Available online at www.sciencedirect.com

ScienceDirect

Procedia Engineering 207 (2017) 1212–1217

Procedia Engineering

www.elsevier.com/locate/procedia

International Conference on the Technology of Plasticity, ICTP 2017, 17-22 September 2017, Cambridge, United Kingdom

Forming of Miniature Components from Powders by Combining Field-activated Sintering and Micro-Forming

Y. Qin[a, *], J. Zhao[a], K. Huang[a,b], M. Zulkipli[a], H. Hijji[a], Y. Yang[b], M. Wu[b] and D. Yin[b]†

[a] *Centre for Precision Manufacturing, DMEM, The University of Strathclyde, Glasgow, UK*
[b] *School of Manufacturing Sciences and Engineering, Sichuan University, Chengdu, China*

Abstract

Micro-FAST is a process concept which scales down conventional FAST to the micro-scale process (dealing with miniature an micro-sized components) and it combines the sintering process with a micro-forming process to enable shaping components under coupled multi-fields actions and hence, to achieve high-density, near-net-shaped components with high efficiency. The main techniques developed for overcoming the barriers for the applications of FAST at the miniature/micro-scales include: (i). Directly pressing/forming loose powders in the die without using binders; (ii). Combining heating and shaping to enable complex shapes/features; and (iii). Dedicatedly controlling fusion bonding and material's plastic flow to enable high-quality forming. Forming from powders without using binders significantly shortened the process cycle, which also led to high-purity of the parts formed; Combining forming and sintering has led to high-density components produced as well as achieving complex-shaped components; Large current density (e.g., >100~400 KA/cm^{-2}) enables very high heating rates and using small volumes of materials results in high cooling rates, leading to much shorter forming/sintering cycles which enables consolidation of micro/nanocomposites into bulk-sized components while also preserving their micro/nanostructures.

Peer-review under responsibility of the scientific committee of the International Conference on the Technology of Plasticity.

Keywords: *Powder sintering, Field-activated Sintering Technology, Micro-forming, Micro-FAST*

* Corresponding author. Tel.:+441415483130.
E-mail address: qin.yi@starth.ac.uk

1877-7058 © 2017 The Authors. Published by Elsevier Ltd.
Peer-review under responsibility of the scientific committee of the International Conference on the Technology of Plasticity.
10.1016/j.proeng.2017.10.872

1. Introduction

Increased demands on the micro-/miniature-components due to miniaturization of products and systems have been a driver to the development of micro-manufacturing technology recently. Nevertheless, the challenges are still on dealing with size-effect related issues, improving product quality, delivering multi-material processing capabilities, and addressing all of these with low-cost [1]. At the same time, Manufacture of miniature and micro-components with traditional fabrication techniques for micro-electronics (e.g., photolithography, deposition and etching processes) as well as with micro-machining and forming is not always adequate since only a very limited range of materials could be processed with a particular process, while other manufacturing methods such as conventional sintering and metal-powder injection moulding would need longer process chains. Therefore, Micro-FAST – a novel process used to produce miniature/micro-parts directly from loose powder by combining Field-activated Sintering Technique (FAST) [2] and Micro-forming, was developed with a view to addressing the issues raised above. It has been demonstrated that the process is particularly suitable for forming micro/miniature components due to using very high heating and cooling rates and is of high flexibility for processing different powder-materials. To-date, high-quality parts and high process efficiency for forming with various powder-materials have been achieved [3-6]. This paper reports latest development of this processing technology by describing the recent results obtained and the understanding developed, regarding the process parameters, particle deformations and densification mechanism. The latest work particularly concerns extension of the previous research to a much wider range of the materials and component-forms that had not been addressed before.

2. Process Configuration

Fig. 1 illustrates the micro-FAST process working principle. 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. Fig. 12 shows examples of four possible tool-configurations to form four different parts, indicating the process flexibility. The process would be able to produce high-quality components with desired structures and functionalities, through proper process design and control, with less or no restrictions on the raw-material types and the microstructures, including use of nano-alloys.

