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Consolidation of iron powder by electrical discharge

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ABSTRACT

Capacitor electrical discharge consolidation (CEDC) is a technique that uses the heat of the Joule effect of a high intensity electric current to consolidate powders. In this study, the effect of the precompaction pressure and the number of discharges on the porosity, microstructure and hardness of the compacts is analysed. Furthermore, the sintering results of iron powders obtained through the conventional route (cold pressing and furnace sintering) and by CEDC are compared. Experiments show that at low initial pressures the powder column has the necessary resistance to produce the joule heat necessary for powder consolidation. At an initial pressure of 200 MPa the porosity of the specimens decreases from 0.32 to 0.24, and the Vickers microhardness increases from HV10 29 to HV10 51 after 50 discharges. Copyright © 2022 Elsevier Ltd. All rights reserved.

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1. Introduction

The production of parts from different materials can be done in different ways. Traditionally, the most used way is to press the powder to obtain green (mechanically joined) parts and then sinter them in a furnace at a temperature below the melting temperature of the material, using a protective atmosphere to prevent oxidation of the material. This process is known as powder metallurgy (PM) [1].

Alternative to conventional powder metallurgical techniques have been developed based on the application of pressure and the passage of an electric current. These are relatively fast processes that can even be carried out without a protective atmosphere [2], such as electrical resistance sintering (ERS), or CEDC [3].

The sintering of the material is caused by the heat generated by the Joule effect when the electric current passes through the powder column, which is why it is necessary to use an insulating die, usually of a ceramic nature, to avoid current drifts.

The more extended technique is the ERS [4,5]. In this technique, the process is mainly controlled by selecting the high intensity and low voltage of the current, as well as the current passing time. The

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applied pressure is usually limited because of the use of highly conductive and ductile Cu-based electrodes and the resistance of the ceramic die. A practical way to implement this technique is through the use a spot-welding machine [6], which conveniently adapted can fit the process requirements. One of the drawbacks of this technique can be the limited amount of energy to be applied in a reasonable time (in the order of the second in ERS), because of the maximum intensity and voltage of the current proportioned by the equipment.

Nevertheless, for powders that are difficult to sinter, for instance because of the presence of oxide layers, or when the high temperature time must be minimized, it is necessary to use higher power techniques. An option to consider in this case is to store energy in a capacitor that can be quickly discharged (in the order of the millisecond), at the time that the powders are pressed at relatively low pressures. This is the known as Capacitor Electrical Discharge Consolidation (CEDC), developed during the end of the 1970 s and begin of the 1980 s [7,8]. The required energy is controlled by the capacity and the charge voltage, with a linear and a square dependence on these parameters respectively. In practice, custom high voltage equipment is used. For this, high voltage equipment (2-20 kV) capable of providing energies of 8-75 kJ are generally used [9-13]. This causes heating of the contact points between particles, without excessive effect on the rest of the particle. The equipment used for this study is however a stud welding equipment adapted to perform the discharge on Fe powders. Due

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Abbreviations: PM, Powder Metallurgy; CEDC, Capacitor electrical discharge consolidation; ERS, Electrical resistance sintering; FAST, Field-assisted sintering techniques.

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Fig. 1. Stud welding equipment and pneumatic press used in the experiments.

to the limited voltage of this equipment, of the order of 200 V, successive discharges are carried out to study the effect on the powders.

2. Material and experimental procedure

2.1. Capacitor discharge consolidation equipment (CEDC)

A stud welding equipment of capacitance C = 132 000 μ F and charging voltage V = 200 V is conditioned with Cu electrodes that actuate in a sialon die of 8 mm inner diameter (Fig. 1).

2.2. Material

A commercially pure iron, Atomet 1001HP from Rio Tinto Metal Powders, is used as starting powder (Fig. 2). The main impurities are 0.004 wt% C, 0.091 wt% Mn, 0.0053 wt% S and 0.06 wt% O. This 99.8 % purity atomised powder with mean particle size of 162 μ m (Fig. 2) were measured, the latter by laser diffraction (Mastersizer 2000, Malvern Panalytical Itd., Malvern, UK), was then vibrated until the powder particles reached their tap density [14] of 3.6 g/ cm³ (porosity of 0.63 ± 0.05). The absolute density is 7,9 g/cm³ and apparent density 2.9 g/cm³.

Fig. 3 shows the compressibility curve of the iron powder used in this study (applied pressure (MPa) versus porosity (Θ)).

2.3. Experimental procedure

1 g of Fe powder is introduced inside the sialon die. A pneumatic press applying 200 MPa ensures good contact between the electrodes and the powder. Between 0 and 50 discharges are





Fig. 3. The compressibility curve of the iron powder.



Fig. 4. Final porosity (Θ) versus specific thermal energy (STE).

performed at 5 s intervals. The variation of the porosity (determined by weighing and measuring the specimen), microstructure and HV10 Vickers microhardness (DuraScan 50G5, Emcotest, Kuchl, Austria) of the compacts has been analysed as a function of the number of discharges and green compaction pressure (200, 400, 700 and 1000 MPa). For comparison purposes, a sample of the same Fe powder was prepared by cold pressing and furnace sintering (500 MPa and 1175 °C for 30 min in Ar).

3. Results and discussion

3.1. Porosity evolution

The final porosity of the compacts Θ as a function of the green compaction pressure (P) and the specific thermal energy (STE = 1/



Fig. 2. (a) SEM micrograph of the Fe Atomet 1001HP powder; (b) Granulometric curve of the powder, showing a monomodal distribution.

