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Recycling carbon-steel waste from blast cleaning by powder metallurgy employing the augmented simplex lattice design

Reciclagem de resíduo de granalha de aço-carbono de jateamento por metalurgia do pó empregando o planejamento em rede simplex

Reciclaje de residuos de granalla de acero al carbono por granallado mediante pulvimetalurgia empleando el diseño simplex reticular aumentado

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Abstract
Blasting cleaning is widely used to prepare and clean surfaces, but the waste resultant is generally contaminated and recycling is an obstacle. The present study shows a technical route to reuse the carbon-steel shot blast waste to produce a solid material by powder metallurgy. The waste was selected by sieve and the particles mixtures were analyzed and selected with the aid of a simplex lattice experimental design and tap density. The characterization of waste particles was conducted in terms of their morphological shape and their technological and physical characteristics. The density of green compacts and sintered samples reached 7.11 g.cm^{-3} and 7.06 g.cm^{-3}, respectively. After quenching and tempering heat treatments, the sintered steel exhibited a martensitic microstructure with a hardness of 430 HV. The sintered bodies showed promising properties and the powder metallurgy method for recycling shot blast waste was considered viable.
Keywords: Powder metallurgy; Tap density; Blasting media waste.

Resumo
A limpeza por jateamento é amplamente utilizada para preparar e limpar superfícies; no entanto o resíduo resultante geralmente é contaminado, tornando a reciclagem um obstáculo. O presente estudo apresentou uma rota técnica para reutilizar o resíduo do jateamento de aço carbono para produzir um material sólido através da metalurgia do pó. O resíduo foi selecionado por peneiras e as partículas da mistura foram analisadas e selecionadas com o auxílio do método experimental de design de simplex aumentado e densidade a batida. A caracterização das partículas de resíduo foi conduzida em termos de seu formato morfológico e suas características tecnológicas e físicas. A densidade dos compactos verdes e amostras sinterizadas alcançaram 7.11 g.cm^{-3} e 7.06 g.cm^{-3} respectivamente. Após os tratamentos térmicos de têmpera e revenimento, o aço sinterizado exibiu uma microestrutura martensita e dureza de 430 HV. Os corpos sinterizados apresentaram propriedades promissoras e o método de metalurgia do pó para a reciclagem do resíduo de jateamento foi considerado viável.
Palavras-chave: Metalurgia do pó; Densidade batida; Resíduo de jateamento.

Resumen
La limpieza por granallado es ampliamente utilizada para preparar y limpiar superficies; con eso, el residuo resultante generalmente se contamina, haciendo con que el reciclaje sea un obstáculo. Esta investigación presentó una ruta técnica para reutilizar el residuo del granalla de acero al carbono para producir un material sólido a través de la pulvimetalurgia. El residuo fue seleccionado por tamices y las partículas de la mistura analizadas y seleccionadas con el auxilio del método experimental de diseño de simplex aumentado y densidad compactada. La caracterización de las partículas de residuo fue conducida por su formato morfológico y sus características tecnológicas y físicas. La densidad de los compactos verdes y muestras sinterizadas alcanzaron 7.11 g.cm^{-3} y 7.06 g.cm^{-3} respectivamente. Después de los

tratamientos térmicos de templado y revenido, el acero sinterizado mostró una microestructura martensita y dureza de 430 HV. Los cuerpos sinterizados presentaron propiedades promisoras y el método de pulvimetalurgia para el reciclaje de residuo de granallado fue considerado viable.

Palabras clave: Pulvimetalurgia; Densidad compactada; Residuo de granallado.

