Fabrication of Miniature Components from ZrO_2 Powder by Combining Electrical-field Activated Sintering Technique and Micro-forming

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Abstract. There is an increased demand for miniature/micro products (such as MEMS) and nanotechnology-based products (such as nano-materials). Micro-manufacturing is a link between Macro- and Nano Manufacturing and an effective means for transferring nanotechnology-product designs into volume production. The micro forming has the potential for low-cost, high volume manufacturing applications. In order to meet the high demands on miniaturised products, a rapid production technique and the system, high flexibility, cost-effectiveness and processing a wide range of materials are needed. Recently, a series of studies have been undertaken to investigate forming miniature/micro-components by using a combination of micro-forming and Electrical-field activated sintering (Micro-FAST). The process uses low voltage and high current density, pressure-assisted densification and synthesis technique, which renders several significant merits. The work to be reported in this paper will be focused on the forming of miniature components from Zirconia (ZrO_2) powder, without using binders. Several processing parameters have been investigated, such as pressure, heating rate, heating temperature and holding time, which helped to obtain high-quality parts. Using graphite dies and punches, sample parts (solid cylinders of Ø4.00mm × 4.00 mm) were formed. These were subjected to detailed examinations and analysis, such as analysis of the relative density, hardness at the necks formed among the particles and in the particle bodies, as well as the microstructures. The results showed that directly forming the parts from loose powder is feasible, and by properly designing and control the processing parameters, high-quality parts could be achieved, among which heating temperature and holding time are extremely important. At the same time, due to low conductivity of the powder material, carefully designing the tooling is essentials for ensuring properly heating, pressurisation and cooling.

Keywords: Micro forming, Sintering, Powder

1 Introduction

Zirconia (ZrO_2) is one of the most favourable materials for a new generation of ceramics due to excellent mechanical properties. When compared to other ceramic materials, zirconia has unique strength at room temperature. Moreover, Zirconia has high fracture toughness, high density, high hardness and wear resistance, good frictional behaviour, high-temperature capability, non-magnetic, low thermal conductivity, electrical insulation, the coefficient of thermal expansion similar to iron, and modulus of elasticity similar to steel. Nowadays, zirconia is being used widely in the production of many products, such as such as structural ceramics, dentistry, wear parts, ceramics bearings, etc.… [1-5]. Nowadays, there are high demands for micro- or miniature-components and there is a fast growth in its applications in several areas, such as automotive industry, telecommunications, biomedical industry, information technology and home-use electronics products. According to some market research, microelectromechanical systems (MEMS) market had a double-digit growth after 2010 and the estimation for

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MEMS market in consumer electronics grows at a compound annual growth rate of 19.8% during the forecast period 2009–2015 [6-8]. However, there still some challenges regarding the size-effect related issues, improving the quality of the product, delivering multi-material processing capabilities, and addressing all of these with low-cost. Moreover, the manufacturing of micro- products with traditional manufacture techniques for micro-electronics (e.g., photolithography, deposition and etching processes) is not always suitable since only a limited range of materials can be handled with a particular process, while other manufacturing methods such as conventional sintering and metal-powder injection moulding would need longer process chains. Therefore, the non-traditional manufacturing technologies have been developed to improved productivity and economic effectiveness [6, 9]. The Electric Current Assisted Sintering (ECAS) or Field Assisted Sintering Technique (FAST) is one of the promising technologies that have been used widely these days. The FAST technology is a general term for a class of consolidation methods which combining external electric field/currents with mechanical pressure for powder sintering. Comparing with other sintering processes, such as pressure sintering or hot pressing, the FAST process has many benefits. This methods has faster heating rate, lower sintering temperature, 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 [10, 11]. Therefore, a novel process used to produce miniature/micro-parts directly from loose powder by combining (FAST) and Micro-forming, was developed to address the issues raised above and it called and called Micro-FAST. It has been proved that the Micro-FAST process is particularly appropriate for forming micro/miniature components due to using very high heating and cooling rates and is of high flexibility for processing different powder-materials. The process uses low voltage and high current density, pressure-assisted densification and synthesis technique, which render several significant merits. To-date, high-quality parts and high process efficiency for forming with various powder materials have been achieved [12-14]

2 Process Configurations and Tools

The target samples to be formed were designed as size of Φ 4mm x 4mm cylinders. The loose powders were weighted using a precision electric balance according to the calculated value (see Eq. 1) and directly filled into the die cavity.

