euspen's 18th International Conference & Exhibition, Venice, IT, June 2018
www.euspen.eu

Process parameter influence on Electro-sinter-forging (ESF) of titanium discs

Emanuele Cannella[1, 2], Chris Valentin Nielsen[2], Niels Oluf Bay[2]

[1]*IPU Innovation Factory, 2800 Kgs. Lyngby, Denmark*
[2]*Technical University of Denmark (DTU), Department of Mechanical Engineering, 2800 Kgs. Lyngby, Denmark*

emcann@ipu.dk

Abstract
Electro-sinter-forging (ESF) is a sintering process based on the resistance heating principle, which makes it faster than conventional sintering. The process is investigated as a function of the main process parameters, namely compacting pressure, electrical current density and sintering time. The present work is focused on analysing the influence of these process parameters on the final density of a disc sample made from commercially pure titanium powder. Applying the design of experiments (DoE) approach, the electrical current was seen to be of largest influence. The maximum obtained density was 94% of the bulk density of pure titanium. Density measurements were carried out by measuring the mass and volume separately. The volume was estimated applying two methods, namely the Archimedes' suspension method and 3D scanning to build and measure the volume mesh of the sample. The density calculations proved to be compatible.

Electro-sinter-forging; titanium powder; design of experiments; density measurements;

1. Introduction

Sintering processes generally deal with ceramic and metal powders. The main advantages of sintering are the possibility of manufacturing near net-shape components and to process material only available as powder. Machining is avoided and post-removal processes are limited to the surface polishing, whereby material waste is kept minimal. A conventional sintering process consists of two main phases, namely compaction and sintering. Firstly, the powder is compacted in a closed-die setup. The manufactured powder compact is called green body. On the second phase, the green body is ejected from the die and kept in a temperature controlled sintering oven for a specific time [1]. The oven temperature is below the melting temperature of the sample material. Complete sintering can last up to 30 minutes, depending on the material. As a consequence of the high temperature, the particles are joined by metallic bonding. New sintering techniques, Electro-current-assisted-sintering (ECAS) processes [2-3], are focused on decreasing the total process time. Problems connected to grain growth, oxidation and extended production time are avoided. These processes make use of Joule heating to heat the green and/or the die [4-5]. The powder is firstly compacted. The obtained green body is subsequently sintered inside the same die and the compacting pressure is kept during the whole process. The sintered sample is therefore ejected after the sintering has been completed. One of the ECAS processes is Electro-sinter-forging (ESF) [6], where only the compact is heated by Joule heating while the die is electrically insulated.

Sintered components are compared to the bulk properties as reference. Therefore, the final density and porosity distribution are analysed [7]. In the present work, eight different process conditions were tested by applying the Design of Experiments (DoE) approach. Two repetitions were carried out. The three main process parameters, compacting pressure, electrical current density and sintering time, were tested at two different levels, high and low. To conclude the influence analysis, the sintered components were evaluated in terms of density, due to the importance of this property in ESF processes [6].

2. Case study

The sintered component is a disc made of commercially pure titanium powder, see an example in Figure 1. The powder was 99.5% pure with 150 µm particle size. The sintering process was carried out in an electrical-resistance-welding setup by Expert Maschinenbau GmbH, Figure 2, which is mechanically operated by hydraulics and disc springs for follow-up of the force. Therefore, the applied force is kept constant during the whole process. The DC current is delivered by a unit from Harms+Wende GmbH.

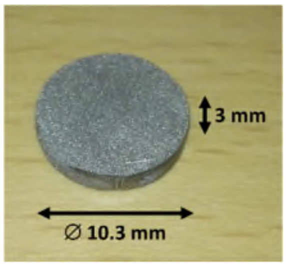

Figure 1: Sintered titanium disc.

Figure 2: Electrical resistance welding setup.

The tool setup consisted of an alumina insert surrounded by a steel container to strengthen the die, Figure 3 and Figure 4. The electrodes/punches were made of electrolytic copper. Teflon tape was used to insulate the steel container from the electrode/container contacts. The density was analysed by measuring the mass and volume of each sample. The mass was measured by a precision balance (Sartorius Stedim Biotech GmbH) and the volume in two different ways, namely the Archimedes' suspension method [8] and 3D scanning (3shape) to build the sample mesh. The volume was estimated from the sample mesh by using a software for dimensional analysis (GOM GmbH).

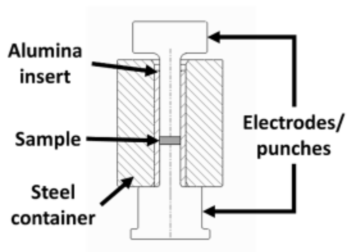

Figure 3: Detail of the tool setup.

Figure 4: Tool setup for ESF.

2.1. Density uncertainty

The density was measured as:

$$d = \frac{m}{V} \quad (1)$$

where *m* and *V* are the mass and volume of the sample, respectively. The density uncertainty was evaluated according to the error propagation formula for independent inputs [9]:

$$u_d = k\sqrt{\left(\frac{1}{V}\right)^2 u_m^2 + \left(\frac{m}{V^2}\right)^2 u_v^2} \quad (2)$$

where *k* is the coverage factor (95%), u_m and u_v are respectively the mass and volume uncertainties connected to the resolution of the measuring instruments. u_m was read from the resolution of the balance to 5 mg. u_v depended on the measuring method applied: 2 mm^3 from the resolution of the 3D scanner and based on the average volume of three repeated measurements per sample for Archimedes principle.