 $M \cdot n \cdot 1/2 \cdot CV^2$, being M the powder mass and n the number of discharges) used to consolidate the powders is shown in Fig. 3.

As shown in Fig. 3, the final porosity is affected by the green compaction pressure and the number of discharges, varying from 0.28 for 200 MPa and five discharges (13.52 kJ/g) to about 0.095 for 1000 MPa (independently of the number of discharges). It could be expected not to find a high dependence of the porosity on the number of discharges, because CEDC is a so quick process that, although acting to form bridges between the powder particles, it usually has little influence on the porosity of the specimens. It is known that a high temperature of 10^3 - 10^4 °C can be attained in the contacts between powder particles, but the particles core does not suffer this temperature increase [15]. Only the pinch effect caused by the current passing acts to shrinkage the powder column when external pressure is not applied [16,17].

Only for the specimens pressed at 200 MPa densification is observed, with the porosity decreasing by 30 % up to 0.235 after 50 discharges.

Fig. 5 shows the porosity distribution in the green compacts and after 50 discharges for the different green compaction pressures.

As can be seen in Fig. 5, the porosity of the specimen with initial pressure of 200 MPa decreases after 50 discharges, while for the other initial pressures no change in porosity is observed. Fig. 4 shows that there is little difference between the centre and the periphery of the specimen, i.e., the porosity inside the specimen is homogeneously distributed, since the fast CEDC process does not overheat the powder particles, but only their contacts, obtaining a porosity distribution similar to that obtained after the precompaction process.

3.2. Microstructural evolution

Fig. 6 shows the microstructural evolution of the specimens and the porosity distribution of the conventionally sintered specimen. It can be seen that only the specimens with green compaction pressure of 200 MPa shows a certain densification, while the consolidation process is not effective for higher green compaction pressures. The reason for this latter is that the powder mass behaves as a good electrical conductor, hence the absence of heat generated by Joule effect. Interparticle boundaries can be easily appreciated in this situation even after 50 discharges.

3.3. Microhardness evolution

Fig. 7 shows the evolution of the microhardness (HV10) for samples with green compaction pressures of 200 and 400 MPa, after different number of discharges.

The microhardness increases with increasing the green pressure, and in the case of the specimens pressed at 200 MPa and 400 MPa, also with the number of discharges. There are no relevant changes in the microhardness of the samples with higher green compaction pressures. These results confirm the analysis of the porosity and microstructure evolution, with the hardness increase being due to the bonds between particles created by the discharges. For green compaction pressures of 700 and 1000 MPa, all microhardness values for different number of discharges are within the experimental uncertainty, with no inter-particle bonds produced because of the low heat generated at the particles contacts.



Fig. 5. Optical macrographs showing the porosity distribution of the CEDC specimens after 0 and 50 discha.

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Fig. 6. Micrographs showing the porosity distribution for (a) different number of discharges with green compaction pressures of 200, 400, 700 and 1000 MPa; (b) the conventionally sintered compact. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)



Fig. 7. Hardness for a compact initially pressed at (a) 200 MPa after 0-50 discharges; (b) 400 MPa after 0-50 discharges.

The conventionally processed specimen, pressed at 500 MPa, resulted in a value of 67 HV10, slightly lower than that of the electrically consolidated specimens compacted at a lower pressure of 400 MPa, even though the high temperature reached during furnace sintering and the much shorter processing time for the CEDC specimens.

4. Conclusions

Specimens obtained by CEDC show a homogeneous porosity distribution. This porosity is strongly dependent on the green pressure (as demonstrated by the compressibility curve of iron powder, Fig. 3), with higher values for low compaction pressures. Samples with green pressures of 200 MPa present a porosity range from 0.32 for green specimens to 0.24 for 50 discharges, showing a densification of 25 % of the powder mass during the consolidation process. For green pressures of 700 and 1000 MPa, no change in porosity is observed after the electrical discharges.

The compacts microhardness increases with increasing the green pressure. For 200 MPa, the microhardness increases from 29 HV10 for the green specimen to 51 after 50 discharges, while for 400 MPa the values range from 41 for the green specimens to 70 after 50 discharges. For the specimens with initial pressures of 700 and 1000 MPa, the efficiency of the consolidation process decreases as the powder mass behaves as a conductor, no heat is generated by the Joule effect, and no bonds are formed between particles. The specimens have the same hardness in green state and after the discharges.

If we compare the CEDC process with the SRE process [18], the compacts obtained have the same porosity value, e.g. for a porosity of 0.24 a slightly higher microhardness of 58 HV1, however, when comparing the CEDC process with the conventional sintering process (67 HV10), it is concluded that the discharge consolidation technique achieves a slightly higher hardness (70 HV10) with a lower starting pressure and with the advantage of being a quick and cheaper process.

CRediT authorship contribution statement

R.M. Aranda: Methodology, Validation, Writing – original draft. **F. Ternero:** Methodology, Validation. **R. Astacio:** Methodology, Validation. **P. Urban:** Conceptualization, Writing – review & editing. **F.G. Cuevas:** Conceptualization, Writing – review & editing.

Data availability

Data will be made available on request.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Petr Urban reports article publishing charges was provided by University of Seville. Rosa Maria Aranda Louvier reports a relationship with University of Huelva that includes: employment.

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