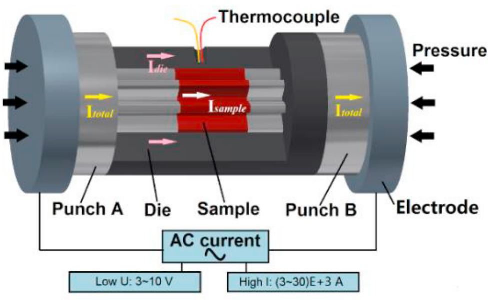


Fig. 1: Micro-FAST process for the forming of miniature/micro-components [2]

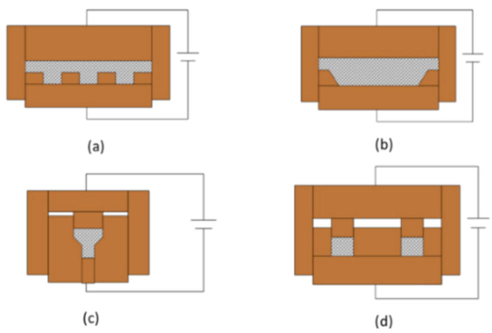


Fig. 2: Examples of possible tool configurations

3. Materials, Tools and Equipment

3.1. Materials

To-date, 25 kinds of powder materials (metallic and ceramics: for example, copper, stainless steel, titanium and alloys, alumina, SMA, PZT, cement, magnet) have been tested with the Micro-FAST process. The powders were produced using different techniques, such as spray drying or high-speed ball milling, with the average particle sizes of 5 ~ 50 micros, either provided by commercial suppliers or from project collaborators. Unlike conventional powder metallurgy, in which the powder is usually mixed with binder or additive before compaction, in the Micro-FAST process, the loose powder is directly fed into the die. By doing this, the binder-removal process is eliminated, which leads to high purity of the formed part and shorter sintering time.

3.2. Tools and Equipment

Depending on the powder materials to be sintered/formed (which determines the sintering temperature to be used, e.g. from 600 ^{0}C ~ 1300 ^{0}C), different tool-materials are selected for the die, punch and other tool-components (refer to the Figs. 1 and 2). Main considerations for the selection of the tool materials for the Micro-FAST process are electrical resistances, feasible working-temperatures, mechanical strengths/toughness and surface-resistance to wear at the elevated temperatures, of the tool-materials. Other considerations include thermal expansion coefficients of the tool-materials and the powder materials, resistance to the high-temperature oxidation and machinability of the tool-materials, etc. To-date, Graphite, Tungsten Carbide, TZM, Ceramics, etc. have been used as the tool-materials for forming different powder-materials and for different tool-components.

So far, three types of the machines have been used for the process development – from the lab. process tests to industrially relevant production, namely thermal simulator Gleeble 3800 from Dynamic Systems (in collaboration with Imperial College London), in-house developed electrical-forming machine at the University of Strathclyde, and an industrial version Micro-FAST machine, developed through the EU-funded Micro-FAST project.

4. Procedures

The loose powders were weighted prior to being fed into the dies. The pre-determined process parameters such as pressure, heating rate, sintering/forming temperature and time were set through the machine interfaces such as the computer screens. 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. For the metallic powders with lower melting points and good electric-conductivity, the sintering temperatures required could be less than 600 °C ~ 900 ºC with a heating rate of 100 ºC/s, while, for the ceramic materials with very high melting-points and low electric-conductivity, sintered temperatures as high as 1300 ºC could be needed with heating rates of 50 ºC/s approximately. In addition, for some powder materials and forming tools, forming/sintering within vacuum would be needed to avoid the oxidation of the powder materials as well as tool-materials. Fig 3 shows two examples of process graphs for two different materials where the temperature/stroke changes vs the time. The graphs reflect the forming stages where pre-loading/heating, heating/forming, forming/sintering, and final densification would occur. The stroke curves indicate the changes of the sample heights and the process of dimensional stabilizing during sintering and cooling. After the parts and tools cooled down, the parts are ejected out from the dies, being followed by polishing and post-inspections.

