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Rem: Revista Escola de Minas, vol. 68, núm. 3, julio-septiembre, 2015, pp. 295-300
Universidade Federal de Ouro Preto
Ouro Preto, Brasil

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http://dx.doi.org/10.1590/0370-44672014680189

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Abstract

The AISI 52100 is a tool-type steel and is more often used in industry for the production of bearings. After the end of its life cycle, it is discarded or remelted, but both processes are considered expensive. Thus, the possibility of reusing this material through the powder metallurgy (PM) route is considered advantageous, since it transforms a waste into another product. To obtain the starting powders, the AISI 52100 steel scrap was submitted to a process of high energy ball milling, which was milled pure and with 1 and 3 % of niobium carbide (NbC) additions. Those additions were performed with the intention of increasing the milling efficiency of the steel, through formation of a metal-ceramic composite with a ductile-fragile behaviour. To determine the morphology and particle size, scanning electron microscopy (SEM) and particle size distribution tests were used. The results indicated that with the carbide addition, a significant increase in the milling efficiency was achieved, being possible to obtain nanoparticles after 20 hours of milling time.

Keywords: AISI 52100 steel; high energy ball milling; carbide; nanoparticle.

1. Introduction

The AISI 52100 is a high carbon, chrome, and manganese steel which finds applications in bearings due to its high strength and resistance to rolling contact fatigue (Li & Wang, 1993; Umbrello *et al*., 2004). In their service life, bearings are subjected to complex multi-axial stress states that change periodically in combination with high operational temperatures (Chakraborty *et al*., 2009; Young & Badeshia, 2004). They must sustain relatively large contact stresses during the extremely high number of cycles that are submitted (Dommarco *et al*., 2004; Christ *et al*., 1992). One of the methods to increase the resistance of bearings produced by AISI 52100 steel is through powder metallurgy (Rhee *et al*., 2007).

Powder metallurgy is a promising route widely used to produce high-strength steels for the fabrication of pieces with complex shapes with less material waste (German, 1998; Esper & Sonsino, 1996). These steels show several important properties, such as high mechanical strength and wear resistance (Selvakumar *et al.*, 2012; Lane & Smith, 1982). However, they usually possess residual porosity and a heterogeneous microstructure (Narasimhan, 1996). The porosity has been shown to significantly influence the fatigue response of steels (Hadrboletz & Weiss, 1997).

To improve the mechanical properties of steels produced by powder metallurgy, the creation of a metal-ceramic composite with carbide addition as reinforcement is advantageous (Trueman *et al.*, 1999). The concentration of this reinforcement in such composites is typically less than 50 % in volume (Lu *et al.*, 2012; Eigen *et al.*, 2003; Yusop *et al.*, 2009).

Carbides are compounds formed by carbon and metal atoms. The metal carbides present important properties, such as a very high melting point (close to 4000^{0}c), great hardness, and good electric and thermal conductivity (Gubernat & Zych, 2014; Kosolapova, 1971). The niobium carbide (NbC) presents interesting characteristics for its use in wear applications, such as great hardness, great toughness, extremely high young's modulus and high melting temperature (3873^{0}c) (Amriou *et al.*, 2003; Sustarsic *et al.*, 2001).

In powder metallurgy, the powders pass through the steps of milling, cold pressing and sintering. Using high energy ball milling, it's possible to produce nanoparticles, which consequently, increase the densification, decrease the porosity in sintered materials and improve the mechanical resistance of the product (Wang & Jiang, 2007). The high energy ball milling utilizes high frequency and high energy impacts from the milling balls to repeatedly forge powder particles together, which causes a greater reduction in the particle's size in comparison with traditional milling (Lu & Lai, 1998; Suryanarayana, 2001).

2. Materials and Methods

The material used in this research was the AISI 52100 steel from the process of hot rolling as a round bar, the same material used to produce bearings. This workpiece had 100 mm of diameter and passed through the step of machining at slow speed and without the use of lubrication to avoid oxygen and oil-soluble contamination. With the procedure described, it was possible to obtain the AISI 52100 steel in the form of scraps, which were used subsequently in the milling process.