1. Introduction

Blast cleaning is an industrial process for surface preparation and surface improvement of a diverse cast, forged, or stamped components. This process propels the blasting media by pressurized air or by centrifugal force in a proper wheel to the surface to be prepared. This long-standing technique is widely used for derusting parts, improving the surface visual appearance, surface cleaning and surface texturing before painting or coating. Blast cleaning is also known as shot blasting, grit blasting, or sandblasting and is one of the most used surface treatments in modern industry. During the blast cleaning process, the abrasive particles called blasting media or abrasive agents are propelled against the surface to be cleaned or treated. Different abrasive materials are used as blasting media, which can be metallic (steel, iron, copper, nickel, slags from those, etc.) or non-metallic (hematite, silica sand, olivine, etc.). Nowadays, steel abrasives are between the most consumed blasting media (Shivpuri et al., 2009); (Means et al., 1991); (Momber, 2008).

The impact of particles against the target surface and the repeated inter-particle collision process causes severe mechanical stress and fragmentation of the abrasive media. The reduction of the particle size caused by fracture unfavorably affects the efficiency of the blast cleaning operation and the reusability of the blasting media (Calboreanu, 1991). The spent abrasive particles, which are smaller in size, have less inertial energy to produce erosion work and are removed from the blast cleaning process by the separation system of the machine. This waste is, in general, disposed of in industrial landfills (Katsikaris et al., 2002) (Prasad & Ramana, 2016) (Buruiana et al., 2013).

However, the blasting waste disposal can result in pollution of industrial sites and, due to the limited availability of landfill areas, the waste management costs have increased. Furthermore, the waste produced by blast cleaning process incorporates a mixture of small blasting media and impurities particles. Those impurities, that come from the component cleaning in varied contamination levels, make the recycling process onerous and often a long-labored procedure, concerning the removal of contaminants from the blasting media for subsequent processing (J. Z. Gronostajski et al., 1998) (Kjeldsteen, 1982) (Kadir et al., 2017) (Simon et al., 2017) (Madany et al., 1991).

There are various researches concerning the development of recycling procedures for blasting waste. Most of them are not economical nor practical to replicate on an industrial scale, besides showing low selling value. One of the solutions is reutilizing the spent blasting media by returning it to the equipment after at least one blast cycle, using a special additive to bind in, which forms new larger particles, each containing the additive and media. However, this recycling route uses patented products which may increase the cost of the process (R Lynn & Paul Parent, 1996). Semi-industrial recycling units of used metallic slags blasting media have been built, nevertheless, its main concern is to recover the lost abrasive of an open dry blast cleaning instead of recycling it. The process involves sorting the particle size by sieving the spent blasting media and blending it with a new abrasive agent (Katsikaris et al., 2002).

Other attempts have been conducted to develop recycling procedures using metallic slag waste as aggregate in the concrete (Allen & Iano, 2008) or asphalt (Means et al., 1991) (Madany et al., 1991) (Buruiana et al., 2013) (Prasad & Ramana, 2016), but the impurities mixed on the blasting waste is a dilemma. Another technical route to recycle the blasting media waste is using binders to form briquettes. After appropriate treatment, the briquette is handled as scrap in a foundry (Peccin Martins, 2010). A disadvantage of this process is that the binder releases gases that can pose environmental and molten metal problems during the melting process. Additionally, during the melting process of the recycled briquettes, a large amount of metal is lost because of the inherent oxidation of the raw material.

There are other recycling demands in ferrous and non-ferrous industrial making processes such as recycling chips from the machining process. This problem is substantially similar to that of the blasting media recycling, which has been alternatively handled through powder metallurgy (PM) methods. In the case of machining operations, chips of iron, aluminum, bronze and possibly other materials can be mixed with commercial powders in various amounts and subsequently processed by powder metallurgy to produce a finished product (J. Z. Gronostajski et al., 1998) (Afshari & Ghambari, 2016) (J. Gronostajski et al., 2002). This alternative process overcomes the disadvantages arising from the conventional remelting procedures carried out by casting operations that, in some cases, almost half of metal is lost due to its oxidation, slag mixing and scraps (J. Gronostajski et al., 2002).

