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DUPLEX STAINLESS STEEL PRODUCTION AND STUDY OF STRESS RELIEF TREATMENT

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Abstract. *This study predicts the production and study of a UNS S31803 duplex stainless steel by powder metallurgy through the chips of reuse. The objective of this work is to analyze the influence of the strain relief treatment; thus, some samples were subjected to heating at 1050 ° C for 30 min, and cooled in water. The chips were milled with addition 3% vanadium carbide. The milling was realized using high energy ball milling in a planetary ball mill during. The milling time for 50 hours in an inert argon atmosphere to avoid oxidation of the powders, at a rotation of 350 rpm. A mass/sphere relationship of 1:20 was used. The particle size analysis and scanning electron microscopy were used in the characterization and identification of the particle size; it demonstrates a 67µm size. The technique of X ray diffraction was used in the identification of phases before and after the milling process. The stress relief treatment realized after the process it was obtained the highest values of density and lowest values of porosity.*

Keywords: *Stainless Steel, High Energy Milling, Strain Relief Treatment, chips, recycling.*

1. INTRODUCTION

Duplex stainless steels are composed of ferrite and austenite in equal volumetric fractions, exhibiting superior mechanical properties and corrosion resistance when compared to other steels in a wide range of applications (Yang, et.al, 2011; Gunn, 2003).

The conventional manufacturing process of stainless steel are complex, while the powder metallurgy is a process a near net shaping, reduces the costs of the process being more attractive for the production of the duplex stainless steel (Garcia, et.al, 2012). The automotive market introduces newly designed sintered parts in large amounts in produced cars. The new cars constructions are equipped with at least six powder metal flanges piece. Stainless steel is the preferred material for powder metal flanges because of its resistance to corrosion and oxidation (Dobrzański, et.al, 2007). High energy milling is the most common method of the plastic deformation to reach the extreme refining of structure (Shashanka & Chaira, 2015).

Reuse of industrial waste has many environment advantages like the energy consumption difference and effluents emission. The high energy mill powder processing technique allows the production of more homogeneous materials. Machining residues can be used for milling, and thus reusing the chips. According to Padilha (2000) produce metals from the recycling, the primary processing uses only 15% of the energy needed to obtain the same amount. The materials recycling avoids the materials discard for the environment, decreases the natural resources extraction and decrease in energy supply in the manufacture of innumerable kind of pieces (Delforge, et.al, 2007).

In the alloy parts in which is necessary corrosion resistance, there is an increase of powder milling application (Klar & Samal, 2007). Due to this application increase, several research works have been developed. Yonekubo, 2014, reused a superduplex stainless steel through the milling of chips coming of a machining process and produced the powder in a planetary ball mill, in the mass/sphere relationship of 1/16, during the time of 100 hours, at a rotation of 300 rpm. Others authors like Shashanka & Chaira, 2015, developed a duplex stainless steel at the planetary ball mill Fritsch at the time of 40 hours and the rotation velocity was 300 rpm. Kuffner, et.al., 2015 in their studies made the high-energy grinding of chips of a SAE 52100 steel, with 0%, 1% and 3% niobium carbide addition in a planetary mill during grinding times of 5, 10, 15 and 20 hours at a milling speed of 350 rpm and a mass/ball of 1:10, found greater efficiency in the milling process with the addition of carbide in the milling process.

This work has the objective to analyze the reused of the UNS S 31803 duplex stainless steel chips through the high energy milling with the addition of 3% of vanadium carbide. Besides this, seeks to analyze the strain relief treatment at the samples through of the characteristics and mechanical properties in the end of the process.

2. EXPERIMENTAL PROCEDURE

The chips were done in a planetary Ball Mill with addition of 3% vanadium carbide for 50 hours. The stainless steel was characterized by the X-ray diffraction technique. The particle size distribution was performed with Microtrac Bluewave S3500® equipment.

Before to the compaction and sintering process, some of the samples were subjected to a strain relief treatment in a temperature of 1050 °C for 30 minutes to relieve the stresses of the material and cooled in water. After the grinding step and stress relief treatment, all samples were compacted and sintered at 1200°C for 60 minutes. The compaction was made with uniaxial press with a load of 700 MPa.

The characterization of stainless steel duplex milled powder was realized using a scanning electron microscope Carl Zeiss EVO MA15. In the secondary electron (SE) mode, the particle size variation and morphology of powder were analyzed. Using the back scatter electron (BSD) and energy dispersive x-ray (DRX and Mapping) modes.

The technique of X ray (X-PERT Pro Model) diffraction was used in the identification of phases before and after the milling process and after a strain relief treatment in a temperature of 1050 °C for 30 minutes.