3. Experimental results

To accomplish the DoE analysis, two levels, low and high, where applied for each process parameter, Table 1. Process values were the nominal one. During the process, both the electrical current density and compaction pressure slightly decreased due to material property changing.

Table 1: Low and high nominal process values used for DoE.

	(P) compacting pressure/MPa	(E) electrical current density/A/mm^2	(t) sintering time/ms
Low (-)	69	58	100
High (+)	115	115	200

The density results showed compatibility between the two applied methods, Figure 5. The error bars in Figure 5 are based on Eq. (2).

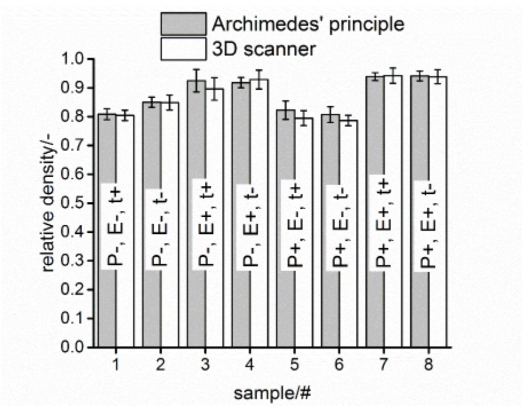

Figure 5: Calculated relative densities for the sintered samples. Bulk density 4.5 g/cm^3.

The DoE results in Figure 6 highlighted the electrical current density as the most influencing factor on the final density. The error bars in Figure 6 are the largest ones estimated with Eq. (2), which corresponds to 4% of the bulk density.

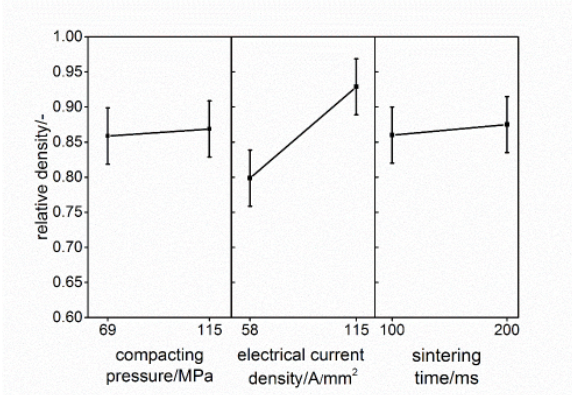

Figure 6: DoE results showing the influence of process parameters on relative density.

4. Conclusions

The influence of the process parameters on the final density in electro-sinter-forged discs was investigated by the DoE approach. For the experiments, commercially pure titanium powder was used. Two levels for each process parameter were investigated, and eigth different experimental conditions were tested. The results show, that the electrical current density is the most significant parameter on influencing the relative density in Electro-sinter-forging. With the high current density, a relative density up to 94% was achieved. Sintering time and compacting pressure did not show a similar influence. Further investigations could be focused on testing larger values for these parameters and establishing a trade-off combination for this case study. However, the melting point of the material has to be considered the limiting condition. Other conductive powders can be investigated to prove the same trend. Measurements of relative density proved compatibility in volume measurements based on Archimedes and use of 3D scanning. The two methods can be considered equivalent for volume measurements. 3D scanning can be suggested in the case of volume measurements of sintered samples because fluid penetration problems connected to Archimedes' method can be avoided.

Aknowledgements

This research work was undertaken in the context of MICROMAN project ("Process Fingerprint for Zerodefect Net-shape MICROMANufacturing", http://www.microman.mek.dtu.dk/). MICROMAN is a European Training Network supported by Horizon 2020, the EU Framework Programme for Research and Innovation (Project ID: 674801).

References

[1] R. H. R. Castro, Sintering, vol. 35, R. H. R. Castro and K. van Benthem, Eds. Springer, 2013, pp. 1–16.
[2] S. Grasso, Y. Sakka, and G. Maizza, Sci. Technol. Adv. Mater., vol. 10, no. 5, p. 53001, 2009.
[3] Z. A. Munir, U. Anselmi-Tamburini, and M. Ohyanagi, J. Mater. Sci., vol. 41, no. 3, pp. 763–777, 2006.
[4] O. Van der Biest, Ceram. Compos. Process. Methods, pp. 43–73, 2012.
[5] J. R. Groza and A. Zavaliangos, Mater. Sci. Eng. A, vol. 287, no. 2, pp. 171–177, 2000.
[6] A. Fais, J. Mater. Process. Technol., vol. 210, no. 15, pp. 2223–2230, 2010.
[7] A. Fais and G. Maizza, J. Mater. Process. Technol., vol. 202, no. 1–3, pp. 70–75, 2008.
[8] S. W. Hughes, Phys. Educ., vol. 40, no. 5, pp. 468–474, 2005.
[9] ISO/IEC Guide 98-3:2008, 2008.