EVALUATING THE REDUCTION OF POROSITY OF Fe-Cr ALLOY PRODUCED BY POWDER METALLURGY

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ABSTRACT

The powder metallurgy technique has been used to produce a wide variety of products metallics. Normally, the industry uses rigid dies to uniaxially press the material because it is a cheap technique that allows automation. However, after the sintering step, care must be taken about the porosity remaining in metallic parts. Thus, some parameters or additional steps must be evaluated to obtain high densification and, consequently, better mechanical properties. In general, few works in the literature approach this subject in a comparative way. In this sense, this work will vary some parameters such as the use of lubricant, two methods of compaction, forging and annealing after sintering to evaluate the porosity in each step performed of a Fe-Cr alloy. The results prove that performing a second compaction method after uniaxial compaction significantly reduces porosity. As well, the implementation of thermomechanical and thermal treatments improves the final densification.

Keywords: *Powder Metallurgy. Porosity. Hot Forging.*

INTRODUCTION

The production of alloys by powder metallurgy technique proves to be advantageous in several cases, such as in the production of ODS (*oxide dispersion strengthened*) alloys, and high melting point metals. In the case of ODS alloys, the possibility to use low temperatures in powder metallurgy, compared to conventional metallurgy, prevents the formation of undesirable phases and the coarsening of oxide particles(1,2). However, it is impossible to obtain a full density alloy by powder metallurgy. The remaining porosity is a stress concentrator that impairs final mechanical properties, as well as magnetic, wear, corrosion, and thermal conductivity properties of the alloy(3,4).

Powder metallurgy consists of milling the starting powders followed by pressing and sintering. Milling or mechanical alloying is an economical method that converts a heterogeneous mixture into extremely fine particles with similar compositions (1,5). During pressing, the densification and shaping of milled powders occur simultaneously with the application of pressure, resulting in a green compact with a certain strength that allows its handling(6). Green strength is influenced by pressure and time pressing, lubrication method, characteristics and amount of powder(3,4,7). After pressing, the powders are sintered. The sintering is a heat treatment with a temperature below the melting temperature of the main constituent that generates an increase in strength and density due to joining the particles by diffusion(6). Some factors during this process, such as incomplete sintering due to insufficient temperature and time and oxidation processes, can impair the reduction of pores and, consequently, the final density of the material(4,7,8).

To reduce the remaining porosity and, consequently, improve the mechanical properties of the final product, it is necessary to carefully adjust the parameters during the powder metallurgy steps. An example is the use of lubricants, such as ethylene bis stearamide, polyvinyl alcohol, or boron nitride, to facilitate the accommodation of particles during pressing. In addition, some secondary techniques can be added before sintering, for example, cold isostatic pressing and double-pressing, or after sintering, such as hot rolling or hot forging(1,4,6,7,9-11).

Cold uniaxial pressing is the most common method for the shape of ferrous parts produced by powder metallurgy, being very suitable for large scale production(6). In many applications, high densification is required to ensure good mechanical properties. However, few works study the application of different parameters during and/or after the powder metallurgy technique to reduce the remaining porosity that is quite common in this technique. In this sense, some parameters used in powder metallurgy, such as the addition of lubricant, two pressing methods followed, and additional steps, were studied to increase the final densification of Fe- Cr alloys.

MATERIALS AND METHODS

Fe-14Cr alloys were produced by powder metallurgy. Metallic iron and chromium powders obtained (Dinâmica company) have different particle sizes and purity of 99% and 98%, respectively, according to the company information.

The metallic iron and chromium powders were milled in a RETSCH PM100 high energy ball mill. Mechanical milling was performed for 48 hours in cycles of 4 min/350 rpm and 1 min/0 rpm to prevent overheating and to reduce the contamination of the sample. Stainless steel balls with 10 (50%) and 3 (50%) mm in diameter and a ball-to-powder ratio of 10:1 under argon atmosphere were used as the milling media. After the milling process, different processes were carried out to determine the best parameters, as shown in Table 1.