The scraps were milled pure and with 1% and 3% of NbC addition (Table 1). The milling was realized using high energy ball milling in a planetary ball mill during the milling times of 5, 10, 15 and 20 hours in an inert argon atmosphere to avoid oxidation of the powders, at a milling speed of 350 rpm and a mass/sphere relationship of 1:10.

Composition	Material	AISI 52100 weight (g)	NbC weight(g)
1	AISI 52100	30	0
2	AISI 52100 + 1% NbC	29.7	0.3
3	AISI 52100 + 3% NbC	29.1	0.9

Table 1
Mixtures composition

The characterization of the raw materials and their compositions was realized using a scanning electron microscope Carl Zeiss EVO MA15. In the secondary electron (SE) mode, the particle size variation and morphology were analyzed. Using the back scatter electron (BSD) and energy dispersive x-ray (EDS) modes, the carbide distribution was evaluated. Particle size distribution was performed in a Malvern Mastersizer 2000 particle size analyzer.

3. Results and Discussion

The initial characterization of the AISI 52100 steel scraps and the niobium carbide are shown in Figures 1 and 2. It can be observed that the AISI 52100 steel scraps from the machining process showed a mean size of 1200 μm (Figure 1).

Figure 2 shows the microphotograph of NbC. In this figure, it is possible to note that the particles presented heterogeneous granulometry varying from 1 to 5 μm.