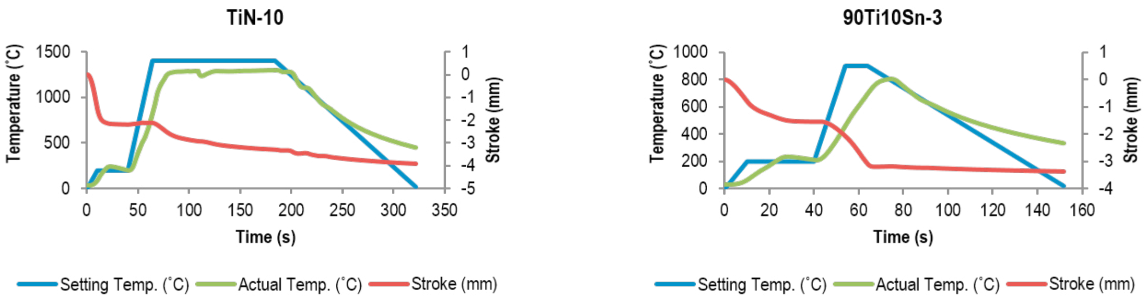


Fig. 3. Process graphs of the forming/sintering of two sample parts (4.0 mm in diameter and 4.0 mm in height).

The recorded process graphs and formed parts were examined using the following methods: appearance check and morphological measurement of the parts' geometry, relative density calculation, microstructural observation under SEM/TEM and mechanical property analysis (such as Micro/Nano-hardness tests). Once a part was ejected out from the die, the first assessment was on its physical appearance, such as the colour, completeness of the outer profiles/forms, surface roughness, contamination, etc. Relative density is one important measure used to assess the quality of a formed part. Based on the Archimedes method, the density of a sample was measured using an electronic analytical balance. The relative density of the formed part was determined simply by calculating the ratio of the measured density to the powder-material's theoretical skeleton density. In order to evaluate the mechanical property of the formed parts, Nano-hardness tests were conducted to evaluate the strength of the particle centre and sintering necks, using Nano Test 600.

5. Results and Discussion

All types of the powder materials studied have been successfully formed into solid bulk-components, with a relatively low temperatures and sintering time. The results demonstrated process feasibility and efficiency for those materials and component-forms attempted.

5.1. Quality of the parts formed

With good setting and controlling processing parameters, all parts, including cylinders, ring and triangle parts, were successfully formed with different powder-materials – some examples are shown in Fig. 4. In general, very good die-filling could be achieved with proper pressurisation and powder-flows during forming, being reflected by good appearance of the parts formed. There was time when the part geometry could not completed perfectly due to the inaccurate powder-volumes used, e.g. either less or excessive powder was fed into the die-cavity. It was also understood that surface appearance such as roughness is prescribed not only by the fusion-bonding of the particles achieved at the surface but also by the extent of the electro-plasticity induced in the particles. More components with more complex geometries have been attempted, such as the components with thin-walled structures (which are extremely challenging) and gears. These will be reported in the future publications.

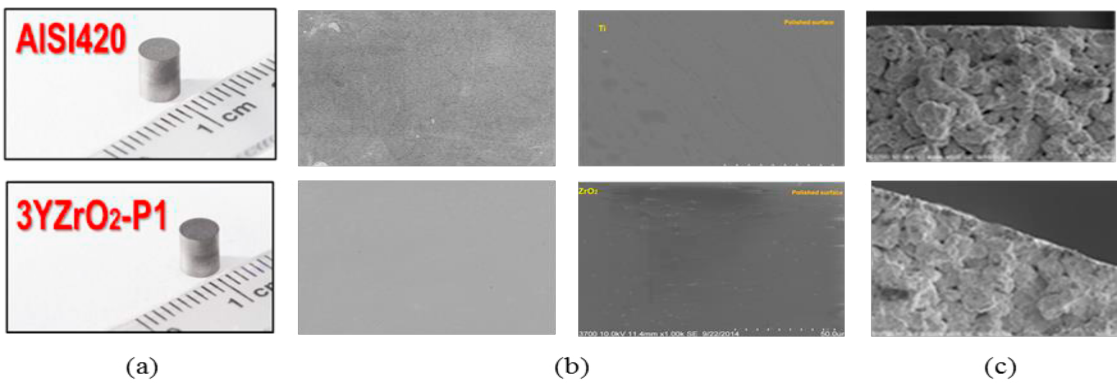


Fig. 4. Examples of: (a). Sample parts formed; (b). SEM micrographs of the parts (NiTi, Ti, AISI, ZrO_2) selected; and (c). Two samples formed under low and high pressure respectively.