Table 1: Samples and its parameters of fabrication

Samples	Powder Metallurgy				
	Pressing		Sintering	Additional steps	
	Uniaxial	Cold Isostatic		Forging	Annealing
1	1 ton/ 300 s	-	2 h	-	-
2	1 ton/300 s + PVA	-	2 h	1050 °C	-
3	1 ton/300 s	15 ton/90 s	2 h	1050 °C	-
4	1 ton/300 s	15 ton/90 s	2 h	1200 °C	1100 °C/1 h

Cold uniaxial pressing was performed in a circular die with a diameter of 30 mm. A load of approximately 1 ton was applied for 300 s on all samples. In sample 2, polyvinyl alcohol (PVA) was applied as a lubricant to facilitate the pressing which used 15% by weight of a 2.5% solution of PVA. In samples 3 to 6, the use of two consecutive pressing methods was studied. For cold isostatic pressing, samples were placed in a flexible mold under a vacuum for the process. Ethyl alcohol was used as a pressurizing fluid in a Marcon MPM-30 hydraulic press. After the pressing processes, the samples were sintered in an Ar atmosphere at 1100 °C with a heating rate of 10 °C/min and cooled to room temperature in the furnace. In sample 3, before sintering, a pre-sintering at 300 °C for 30 minutes was done to eliminate lubricant.

Finally, after sintering, additional steps were applied to the study. The hot forging was done using a Nowak hydraulic press, model PM45TON, and an INTI oven. The sintered samples were transferred to a furnace at 1050 °C (samples 2, and 3) or 1200 °C (samples 4) for 5 minutes. Then, the hot samples were immediately transferred to the press and forged. This process was repeated by three times until reaching half of the initial thickness. In addition, some samples were also annealed in an Ar atmosphere at 1100 °C for 1 hour at a heating rate of 10 °C/min and the furnace cooled to room temperature. An INTI FT-1200 tubular oven was used for the sintering and annealing process.

The study of porosity and characterization of the materials obtained were carried out using scanning electron microscopy, and dilatometric tests. After heat treatments (sintering, forging, and annealing), samples were mechanically ground and polished with a silicon colloid suspension. Scanning electron microscopy (SEM - Vega3, Tesca) was used to evaluate the microstructure. The porosity area fraction was calculated using an image analysis tool (ImageJ). The dilatometric tests were performed under vacuum in the sintered and annealed conditions using Netzsch DIL 402C dilatometer. The samples were subjected to a constant heating and cooling rate of 10 °C/min up to 1200 °C/5 min.

RESULTS AND DISCUSSION

Initially, the use of lubricant was studied. According to YU *et al.*[12], the use of lubricant allows a greater union between the particles of the metallic powders, facilitating the sintering process and reducing the porosity of the material. This statement is confirmed by analyzing the images obtained by SEM (Figure 1a e b). The sample with lubricant (Sample 2) showed a smaller porosity area fraction, 4.6 % when compared to the sample without lubricant (Sample 1), 14.7%. However, it is noted that the lubricant was not completely eliminated during the pre-sintering step when performing an additional step to improve the densification. As shown in Figure 1c, there is a variation in composition between the grain (light gray) and boundary (dark gray) regions. During the hot forging, the retained lubricant in the microstructure agglomerated, mainly between the grains due to the reduction of voids. Therefore, the use of the lubricant was discarded, since obtaining a heterogeneous microstructure can impair the final properties of the alloy.

The application of a second pressing method also can help to reduce the pores without contamination. Cold isostatic pressing was carried out after cold uniaxial pressing (Sample 3). In addition to not generating any unwanted impurities, the cold isostatic pressing applies the load evenly throughout the sample, reducing neutral zone effects from uniaxial pressing[13]. As shown in Figure 1d, sample 3 showed a significant reduction in pores compared to sample 1, indicating that a second pressing method helps to increase the densification of the material. However, it is still possible to observe pores and remaining boundaries. In this sense, an additional step, hot forging, was carried out after sintering.

Hot forging is an effective way to reduce residual pores after sintering and obtain fully dense materials[10]. In their studies, Alshammari *et al.*[10] showed that the application of the hot forging process after sintering generates samples with a density above 99.5 %. Analyzing sample 3 after the hot forging, there is a porosity reduction from 5.7 to 2 %, but it is still possible to visualize grain boundaries from the powders (Figure 1e). These boundaries can not be accounted for using the ImageJ analysis tool, inducing a porosity smaller than the real. Thus, a forth sample was prepared, in which the forging temperature was increased. According to Tang and Chang[14], higher temperatures increase the rate of atomic diffusion by thermal activation, generating greater filling of voids and reduction of remaining boundaries. In addition, another heat treatment, annealing, was applied to reduce the stresses from the forging and also improve the densification of the material.