The powder metallurgy processing involves a powder mixing stage, followed by the compaction stage, in which the powders are pressed into a pre-form cavity and subsequent sintering, which involves heating to a temperature below the melting point of the major constituent to create a coherent object (Thümmler & Oberacker, 1993). The sintering process is a thermal treatment to bond the particles, providing mechanical strength to the body. Throughout the heating, atomic motion accelerates and grows the bonds between particles contacts (German, n.d.). PM processing can offer some advantages over other metalworking practices, including improved homogeneity of microstructure, good material utilization, lower slag content, ability to make complex compositions, as well as improved control of composition and micro-porosity. When combined with powder selection criteria, these advantages can also be taken as a feasible solution to reprocess blasting media waste into materials or products for other purposes.

This work proposes a method of recycling carbon steel blasting media waste by powder metallurgy with the aid of design of mixture experiments. The simplex lattice designs comprise a group of designs of mixture experiments that are useful in designing a wide variety of products, through studying the effects of mixture components on the response variable. Mixture experiments are a special type of the response surface methodology statistical technique, which can be used in place of the one-factor-at-a-time experimental strategy to optimize unfamiliar and non-commercial powders. Although frequently run in practice, the one-factor-at-a-time approach is time-consuming and fails to reach the true optimum response because it ignores the interactions between the factors (Hinkelmann & Montgomery, 2012). For that reason, a simplex lattice design was used in this work, to optimize the packing density of mixtures of blasting media waste concerning the size, morphology and density of the powder to be recycled.

Based on the packing density concept (German, 1992), it is hypothesized that the density of the sintered bodies is linked to the packing density of powder mixtures, which contains the blasting media waste with a multimodal particle size distribution. In this context, tap density measurement was used as the response variable of the augmented simplex lattice design to select the blasting media waste powder mixture with optimized technological properties. The tap density method specifies the density of the powders taped in a container according to their volume, mass and number of taps. Thus, the proposed study for recycling blasting media waste shows that producing sintered compacts with appropriate density and mechanical properties is possible.

2. Methodology

2.1 Processing and characterization of the blasting media waste

Blasting media waste was collected according to ASTM B215-15 standard, followed by a primary separation by a sieve to remove coarse particles bigger than 300 μm, which were not considered in this research. A small sample was separated, cleaned in acetone and dried, to be analyzed by field emission scanning electron microscopy (FESEM) and energy dispersive X-ray spectroscopy (EDS). The remaining particles were used as received in further procedures.

The particle size distribution was obtained using the ASTM E11-04 standard sieves system that was mounted on a mechanical vibrator and agitated for 15 min. The characterization of particle shape was conducted by image analysis and the projected 2D silhouette of particles was considered for this purpose. In total, 117 images were obtained with a resolution of 768 x 960 pixels each, resulting in 6,714 particles processed in the free ImageJ software. Equations (1), (2), and (3) allowed to estimate the circularity index (Cir), aspect ratio (AR) and roundness index (Rou) of the blasting media waste particles (Rodriguez, J.M., Johansson, J.M.A., Edeskär, 2008).

Circularity $$(\text{Cir}) = 4\pi \frac{area}{(perimeter)^2} \tag{1}$$

Aspect Ratio $$(\text{AR}) = \frac{Major\ axis}{Minor\ axis} \tag{2}$$

Roundness $$(\text{Rou}) = 4 \frac{area}{\pi\ (Major\ axis)^2} \tag{3}$$

The specific gravity of the particles was measured in a water pycnometer using 5 mL of distillate water, according to ASTM D854-02 standard.

2.2 Technological properties of the blasting media waste

Besides the physical and chemical characteristics, the technological characteristics of the individual powder and powder mixtures with different particle sizes were evaluated. The apparent density was measured according to the Arnold test described by the ASTM B703-10 standard. This method consists in collecting a small volume of powder, by sliding a cylindrical sleeve containing the powder over a cavity in a die block. The mass of the powder and the known volume are calculated and the apparent density is measured.

The flow rate properties of the powder were evaluated according to ASTM B213-13 standard using a Hall flowmeter funnel that allowed assess the time to flow 50 g of the sample powder.