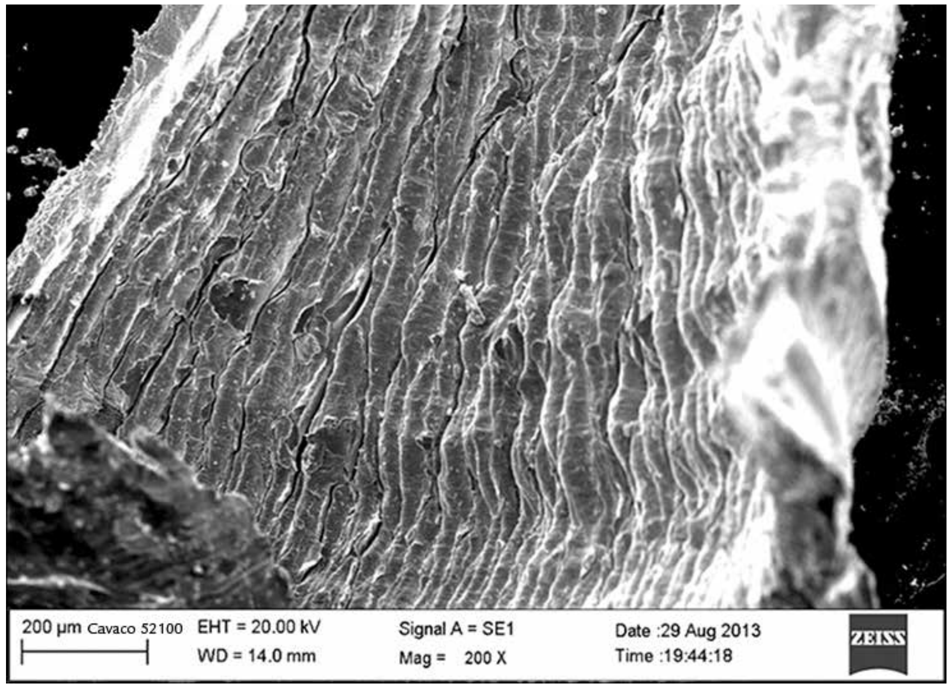

Figure 1
AISI 52100 steel scrap

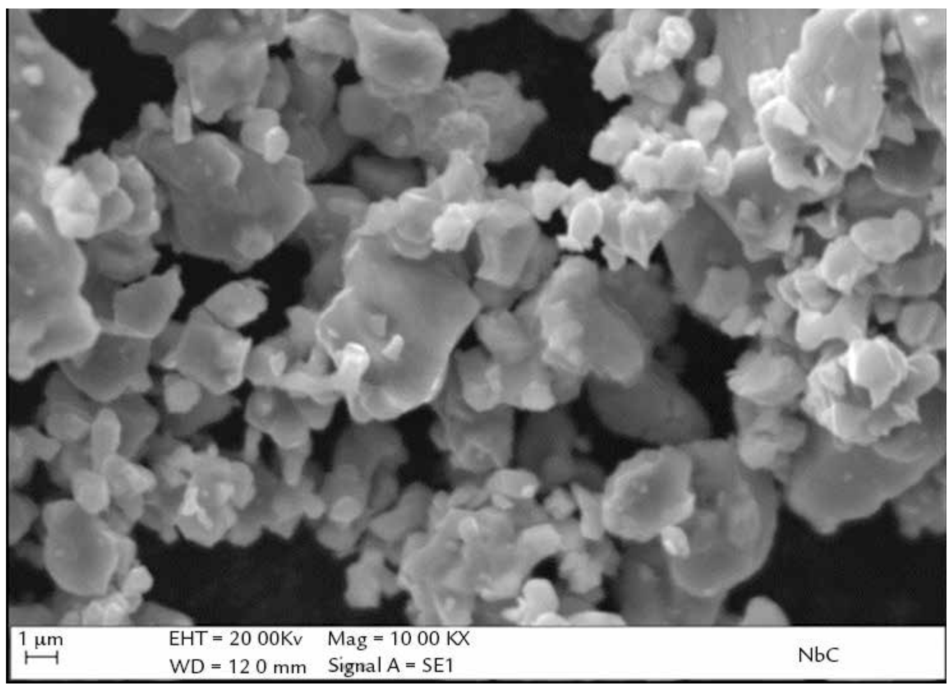

Figure 2
NbC powder

It can be observed in Figure 3a that after 5 hours of milling, the scrap of the pure AISI 52100 steel maintained its morphology and dimension in comparison with the scrap before the milling process (Figure 1). By increasing the milling time to 20 hours, the scraps of the AISI 52100 steel were transformed into particles with angular morphology and medium-sized particles located between 5 and 40 µm as shown in Figure 3b.

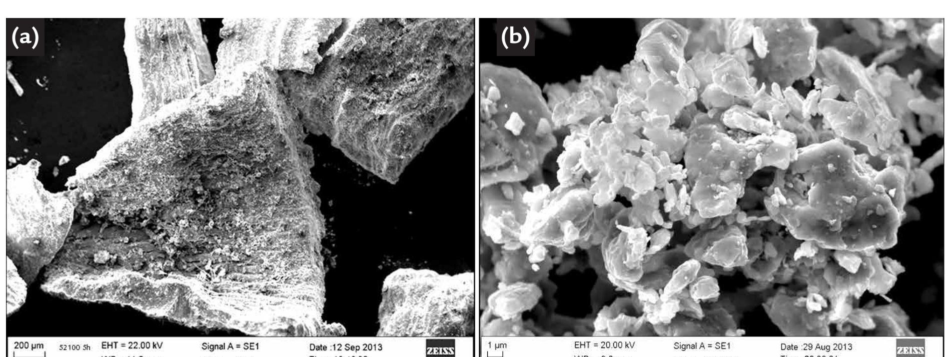

Figure 3
Pure AISI 52100 steel milled for
(a) 5 hours (b) 20 hours

By performing the milling of the AISI 52100 steel scraps for 5 hours and with a 1% of NbC addition, it was possible to obtain particles with a flaky morphology and sizes between 20 and 200 µm (Figure 4a), however, with a large volume of smaller particles. When maintaining the NbC percentage at 1% and continuing the milling of the AISI 52100 steel for 20 hours, it was possible to observe the formation of clusters with a dimension around 20 µm and the presence of nanoparticles (Figure 4b). Thus, comparing the milling process of the pure AISI 52100 steel with a 1% of NbC addition, under the same milling condition, a significant reduction in the obtained particles was observed, demonstrating an increase in the milling process efficiency.