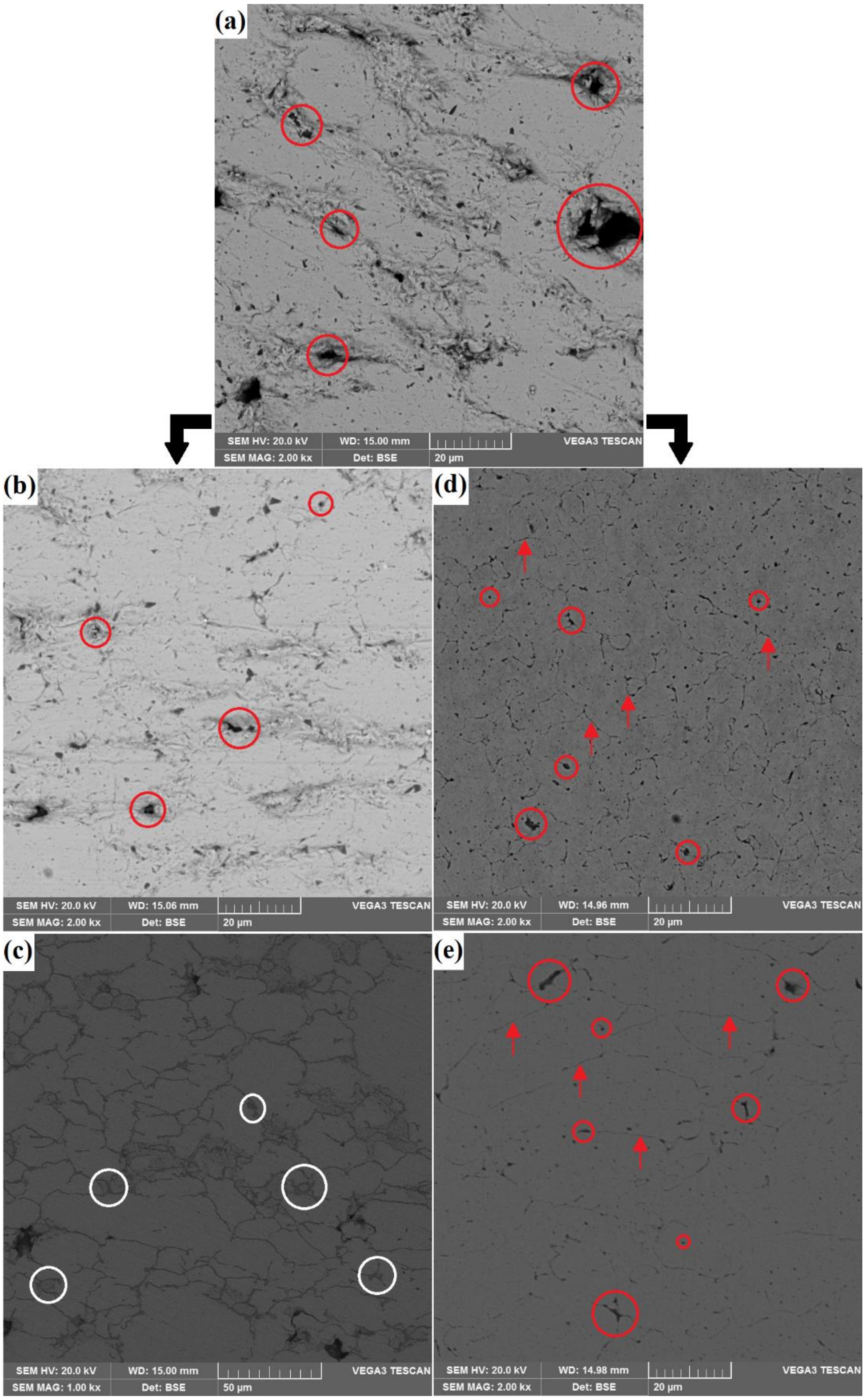

Figure 1: Images SEM of uniaxially pressing samples (a) without and (b) with PVA, (c) with PVA after hot forging, (d) with cold isostatic pressing, and (e) with cold isostatic pressing after hot forging. Red and white circles indicate pores and lubricant residues, respectively, and red arrows indicate remaining boundaries.

As shown in Figure 2, the hot forging and annealing steps reduced the residual boundaries and pores present in the microstructure after sintering. This observation corroborates the estimated porosity of 5.7, 5.3 and 3.7 % for sintered, forged and annealed sample 4, respectively. In addition, the annealed sample presents rounded pores, evenly distributed throughout the microstructure. According to Alshammari *et al.*[10], the presence of rounded pores indicates that the samples reached the last stage of sintering.