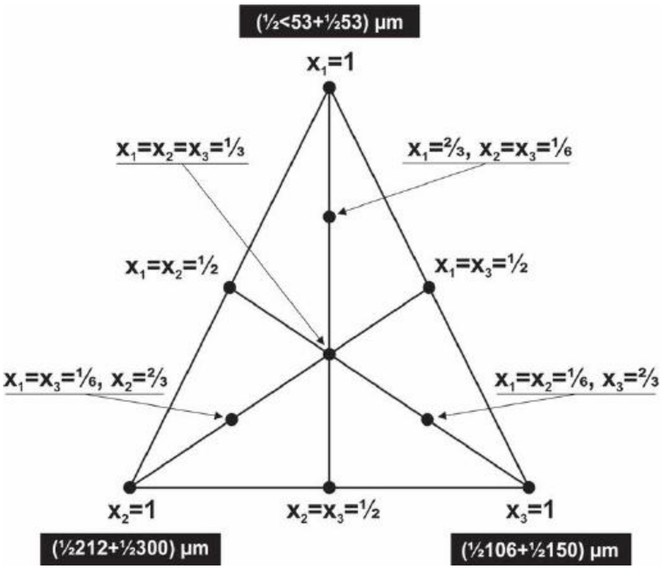
2.3 Design of mixture experiments and powder mixtures selection

The close-packing of a spherical and monomodal particle distribution is known to achieve a maximum of 74% of the total space volume filled. The voids have to be filled with smaller particles to increase the density. Andreassen's packing model considers a continuous distribution of particles sizes filling the voids. Other models as Furna and Alfred follow a similar theory (Tu et al., 2015) (Yousuf et al., 2019).

The blasting media waste has a particle shape between spherical, slightly irregular and elongated in various sizes. Irregular particle shapes are challenging to reach high densities and their shape interferes negatively with packing, increasing the voids. Therefore, this study proposes that the green body obtained in the powder metallurgy process will acquire higher density using particle size selection and tap density methods.

The augmented simplex lattice design was used to formulate powder mixtures with a wide particle size range. The triangle shape in a trilinear coordinate system is shown in Figure 1, which represents the simplex lattice design. Each vertice represents a pre-mixed blasting media waste powder with fine, medium and coarse particles size (Tauxe et al., 1988). The X1 vertice represents a mixture of a 50% < 53 µm and 50% 53 µm particles size measured in mass, X2, a mixture of a 50% 106 µm and 50% 150 µm, and, X3, a mixture of a 50% 212 µm and 50% 300 µm. All particle sizes were separated by sieving. Overall, ten mixtures were tested, four of them represent the inner points of the augmented simplex lattice design and one represents the centroid.

Figure 1: Augmented simplex lattice design



Source: Montgomery (2017).

The tap density experiments were conducted following the ASTM B527-15 standard using a graduated cylinder container with a volume of 25 cm3 mounted in the tap density equipment. The apparent tap density was calculated with the known mass and measured volume in steps of 0, 10, 100, 500, 1000, 2000, 5000 and 10000 taps.

2.4 Powder metallurgy – samples processing and characterization

The powders mixtures that exhibited the highest apparent density from tap density tests were, subsequently, processed by powder metallurgy to obtain consolidated bodies. The compaction step was conducted at a pressure of 1000 MPa in a uniaxial die. The green compacts had a final size of about 10 mm in diameter and 3 mm in thickness. Afterward, the sintering step was conducted at 1165ºC in an argon atmosphere for 60 min. Some samples were quenched at 800ºC in oil and then tempered at 500ºC for 2 h.

The density of the sintered bodies was measured by the Archimedes method in distillate water. The porosity was evaluated by image analysis with the aid of the software ImageJ. The Vickers hardness of the blasting media waste particles and the sintered bodies were measured using a micro-hardness tester (SHIMADZU, model HMV-2T).

In order to characterize the microstructure, the blasting media waste and the sintered bodies were ground and polished. For the metallography evaluation, the samples were etched by immersion in Nital 0.5% for 10 to 30 s and evaluated by FEGSEM.