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

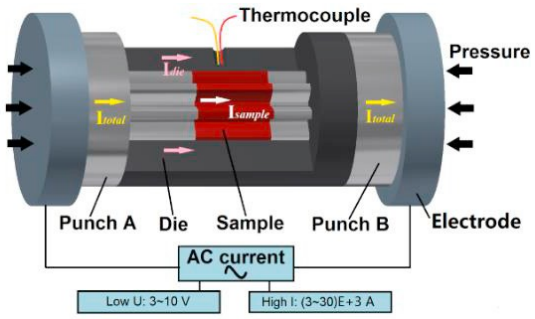

Figure 1 Illustration of the Micro-FAST process for the forming of miniature/micro- components [12]

Fig. 1 shows the principles of the micro-FAST process is being illustrated. The closed die and punch set with filled powder was then placed horizontally between two electrodes on the Gleeble 3800 machine as shown in figure 2. The Gleeble-3800 thermal simulation 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). By applying an external pressure and electrical-field through both punches, the Joule heat induced in a powder material and/or the die by the high electric current, plus possible electric plasticity in the powder material, could enable a combination of powder sintering and forming and hence, produce near-net-shaped micro-products within short time.

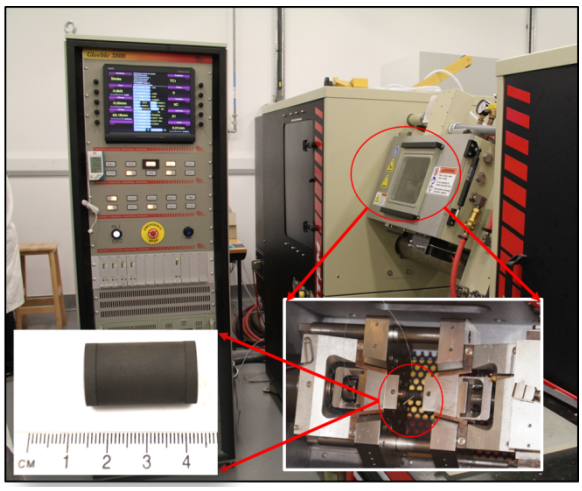

Figure 2 Tools set used and experiment setup with Gleeble-3800 [11]

The process parameters such as pressure, heating rate, temperature and time are input through a computer-based interface employing QuikSim Software. 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. This process can produce high-quality components with required structures and functionalities, through appropriate process design and control, with less or no restrictions on the raw-material types and the microstructures, including the use of nano-alloys.

3 Experimental Procedures

Zirconia (ZrO_2) powder with an average particle size of <50 μm was used for experiments. The theoretical bulk density for the used powder is 5.92 g/cm^3. The received powders were sufficient and ready to make up a sample with the size of Φ 4.0mm×4.0mm (solid cylinders). The experiments were conducted with a variation of different key parameters, such as pressure, heating rate, maximum temperature and holding time. The loose powders were weighted prior to being fed into the graphite dies (see figure 3). The graphite cylindrical dies and punches were designed and manufactured for the experiments to form 4.0mm×4.0mm 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. As mentioned, the pre-determined process parameters (pressure, heating rate, sintering/forming temperature and time) were set through the machine interfaces. Upon starting a program, a constant pressure was applied onto the powder through applying a force onto a punch, while the powder and die were heated up through a high density AC current passing through the powder and/or the die/tool-component, until the desired temperature was reached and the heating cycle completed. The temperature and pressure may be maintained for a given period of time for forming and densification, which was controlled by closed-loop control facilities. The ZrO_2 material has a very high melting-point and low electric conductivity. Therefore, the sintered temperature that has been set for the experiment was between 1100℃ and 1400 ℃ with a heating rate of 50℃/s.

Figure 3 Graphite Punch and Die set [15]

Figure 4 shows the steps and the sequence of the sintering process of ZrO_2 powder. At the beginning of the process, there was a gap between the punches and die. The gap decreased steadily during the heating period as the temperature increased. It can be shown that the gap gone during the temperature holding period which shows an ideal current flow. It is worth mentioning that the temperature distribution indications a high uniformity during the whole sintering process irrespective of whether or not the gap disappeared.