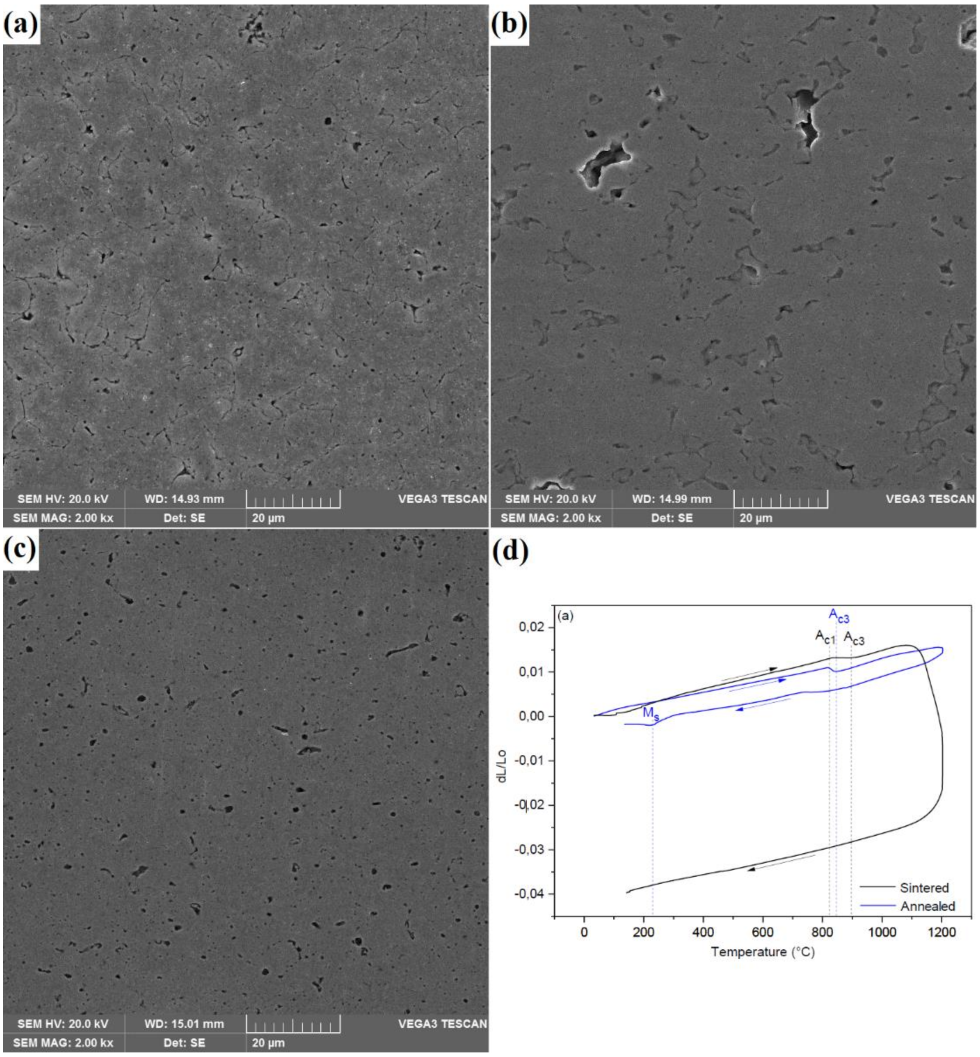

Figure 2: Images SEM of sample 4 (a) sintered, (b) forged, (c) annealed, and its (d) dilatometric curve.

The porosity reduction can also be observed when analyzing the dilatometry curves of the sintered, and forged and annealed sample 4 (Figure 2d). The high shrinkage in the sintered sample masked the peaks of phase transformations and/or formation of intermetallic phases. It is possible to identify only a small variation referring to the austenitic transformation in which the beginning and end are represented by the values of Ac1 (830 °C) and Ac3 (900 °C), in black. With the decrease in shrinkage the in annealed sample, the peak of austenitic transformation became more evident, with the values of black Ac1 and blue Ac3 equal to 830 and 845 °C, respectively. It is also possible to observe in the annealed samples the peak referring to the beginning of the formation of martensite (Ms), 225 °C. The smaller shrinkage presented by the annealed sample can be explained by a smaller amount of pores. So, these results indicate that the additional steps produce an effective pores reduction as well as the performance of two consecutive pressing methods.

CONCLUSIONS

The application of two pressing methods in samples 2 e 3 presented a significant reduction of porosity when compared the sample 1. In addition, it is more advantageous than the application of lubricant that can generate unwanted contamination as presented in sample 2. Finally, the application of additional steps causes an improvement in densification, mainly when using a higher temperature in the forging followed by the annealed step.

AKNOWLEDGEMENTS

The authors acknowledge the FAPEMIG for the financial support.

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