder-sintering approach 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 work, the concentration is on the application of combining the FAST (Field-activated Sintering Technology) with the forming process to produce 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. By using the combination between the two processes (forming and FAST) for making small components powder materials, the densification can be achieved 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 [7, 8] however, the feasibility of forming ceramic materials still needs to be investigated further. In this work the MSZ powders will be examined by using Micro-FAST process to investigate the feasibility of producing micro components from this powders.

2. Experimental Procedure

Two kind of MSZ powders were used as raw materials. The first one is MSZ without any binders and its bulk theoretical density is 5.82 g/cm^3. The second MSZ was with 5 wt% organic additives or organic binders and the theory density for this one was 5.72 g/cm. These organic materials burn out in two steps. Around 2.2 wt% organic material can be removed at 200°C and the other 2.6 wt% can removed around 380°C. The theoretical bulk densities going to be used as a reference to calculate the percentage relative density for each sample produced by the MSZ powder materials. The weight of the powder was calculated using a precision electric balance according to the calculated value (see Eq. 1) and then it filled into the die.

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

The experiment was conducted using the Gleeble 3800 thermal mechanical machine from Dynamic System. The parametrs that has been used in the experiment is been controlled by 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. **Error! Reference source not found.** shows the machine and the arrangement of the tooling for the experiments. The electrical field produced by the Gleeble 3800 machine has a high current (3000~30000 A) and low voltage (3~10 V).

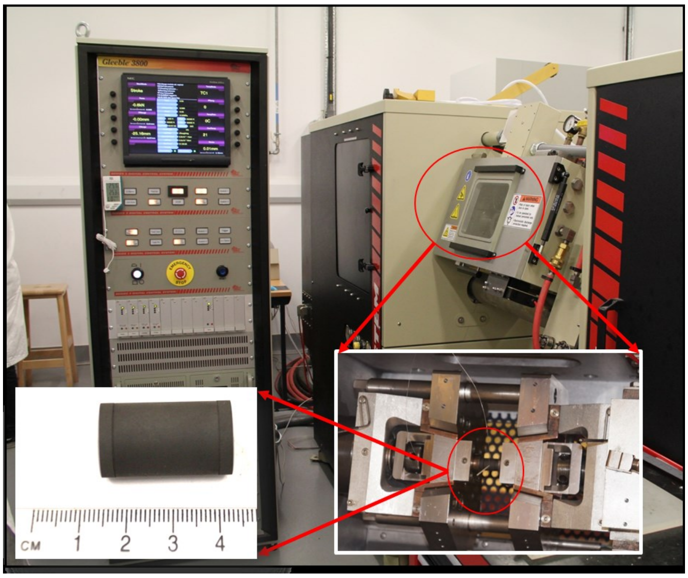

Figure 1 Tool set used and experiment setup with Gleeble-3800.

The received powders consisted of agglomerates which are sufficient to make up a sample with the size of Φ 4.0mm×4.0mm and Φ 2.0mm×2.0mm (solid cylinder). As shown in Figure 2, the closed die and punch set with filled powder was then placed horizontally between two electrodes on the Gleeble 3800. The selection of the graphite material was made by considering that the value for the thermal expansion co-efficient of the punch and the die must be less than that of the powder material being tested. to prevent any stuck between the punch and the die or sample becoming stuck during the ejection process. In addition, by using graphite, a higher sintering temperature can be used (up to 2500 °C or more). The graphite material has a low mechanical strength at high temperature is a major issue, a higher forming pressure cannot be applied during the electrical field activated sintering process [9, 10].

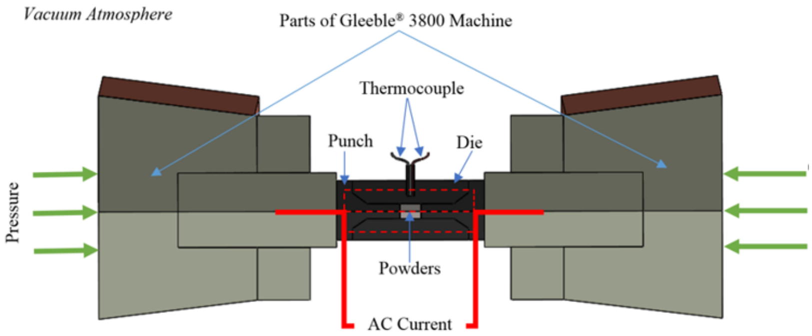

Figure 2 Schematic drawing of the Gleeble-3800 machine and Illustration of the Micro-FAST sintering and forming process

The experiments were conducted with variation of different key parameters, such as pressure, heating rate, maximum temperature and holding time. The MSZ 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 1400 ºC. As mentioned before the MSZ with binders has 5 wt% organic additives. Around 2.2 wt% organic material can be removed at 200°C and the other 2.6 wt% can removed around 380°C. Therefore, in order to form the MSZ powder with binders properly, the organic materials burn out in two steps during the sintering process. For that reason, the total time for the process is going to be longer.