It was also analyzed the apparent density of the material sintered by the principle of Archimedes where, the sample was immersed in distilled water for 24 hours, according with the NBR 6220:2011, as show in the Eq. (1).

$$Densidade\ (\rho) = \frac{m_U}{m_U - m_I} \cdot \rho_{água} \tag{1}$$

For microestrutural analysis, the samples were cold drawn, sanded and polished. The metallographic experiments were realized through of optical microscopy (Olympus, model BX41). The porosity of the polished samples were studied through of image analysis used the Stream Basics software, in which the pores were identified like black pixels being possible the area calculation of each pore by percentage of surface area.

To evaluate the mechanical properties, micro hardness tests were realized on the samples with a load application of 1,96 N.

3. RESULTS AND DISCUSSION

The initial characterization of the steel chips UNS S31803 processed by machining, Figure 1, shows the average size of 8000 μm with plastic deformation at the surface originated by the machining tool. The chips used for the milling shows an irregular morphology with various sizes.

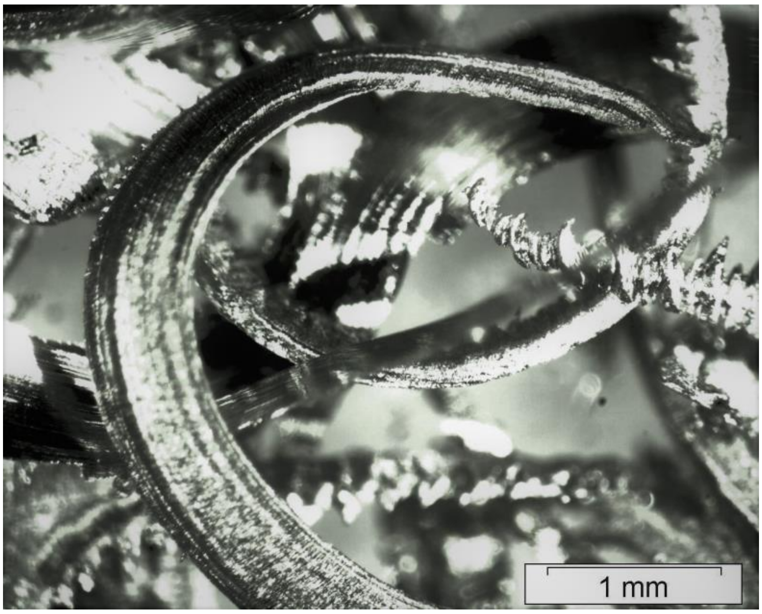

Figure 1. Duplex stainless steel UNS S31803 in chips form.

The Figure 2 shows the powder morphology of the duplex stainless steel UNS S31803 after the high energy milling process with the addiction of 3% of vanadium carbide. Can observe in this figure that the chips material forms were transformed in particles with irregular morphology, with heterogeneous sizes ranging from 10 by 120µm. There was a decrease of the order of 160 times of the particle size in relation to original chips size. Being the particle average size of the milling chips being around 48,91µm, showing a size significant decrease.

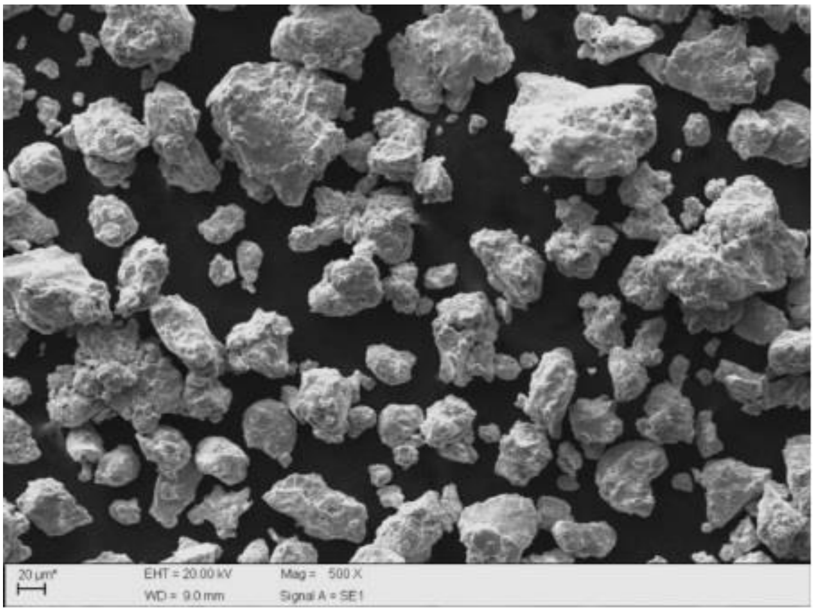

Figure 2. Particles morphology after the high energy milling for 50 hours of the steel UNS S31804 chips with the added of 3% of vanadium carbide by MEV.