For the metal powders that have been tested so far, a relative density up to 99.89% has been achieved (Fig. 4) for NiTi alloy, 98.49% for an Titanium Alloy and 95.83% for Stainless Steel. In addition, even with higher melting points and low electrical-conductivities, all of the ceramic parts could be formed to high relative densities, e.g., Zirconia 99.57% and Alumina 92.68%. Higher densities could be achieved with further process optimization.

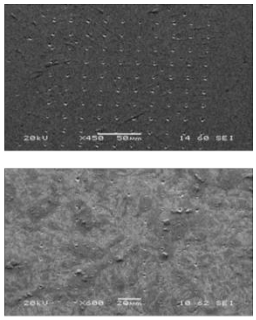
When the relative density reached above 90%, pores in the sintered parts were not obvious, being observed in SEM analysis. For powder metallurgy, it may not be necessary to achieve a part with 100% density, i.e., without any residual pores between particles. It is, however, important to ensure that the residual pores are small and uniformly distributed. For good formed parts, particle deformations and inter-particle necking could be observed clearly without seeing large pores. In addition, because of the short sintering time, there was no significant coarsening or grain-growth observed.

The formed parts were, generally, of high accuracy (Fig. 5(a)), considering being a high-temperature sintering/forming process. When the relative density was very high, e.g. 98.55% for the Ti-4 sample, the dimensional variation was about 10 micromeres for a part having a diameter of 4 mm.

In nano-indentation, 100 indentations were made on each sample: 10 in the sintering neck area and 10 in the particle centre (Fig. 5 (b)). It was found that the average Nano-hardness at the sintering necks for a Titanium-Tin alloy sample was 5.8101GPa, and for Titanium (Ti), it was 2.7994 GPa. In the particle centres, for Titanium-Tin alloy, it was 5.8520 GPa, and for Titanium (Ti), it was 2.7454/GPa. It is shown that the similar hardness values were found at both, the centres of the particles and necks at the interfaces for both materials, which indicates that good bonds among the particles had been formed. Some sintering necks even have higher hardness, comparing to the particles.

Parameters and Results 	Units	Ti-1	Ti-2	Ti-3	Ti-4
Diameter of the Sample (Ø: 4.00)	mm	3.97	3.97	3.98	3.99
Height of the Sample (H: 4.00)	mm	4.11	4.10	4.02	4.01
Sample Weight Before Cleaning (BC)	g	0.2132	0.2175	0.2064	0.2142
Sample Weight After Cleaning (AC)	g	0.2118	0.2144	0.2048	0.2121
Different Sample Weight (BC-AC)	g	0.0014	0.0031	0.0016	0.0021
Relative Density	%	92.24	95.57	96.23	98.55

(a)



(b)

Fig. 5. Measured data and nano-hardness tested samples: (a). Data for four Ti-samples; (b). Ti and Ti-Tin nano-indented sample.

5.2. Process parameters, particle deformations and densification mechanism

Quality of a formed part, e.g., relative density and pore distribution, is influenced by several material and process parameters, as illustrated in Fig. 6. The main parameters influencing the densification process are sintering/forming temperature and forming pressures applied. Comparing to a conventional powder metallurgy process where the sintering temperature is usually very high and it also takes very long time for removing binders (which also occurs in an micro-injection moulding process involving using metallic powders and post-sintering), e.g. several hours, Micro-FAST uses lower sintering temperatures and takes only a couple of minutes to complete the whole process cycle (Fig. 3). If the cooling process takes place separately from a forming/sintering facility, the whole forming-cycle could be even shorter, which suggests technological and economic merits, e.g., small micro-structures preserved without significant grain-growths. Recent effort in forming nano-phased materials with Micro-FAST further demonstrated this benefit.

Being different from a conventional sintering process, pressurization is of significant effect on the densification process, reflected by improved material-flows, increased particle deformations and rapid growth of the necks, comparing to that in a non-pressure process. This particular benefit takes advantages of the high-current-density induced plasticity in metallic powders to promote plastic deformations of the particles further.