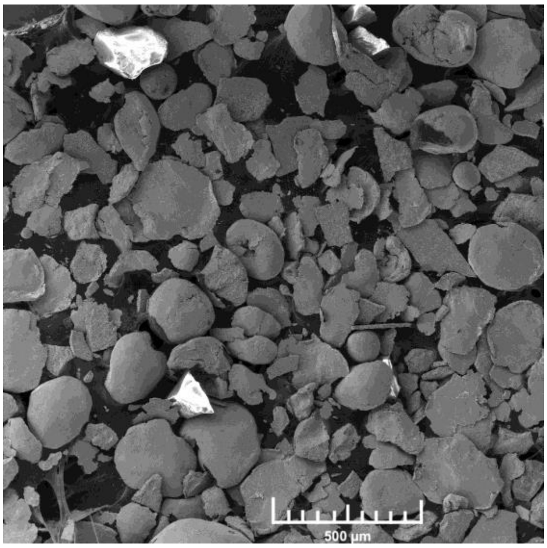
3. Results and Discussion

3.1 Particles characterization

The particle size distribution of the blasting media waste showed a Gaussian distribution with an unproportioned cumulative 80 µm size. The mean size was 90.4 µm, the D10 was 50.4 µm, D50 84.2 µm and the D90 192.4 µm.

The particle morphology showed a circularity number of 0.76, an aspect ratio of 1.53 and 0.89 for roundness. The blasting media waste exhibited a spherical shape for the bigger particles and an irregular shape for the smaller particles, as shown in Figure 2. The irregularity and non-circularity of the small particles are attributed to the fragmentation of the blasting media during the blast cleaning process and the contamination particles mixed with the abrasive media waste. Small quantities of aluminum, titanium and calcium were found in EDS analysis. Those elements are usually found in automotive inks. The source of the impurities was recognized to be from the removed paint from trucks frames that are occasionally cleaned with the blast cleaning process and carried to the blasting media waste mixture.

Figure 2: Morphology of the abrasive media.



Source: Authors.

The Arnold density showed a density of 2.36 g.cm-3 for the < 53 µm particle and, with the increase in the particle size, the density increased likewise, achieving 4.08 g.m-3 for the 300 µm particle. The pycnometer density of the abrasive waste exhibited a similar pattern. The particle size of the contaminant was smaller than those of the blasting media waste, and their densities are known to be lower than the density of the carbon steel. This may be the main reason for the variation in density values as a function of particle size, as shown in **Erro! Fonte de referência não encontrada.**. In addition, the morphology of the blasting waste particle had a substantial influence on the Arnold analysis, indicating a low packing density for the irregular particles that are smaller in size (Merkus, 2009).

Table 1: Pycnometer density and apparent Arnold density.

Size (µm)	**< 53**	**53**	**106**	**150**	**212**	**300**
Arnold apparent density (g.cm^{-3})	2.361	2.360	2.678	3.426	3.843	4.082
Pycnometer density (g.cm^{-3})	5.405	6.288	6.520	6.950	7.199	7.359