3. Results and discussion

All experiment of MSZ powders have been successfully formed into solid cylindrical components. These formed samples were examined carefully using the sample geometry morphological measurement, calculating the relative density by the Archimedean approach, and microstructural observation under SEM. For the MSZ with binders, the results show that the highest relative density of 95.45%. Moreover, the results from MSZ powder without any binders shows a very promising results, which the relative density reached 97.53%. However, the previous results are for the 4.0mm×4.0mm cylindrical parts. For the Φ 2.0mm×2.0mm dies, the sintering temperature increased to 1400 ºC and the the results showed the highest relative density, wich is 99.47%. Table 1 shows the tested materials and its processing parameters and relative density.

Table 1 Details of the process parameters for the MSZ powders

Sample no.	**Materials**	**Sintering temperature (°C)**	**Heating Rate (ºC/s)**	**Sample size (mm)**	**Holding time**	**Pressure (Mpa)**	**Sintering cycle time (s)**	**Sample relative density**
1	MSZ with binders	1300	25	4x4	60	75-125	442.8	93.08 %
2	MSZ with binders	1300	25	4x4	5	75-125	387.8	93.51 %
3	MSZ with binders	1200	100	4x4	120	75-125	475.2	95.45 %
4	MSZ without binders	1300	50	4x4	120	125	309	96.36 %
5	MSZ without binders	1300	25	4x4	180	125	391	97.50 %
6	MSZ without binders	1300	25	4x4	120	125	331	97.53 %
7	MSZ without binders	1300	25	2x2	120	125	331	94.50 %
8	MSZ without binders	1300	100	2x2	120	125	298	95.34 %
9	MSZ without binders	1400	25	2x2	60	125	285	99.47 %

As shown in Table 1 the experiment that has been conducted for the MSZ with binders started started with 200°C and 380°C heating temperatures respectively to remove the organic binders, and during that time the 75 Mpa pressure is being used, and then increased to 125 Mpa after removing the binders and starting the sintering process.

Figure 3 shows the microstructure and the morphology of samples 3, 6 and 9. The formed samples are very good and strong and it not easy to be break. The formed samples were found to have similar dimension with the design of the dies (Φ 4.0mm×4.0mm and Φ 2.0mm×2.0mm). All three samples in Figure 3 look very good and shiny. The microstructure of the MSZ with binders sample shows has a really good surface and there are no pores. However, even though the MSZ without binders samples showed a high relative density, but there are a lot of pores on the surfaces of the two samples shown in Figure 3.

The EDS results showed an existence of carbon in the samples. This is might be due to 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 any contamination by other elements such as carbon when graphite dies and punches are used.

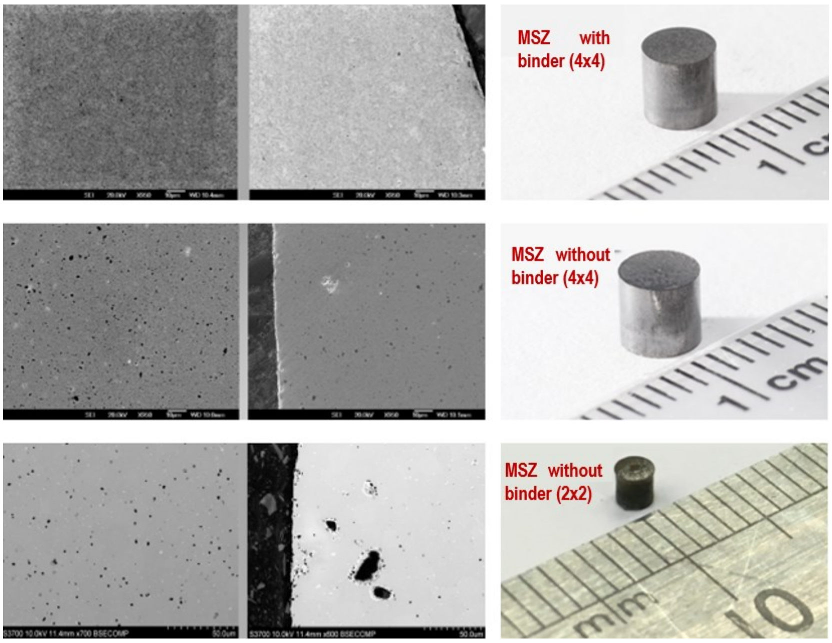

Figure 3 Formed samples (solid cylinder) with a size of Φ 4.0mm×4.0mm and Φ 2.0mm×2.0mm and thier SEM micrograph

4. Conclusion

In this paper, Micro-FAST showed the capability of the rapid forming of micro-components with MSZ material. In general, MSZ materials can be sintered successfully at a relatively low sintering temperature and sintering time.

The density for the MSZ with binders sample reached 95.45% with a pressure of 125 MPa, a heating temperature of 1300 °C, a heating rate of 25 °C/s and a holding time of 120 s. The optimum parameters for the MSZ without binders sample with a 99.47% relative density were: a pressure of 125 MPa, a heating temperature of 1400 °C, a heating rate of 25 °C/s and a holding time of 60 s.

This efficient process has the potential to save time compared to the conventional process. Moreover, it can be suggested that with different bulk theory densities, the similar process parameters can be used to gain higher densification.

Future work will be focussed on the investigation of influential sintering parameters in order to optimise the process and to increase its repeatability, to increase the quality of the parts to be manufactured.

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