While those two parameters (heating-temperature and forming-pressure) were constant, the heating rate seems to have a non-ignorable role in determining the final density formed, for the metallic materials tested. This may relate to the electrical-concentration at the contact areas, creep property of a material and electro-plasticity induced deformation of the particles. Early analysis [3] has already revealed the particle deformations to be a key mechanism in the Micro-FAST process, being different from a conventional powder-metallurgy process. How plastic flows of particles in Micro-FAST could influence in-process fusion bonding of the particles to form necks at the contact areas and hence, influence quality of the interfaces formed among the particles, needs to be investigated further.

Nevertheless, only small volumes of the materials being involved in Micro-FAST enables fast heating rates that could not, normally, be achieved in an FAST process for the sintering of a large sized component, which indicates an unique merit of electrical-field activated sintering for the forming of small sized components.

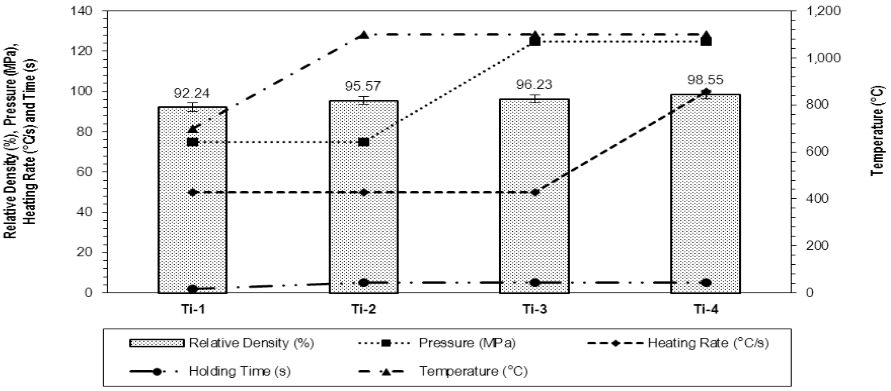


Fig. 6. Illustration of relative density and influences from process parameters for Titanium sample parts

6. Conclusions

The work conducted so far demonstrated that Micro-FAST is a rapid process for the forming of micro/miniature-components with a wide range of materials, especially with difficult-to-cut and difficult-to-form materials, which provides an efficient alternative to the micro-manufacturing technology. Different kinds of powder materials with various particle sizes have been sintered successfully to the different shapes/geometries, including that from Ceramic materials such as Zirconia and Alumina. The sintered micro-parts were assessed on morphology, relative density, micro-structures and mechanical properties, which showed good quality having been achieved. Comparing to other conventional sintering processes and some micro-manufacturing processes involving sintering, Micro-FAST uses relatively low sintering/forming temperatures and short processing time, which renders several advantages over other processes/technologies and hence, it is a promising micro-manufacturing technology. The challenges to its engineering applications mainly concern tool-design and process realisation in industrial environment. These are being addressed by the EU Micro-FAST consortium.

Acknowledgements

The authors would like to acknowledge the fund from the European Commission through FP7 FOF Micro-FAST Project (GA No. 608720) for supporting the development of Micro-FAST manufacturing facilities. The fund from UK Royal Society through an international exchange programme to support the research in the Micro-FAST process is also acknowledged. The authors would like to specially thank Prof. Jianguo Lin and his colleagues in Imperial College London for providing Gleeble machine for conducting the experiments, and thank EU Micro-FAST project partners who provided powder materials for conducting the experiments.

References

[1] Y. Qin, Micro-Manufacturing Engineering and Technology (2nd Edition), Elsevier, Oxford, May 2015.
[2] R. Orru, et al, Consolidation/synthesis of materials by electric current activated/assisted sintering, Materials Science and Engineering R, 63, pp. 127-287, 2009.
[3] K. Huang, Y. Yang, Y. Qin, G. Yang & D. Yin, A new densification mechanism of copper powder sintered under an electrical field , Scripta Materialia, 99, pp. 85–88, 2015
[4] D. Lu, Y. Yang, Y. Qin, & G. Yang, Forming Microgears by Micro-FAST Technology, Journal of Microelectromechanical system, 22 (3), pp.708-715, 2013
[5] J. Zhao, Y. Qin, K. Huang, M. Zulkipli and H. Hijji, Forming of micro-components by electrical-field activated sintering, MATEC Web of Conferences, 21 (10001), 2015