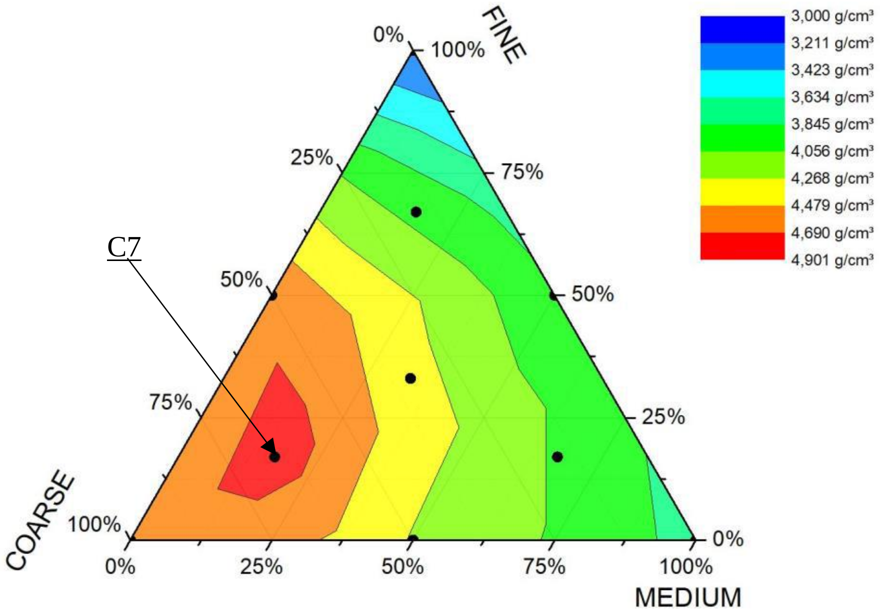
Source: Authors.

The flow rate of the < 53 µm blasting media waste was null, which can be attributed to the small size and irregular morphology of its particles, making them difficult to flow through the funnel Hall meter. The increase in particle size reduced the flow time and further improved the flow. The flow time for particles sizes of 53 µm was 37.1 s, and for 300 µm was 24.6 s for 50 g. These flow times were similar to those of commercial powders.

3.2 Augmented simplex lattice design and tap density response

Figure 3 shows the plot of the surface response of tap density response for all powders mixtures obtained according to the augmented simplex lattice design shown in **Erro! Fonte de referência não encontrada.**. The mixture named C7 formulated with 1/6 of fine + 1/6 of medium + 2/3 of coarse particle size showed the highest tap density among the analyzed samples. The C7 sample was thus exclusively selected to proceed with the further analyses because of its highest tap density and superior packing.

Figure 3: Surface response of the tap density response from the augmented simplex lattice design.



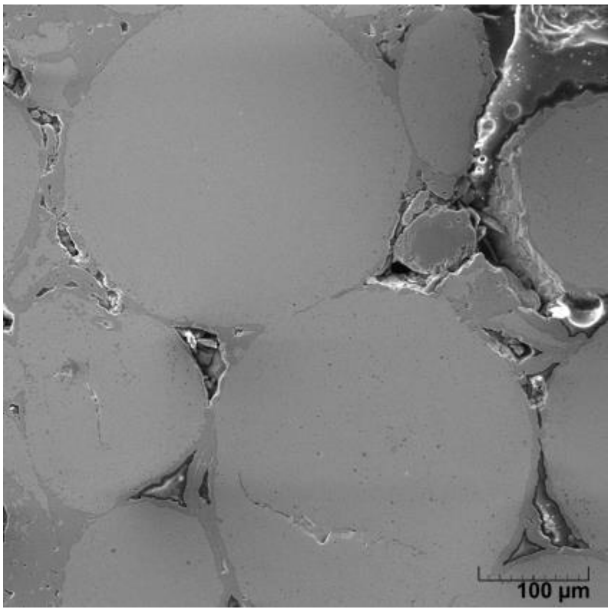
Source: Authors.

3.3 Powder metallurgy samples – density and microstructure

The Archimedes density of the green compact and sintered sample obtained from powder mixture C7 were 7,11 $g.cm^{-3}$ and 7,06 $g.cm^{-3,}$ respectively. The porosity in sintered components is an intrinsic feature from the powder metallurgy process, sometimes desirable but mostly controlled for quality purpose. The sintered sample had a porosity of 11%.

Figure 4 is a FESEM micrograph of the sintered sample, showing pores and necks growth between spherical particles. The two spherical particles connected are related to the first stage of the sintering thermal process. The diffusion over the particle surface and along the interface between particles promotes the growth of these connections. A longer sintering time the sample remains in the furnace should be sufficient for pores rounding and sample shrinkage. Still, the blasting media waste is covered by an oxide layer that slows the diffusion process (Wendel et al., 2020).