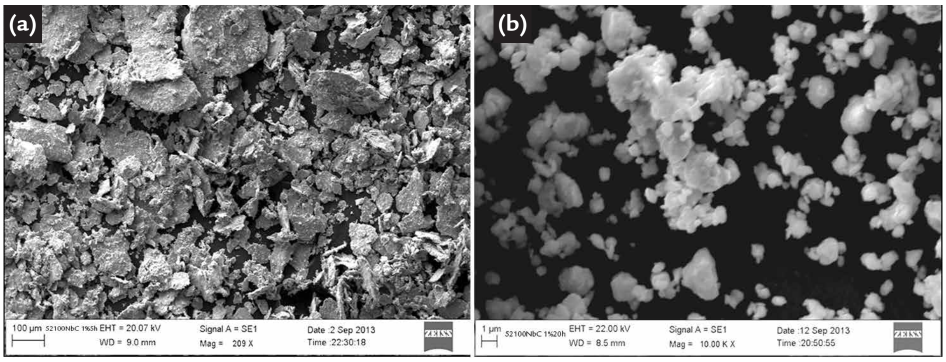

Figure 4
AISI 52100 steel with 1% of NbC addition milled for (a) 5 hours (b) 20 hours

When milling the AISI 52100 steel for 5 hours with a 3% NbC addition, it was observed that there was no change in the particle morphology. However, noted was a significant reduction in the particles size of the AISI 52100 steel in comparison with the milling process using 1% of NbC addition (Figure 4a). In this condition, the particles present sizes between 20 and 170 µm (Figure 5a). Maintaining the NbC percentage at 3% and milling during 20 hours, the AISI 52100 steel formed clusters with approximately 300 µm with medium-sized particles located in nanometric scale (Figure 5b). In addition, a greater homogeneity was observed in the nanoparticle volumes when compared to the steel milled with 1% of NbC.

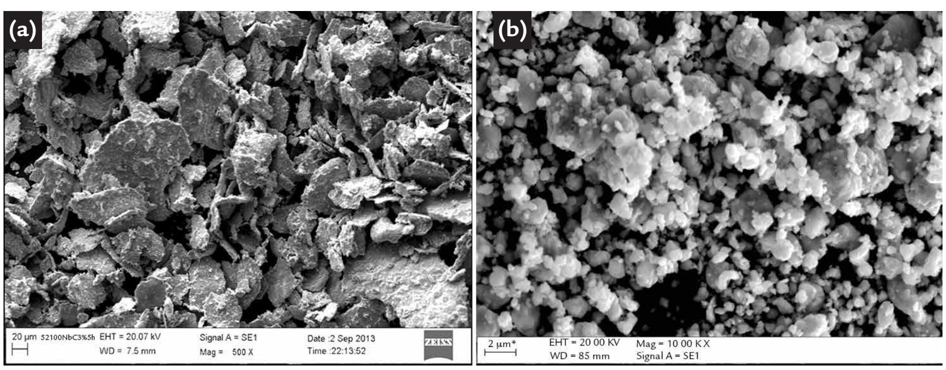

Figure 5
AISI 52100 steel with 3% of NbC addition milled for (a) 5 hours (b) 20 hours

The carbide distribution was evaluated under the scanning electron microscope (SEM) using the energy dispersive x-ray (EDS) mode (Figure 6). It was observed that the NbC particles were located homogeneously on the surface of the steel particles. The NbC particles were identified in Figure 6 by its chemical elements (C and Nb). In Figure 6a, the brighter dots represent the carbon element, and in Figure 6b, the brighter dots represent the niobium element.