The table 1 shows the distribution of sample particle size submitted to high energy milling with addition of 3% of vanadium carbide. The parameters D10, D50 and D90 represent the particle size in which are respectively 10%, 50% and 90% of the material total mass. The values for D10 indicate that 10% of the particles have smaller dimensions by 12.54µm and for the values of D50 and D90 are of the similar form, being 50% of the particles has smaller dimensions by 42.37µm and 90% with smaller dimensions by 90.65µm. The table 2, indicate the average values of the density obtained by the Archimedes method, green density and the sintered density. The measures of the green density were obtained after compaction with a load of 700 MPa, and by the Archimedes method, soon after the sintering by 1250°C.

Table 1. Parameters D10, D50 and D90 and average sizes of UNS S31803 duplex stainless steel particles with the added of 3% vanadium carbide.

Stainless steel particles after the milling	D10 (µm)	D50 (µm)	D90 (µm)	Average size (µm)
	12.54	42.37	90.65	48.91

The density measures was done in the green samples right after the unixial compaction with a load of 2 tons, and by the Archimedes method after the sintering stage by 1200°C for the samples submited to stress relief treatment. This was done with comparison purposes of the samples without treatment. The Table 2 shows that the stress reliaf tratment generated resulted in an increase of the density values of 5.72 g/cm³ to 5.94 g/cm³ for the samples with stress realif treatment. The density obtained was of 76% for the samples after the stress realif treatment compared to the molten sample. The values below that of the steel produced by fusion process, that is of 7.8 g/cm³. This decrease in density of the cast steel to the sintered is expected due to the greater presence of pores in the parts manufactured by powder metallurgy.

Table 2. Green density values, density of the sintering material and the density by the Archimedes method of the stainless steel with the added of 3% of vanadium carbide with and without stress relief treatment.

Treatment	Green density	Sintered density	Density Arquimedes
1050°C	5.59±0.1	5.78±0.05	5.94±0.34
Without treatment	5.19±0.42	5.31±0.36	5.72±0.04

The results of the X ray diffraction analysis are presented on the Figure 3. Observing the presences of austenitic and ferritic phase on the material as received. While the chips being to the milling, it is noted that the peaks decreased of intensity, happening a broadening of the peak, and until it disappears, having a tendency to amorphise that the austenitic peaks disappear and happened the appearance of the martensitic phase. The austenite peaks phase overlap with of the ferritic phase. When analyzing the figure 3.a, it is noted that after the sintering, occurs the absence of austenitic phase, and an increase of the ferrite and martensitic phase. This behavior is associated with the decrease in micro tensions caused by annealing, due to the process of recovery and recrystallization.

With the stress relief process in the temperature of 1050°C, observed the resurgence of the ferritic and austenitic phase, however the martensitic phase remained even after the sintering step at 1200°C for 1 hour, Figure 3.b. Due to the low percentage of vanadium carbide added in the process, being below the detection limit of the gadget it was not observed the carbide presence.

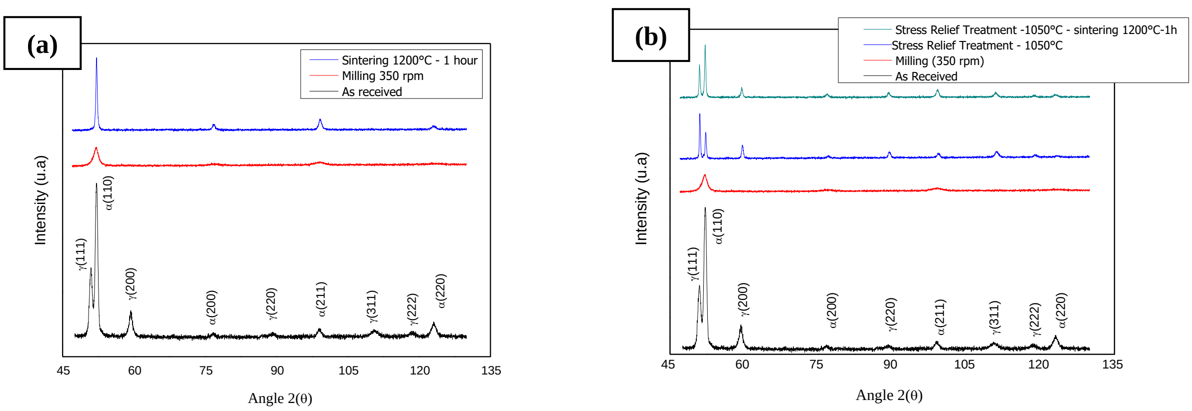

Figure 3. X ray diffraction: (a) stainless steel under the following conditions: (a) sintered at 1200°C for 1 hour; (2) Milling at the time of 50 hours; (3) samples as received. (b) Stainless steel under the following conditions: (1) milling at the time of 50 hours; (2) after stress relief treatment of the powder; (3) sintered at 1200°C for 1 hour; (4) samples as received.