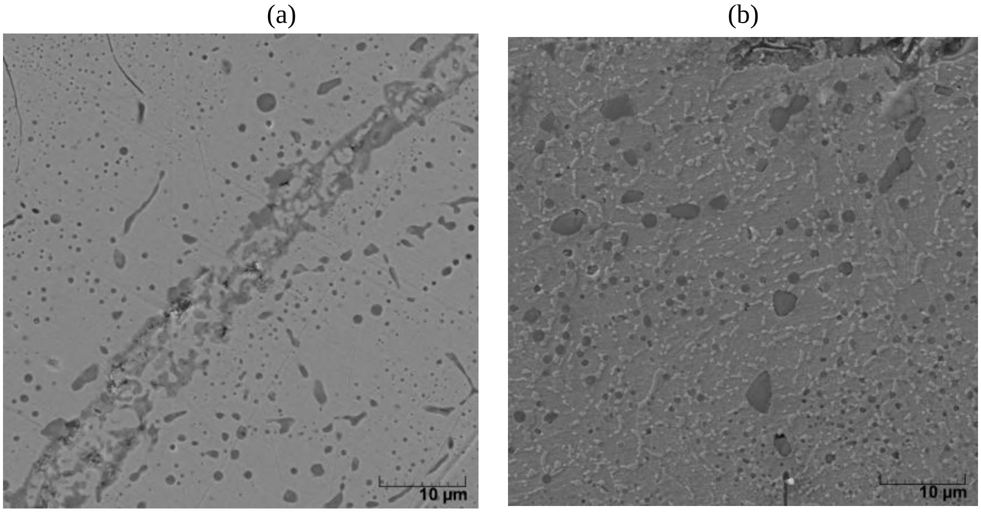
Figure 4: Porosity and neck formation.



Source: Authors.

The microstructure of the sintered sample appeared to be globular cementite in a ferrite matrix, as shown in **Erro! Fonte de referência não encontrada.**(a). The quenched and tempered sintered sample exhibited a fine martensitic microstructure with undissolved carbides, as shown in **Erro! Fonte de referência não encontrada.**(b). The hardness value of the sintered sample was 153 HV, and for the quenched and tempered sintered sample, the hardness was 416 HV due to its recognized thermal process that increases the hardness in some carbon steels.

Figure 5: FESEM micrographs of the microstructure of the sintered samples. (a) Sintered. (b) Quenched and tempered.



Source: Authors.

4. Conclusion

In this work, the simplex lattice design and tap density method were used to produce sintered bodies by powder metallurgy from blasting media waste. The main contribution of the study is a method that allows selecting a powder mixture with the highest density from a non-commercial powder, applying the packing density and mixture experiments design approaches. The blasting media waste and the sintered samples were characterized. The following conclusions were derived from the results of this work:

- The sintered body achieves almost 90% of the theoretical density using the presented method.
- Sintering time for the compacted samples was not sufficient to start the shrinkage process, thus the particle of the blasting media waste is covered with an oxidized layer that is responsible for slowing the diffusion rate during sintering.
- The particle morphology of the blasting media waste differs according to its size: smaller particles have irregular shape and low density and it can be trusted to be from the impurities in the blasting media waste. Therefore, the particle density variation was considered by the augmented simplex lattice design performed together with the tap density method.
- Flow rate and apparent density of the blasting media waste are similar to the commercial powders.
- The microstructure of the sintered bodies is a globular cementite in a ferrite matrix with low hardness inherent to its thermal process of the sintering. The hardness is increased by quenching and tempering heat treatments due to carbon on the composition.
- Mixture experiments for powder waste selection and physical and technological characterization techniques can be applied to produce sintered bodies with low mechanical and chemical properties requirements.

The presented method does correctly achieve a promising densification with an economical raw material, but future efforts are needed to analyze the mechanical properties as tensile strength, elongation and fatigue limit of the sintered bodies. Furthermore, is relevant to study a method to remove the oxidized layer that covers the particles of the raw material in order to accelerate the sintering process.

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