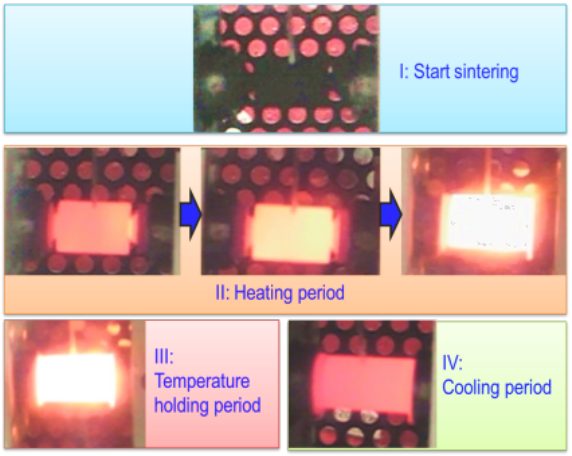

Figure 4 processing Sequence using the Gleeble 3800 [15]

Figure 5 shows the corresponding time-dependent stroke and sintering temperature were achieved with the Gleeble 3800 output setup. The graphs show the setting temperature, the actual temperature and the stroke versus the time during the forming process. In terms of temperature control, it can be seen from the curves that the actual temperature almost matches the setting temperature with only a small delay, from the beginning of the process. Once the stetting temperatures gained, the actual temperatures remain stable without any deviations. Moreover, matching with the applied pressure, the stroke curves decrease constantly: good material flow can be detected as soon as the force is applied. When the maximum temperature is achieved, it can be noticed there is a steep drop in the curve. Then, during the holding time, the stroke has no obvious changes, which means that the compact of the powder can be accomplished rapidly after the powder reaches maximum temperature. Nevertheless, the stroke changes during cooling stage, due to the shrinkage of the part that has been sintered. Comparing with the other sintering methods for ceramic powders [9] , Micro-fast sintering cycle with maximum sintering temperature of 1300°C is considered as a fast process.

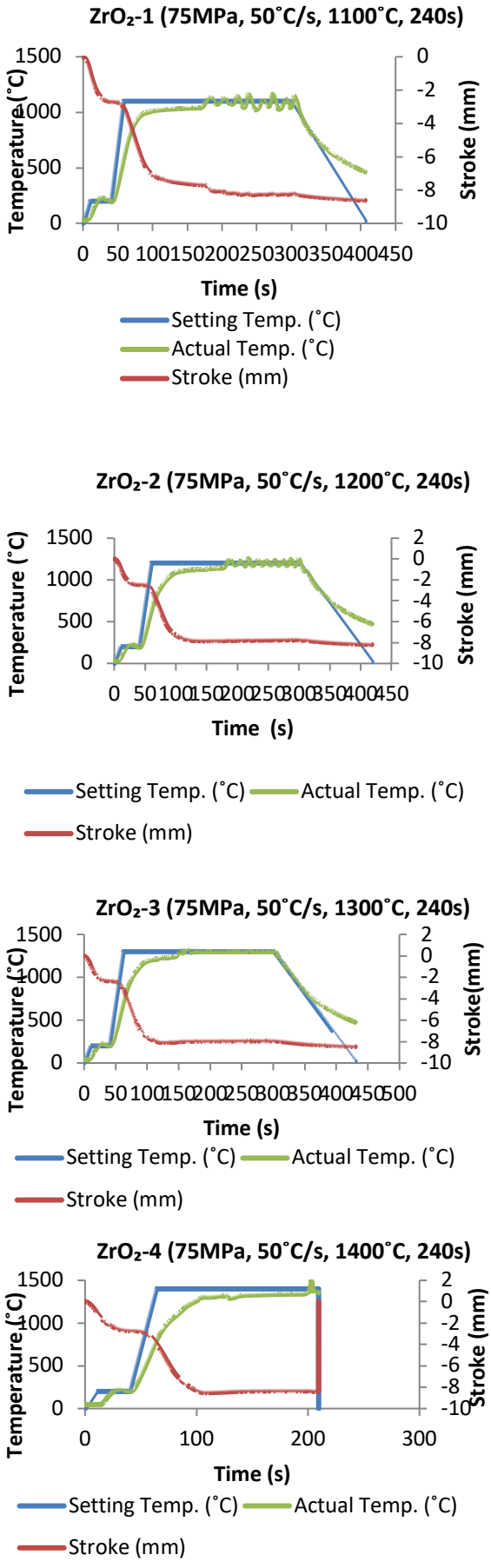

Figure 5 Process graphs of the forming/sintering of four samples parts of ZrO_2

4 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 ZrO_2 powders have been successfully formed into solid cylindrical components.