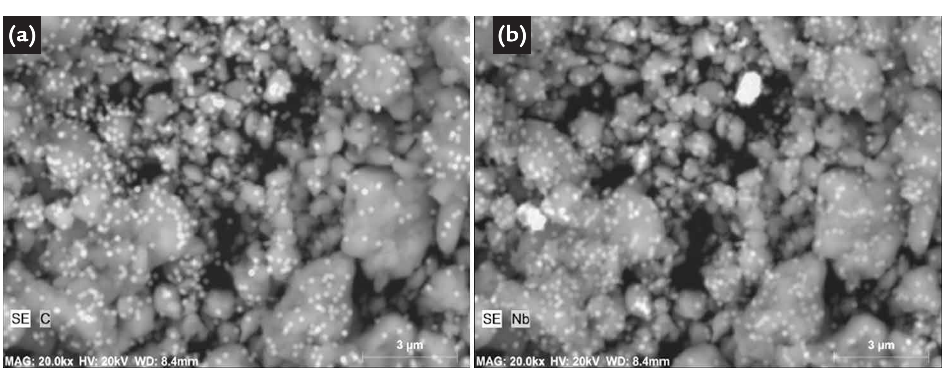

Figure 6
Identification of the NbC chemical elements (a) Carbon (b) Niobium

The results of the particle size distribution test are shown in Figure 7. It is possible to observe that the curve presented a bimodal distribution. When milled with the niobium carbide addition, the steel particles presented greater volume in the range of 1 nm up to 4 nm and that of 10 nm up to 40 nm. When milled without NbC addition, the steel particles presented a greater volume in the range of 2 nm up to 80 nm and that of 100 nm up to 200 nm, showing an increase in the milling efficiency with the carbide addition.

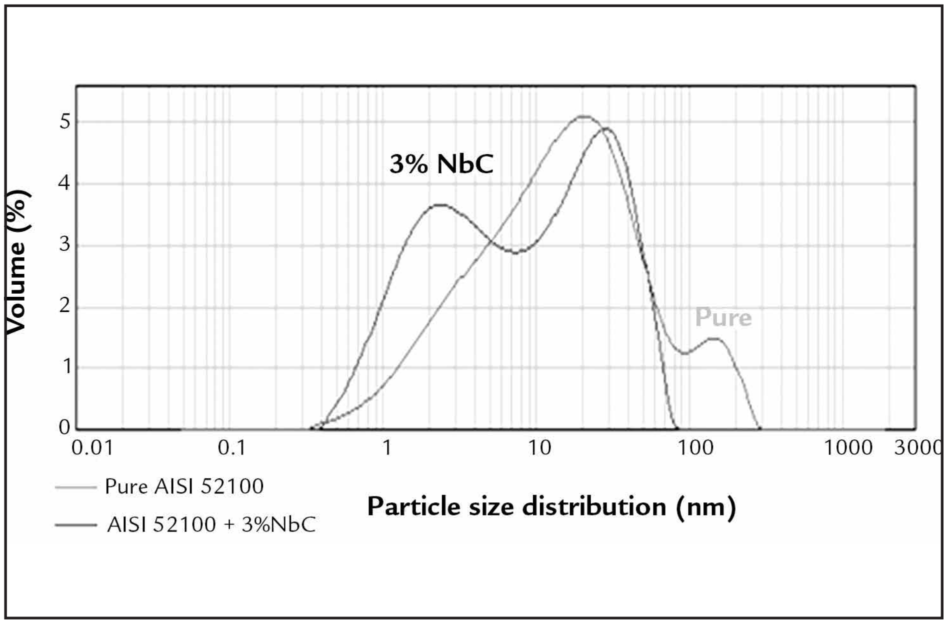

Figure 7
AISI 52100 steel particles size distribution without and with 3% of NbC addition

4. Conclusions

When using the high energy ball milling process, it was possible to obtain powders of the pure AISI 52100 steel and also those with NbC additions. However, with the NbC additions, it was observed that there was a significant increment in the milling efficiency, which enabled the obtainment of powders close to the nanometric scale.

5. Acknowledgements

This work was financially supported by CNPq and FAPEMIG. We would also like to thank the companies Villares Metals and Hermann C. Stark for the material donations and the UNIFEI characterization's laboratory technicians for the technical support.

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Received: 19 September 2014 - Accepted: 10 June 2015.