The Figure 4 represents the porosity test realized with the technique of optical microscopy. It is verified that due stress relief treatment, the sample shows a higher porosity, 9.11%, than that the sample without treatment, 6.25%. However, the sample that was treated has a higher homogenization, evidencing that in the stress relief process the material showed higher diffusion of the particles. Due to the tension relief treatment realized at temperature of 1050° C, the process of diffusion began to occur in the material, and at the end the particle size obtained was greater in relation to the material only subjected to milling. After the sintering process, the density obtained was higher for the sample after the stress relief treatment, due to the highest particle size obtained and lower densification process.

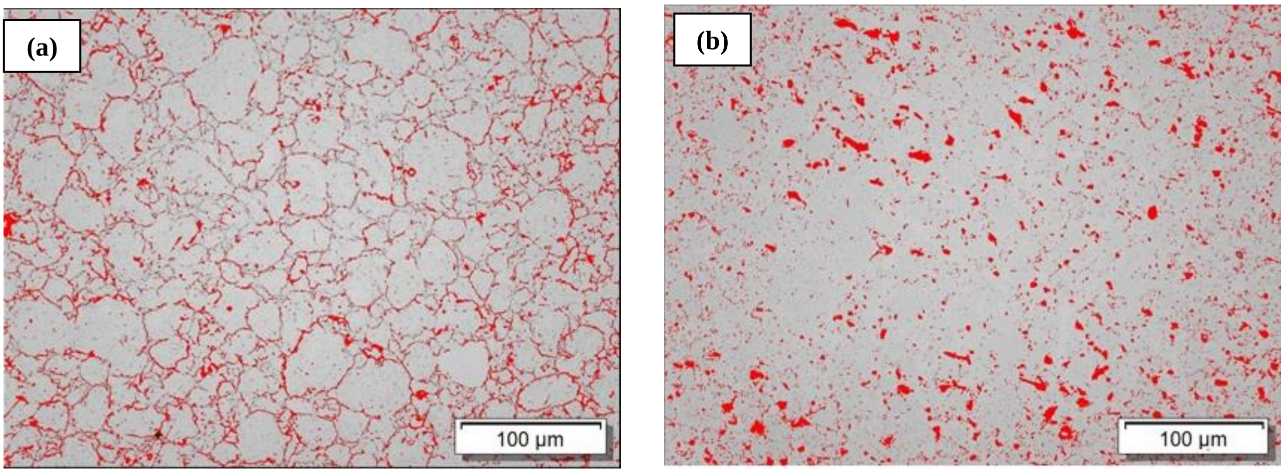

Figure 4. Optical microscopy test: (a) samples without stress relief treatment; (b) samples with stress relief treatment.

The Table 3 represents the micro hardness measures, in Vickers scale, of the samples submitted to the stress relief treatment and the sample without treatment. The sample without treatment has a micro hardness smaller, of 100 HV, and the sample with the treatment has a micro hardness of 113 HV. The microhardness values evidence the fact that the stress relief treatment decreases the material deformations, ensuring higher compaction and a better sintering, improve the mechanical properties of the material.

Table 3. Microhardness of the material submitted to the stress relief treatment at 1050°C and of the material without treatment.

Treatment	Micro hardness
1050°C	113±1
Without treatment	100±6

4. CONCLUSION

Through the high energy milling process, it was possible to recycle the machining chips. After thermal treatment, the ferritic and austenitic phases were observed, characterizing again a structure of duplex stainless steel. There was the presence of the martensite induced by deformation, even after the sintering step. The stress relief treatment realized after the process it was obtained the highest values of density and lowest values of porosity. This shows that after the stress relief treatment the material has a better compaction e sintering, and this is important to the mechanical properties.

5. ACKNOWLEDGEMENTS

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7. RESPONSIBILITY NOTICE

The authors Bruno Gonçalves Andrade, Flavio Henrique Guimarães de Souza, Leonardo Albergaria Oliveira, Claudiney de Sales Pereira Mendonça, Adhimar Flávio Oliveira, Vander Alkimin Ribeiro dos Santos, Gilbert Silva are the only responsible for the printed material included in this paper.