Table 1 The results of the formed samples of ZrO_2

Specimen designation	Sintering temperature (°C)	Sintering cycle time (s)	Sintered Part Density (g/cm³)	Sample relative density
ZrO_2-1	1100	405	5.279	94.27%
ZrO_2-2	1200	417	5.255	93.84%
ZrO_2-3	1300	429	5.427	96.91%
ZrO_2-4	1400	441	5.381	96.09%

As shown Table 1, the highest relative density of 96.91%. It can be predicted that the ZrO_2 powders sintering process can be improved by optimising the process parameters, such as by increasing the sintering temperature and holding time. The formed samples are very good and strong and it not easy tobreak it. The formed samples were found to have similar dimensions with the design of the dies (Φ 4.0mm×4.0mm) (see figure 6)

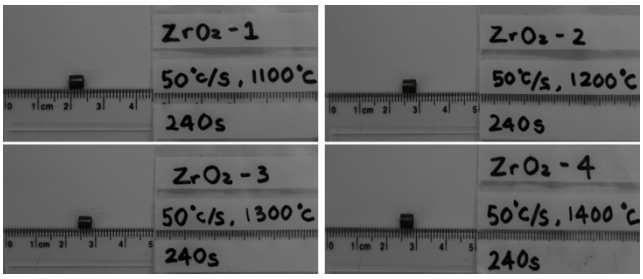

Figure 6 the Formed samples of ZrO_2 (4.0 mm in diameter and 4.0 mm in height).

Figure 7 shows the microstructure and the morphology of ZrO_2-3 sample. The formed samples are very good and strong look very good and shiny. The microstructure of sample shows a really good surface and there are no pores.

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.

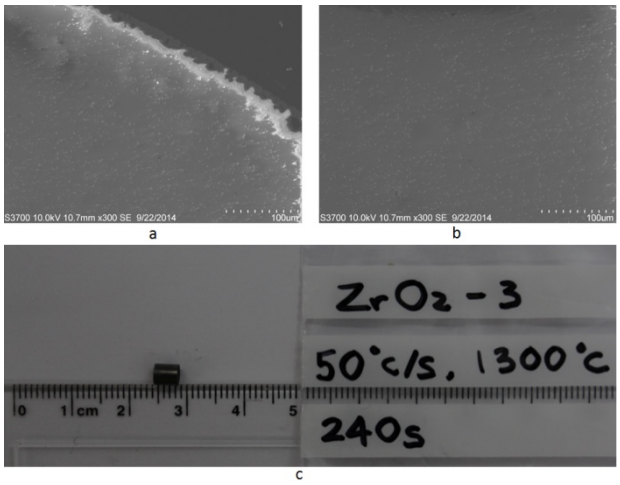

Figure 7 A formed sample (solid cylinder) with a size of Φ 4.0mm×4.0mm and its SEM micrograph

For the hardness test, the nano-indentation is used for the for the formed ZrO_2 -3 sample, 100 indentations were made on the sample: 10 in the sintering neck area and 10 in the particle centre (Fig. 8) It was found that the average Nano-hardness at the sintering for a Zirconia sample was 13.76 GPa. It is shown that the similar hardness values were found at both, the centres of the particles and necks, which indicates that good bonds among the particles had been formed.

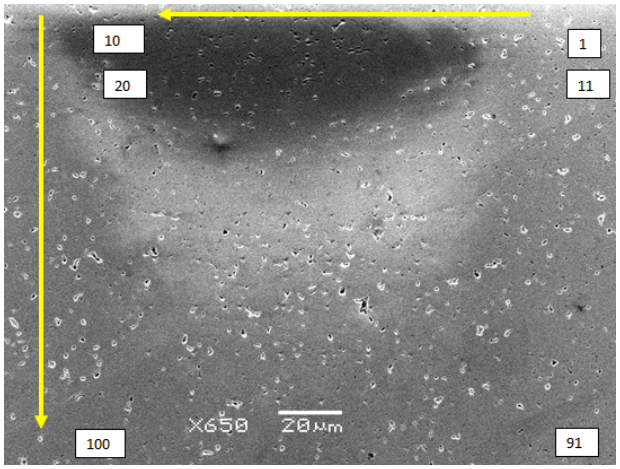

Figure 8 Position of nano-hardness indentation of Position of nano-hardness indentation of ZrO_2 -3 sample by using NanoTest Vantage hardness tester.

Table 2 shows the full datails of the average values for nano-hardness test that has been made using Nano-indintation approach.

Table 2 Average value for nano-hardness test for ZrO_2 -3 by using NanoTest Vantage hardness tester

Results	Units	Means	Errors
Max Depth	nm	373.3352	31.42479
Plastic Depth	nm	301.4467	33.32642
Maximum Load	mN	40.02	0.000001
Hardness	GPa	13.76014	2.329558
Reduced Young Modulus, Er	GPa	209.0603	16.32858
Elastic Recovery Parameter, ERP		0.241675	0.030812
Contact Compliance	nm/mN	2.395085	0.115092
Plastic Work	nJ	2.326256	0.531406
Elastic Work	nJ	301.4467	0.080666

5 Conclusions

The work conducted in this paper demonstrated that the capability of the Micro-FAST as a rapid process for the forming of micro/miniature- components. In general, Zirconia materials can be sintered successfully at a relatively low sintering temperature and sintering time. The Micro-FAST provides an effective alternative to other technologies. The sintered micro-parts were assessed on morphology, relative density, micro-structures and mechanical properties, which showed good quality having been reached. Comparing the Micro-FAST to other conventional sintering processes and some micro-manufacturing processes, it can be shown that the micro-FAST has several advantages over other processes/technologies. Moreover, it can be suggested as a promising micro-manufacturing technology.

The challenges that face this process and its engineering applications mainly concern tool-design and process awareness in environment of the industry. These are being addressed by the EU Micro-FAST consortium.

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

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References

1. Birkby, I. and R. Stevens. Applications of zirconia ceramics. in Key Engineering Materials. 1996. Trans Tech Publ.
2. Manicone, P.F., P. Rossi Iommetti, and L. Raffaelli, An overview of zirconia ceramics: basic properties and clinical applications. J Dent, 2007. 35(11): p. 819-26.
3. Denry, I. and J.R. Kelly, State of the art of zirconia for dental applications. Dent Mater, 2008. 24(3): p. 299-307.
4. Aytimur, A., et al., Magnesia stabilized zirconia doped with boron, ceria and gadolinia. Ceramics International, 2012. 38(5): p. 3851-3856.
5. Lee, S.-Y., Sintering behavior and mechanical properties of injection-molded zirconia powder. Ceramics International, 2004. 30(4): p. 579-584.
6. Qin, Y., Micromanufacturing engineering and technology 2nd ed. 2015, Oxford: Elsevier.
7. Petzoldt, F., Micro powder injection moulding-challenges and opportunities. Powder Inject Mould Int, 2008. 2(1): p. 37-42.
8. Meng, J., et al., Tribological behavior of 316L stainless steel fabricated by micro powder injection molding. Wear, 2010. 268(7-8): p. 1013-1019.
9. Orru, R., et al., Consolidation/synthesis of materials by electric current activated/assisted sintering. Materials Science and Engineering: R: Reports, 2009. 63(4): p. 127-287.
10. Lange, F., Densification of powder compacts: An unfinished story. Journal of the European Ceramic Society, 2008. 28(7): p. 1509-1516.
11. Hijji, H., et al. Fabrication of micro components with MSZ material using electrical-field activated powder sintering technology. in Advances in Manufacturing Technology XXX: Proceedings of the 14th International Conference on Manufacturing Research, Incorporating the 31st National Conference on Manufacturing Research, September 6–8, 2016, Loughborough University, UK. 2016. IOS Press.
12. Huang, K., et al., A new densification mechanism of copper powder sintered under an electrical field. Scripta Materialia, 2015. 99: p. 85-88.
13. Lu, D., et al., Forming microgears by micro-fast technology. Microelectromechanical Systems, Journal of, 2013. 22(3): p. 708-715.
14. Qin, Y., et al., Forming of Miniature Components from Powders by Combining Field-activated Sintering and Micro-Forming. Procedia Engineering, 2017. 207: p. 1212-1217.
15. Zhao, J., et al. Forming of micro-components by electrical-field activated sintering. in MATEC Web of Conferences. 2015. EDP Sciences.