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# RECYCLING OF DUPLEX STAINLESS STEEL THROUGH OF POWDER METALLURGY WITH ISOSTATIC COMPACTION

### **Michele Stanziola Knychala**

Universidade Federal de Itajubá- UNIFEI, Itajubá – Minas Gerais  
michelestanziola@yahoo.com.br

### **Claudiney de Sales Pereira Mendonça**

Universidade Federal de Itajubá- UNIFEI, Itajubá – Minas Gerais  
sales.claudiney21@gmail.com

### **Mirian de Lourdes Noronha Mota Mello**

Universidade Federal de Itajubá- UNIFEI, Itajubá – Minas Gerais  
mirianmottamelo@unifei.edu.br

### **Gilbert Silva**

Universidade Federal de Itajubá- UNIFEI, Itajubá – Minas Gerais  
gilbert@unifei.edu.br

### **Bruno Gonçalves Andrade**

Universidade Federal de Itajubá - UNIFEI, Itajubá – Minas Gerais  
bruno.andrade@unifei.edu.br

### **Valesca Donizeti de Oliveira**

Universidade Federal de Itajubá- UNIFEI, Itajubá – Minas Gerais  
valescadoooliveira@yahoo.com.br

### **Abstract**

*High energy milling applied on duplex stainless steel chips resulting from machining was used in this research for waste recycling. The high energy milling was performed in a planetary ball mill with addition of 3% vanadium carbide, in the time of 50 hours in an argon atmosphere, rotation of 350 rpm and mass/ ball ratio of 1:20. Before compacting, the powder it was subjected to a tension relief treatment at 1050°C for 30 minutes. The samples were compacted in isostatic pressing and were sintered in vacuum at a 1250°C for 1 hour. By means of the granulometric analysis, it was verified that the milling resulted in particles with size around 49  $\mu\text{m}$ . Through the X-ray diffraction analysis, it was observed that the powder resulting from the milling has a tendency to amorphized, while the same material after the stress relief treatment presented the ferrite, austenite and martensite phases, which remained even after sintering. There was a density of the sintered material of the 6.57 g/cm<sup>3</sup> and porosity of 18%.*

**Keywords:** *Stainless steel duplex, chips, high energy ball milling, isostatic pressing.*

## **1. INTRODUCTION**

Nowadays, many industries, such as off shore, chemical, nuclear and oil and gas, make ample use of stainless steel because they have a good combination of mechanical properties and high corrosion resistance (Shashanka & Chiara, 2015)

Duplex stainless steels are known to have a microstructure consisting of two phases, ferrite and austenite, which are balanced to confer properties of both phases (Gunn, 2003). Due to the increase in raw material costs and the production of this steel there is a great incentive to develop new technologies for the reuse of chips resulting from machining in the manufacture of mechanical components. This material recycling is aimed at decrease environmental impacts rationalize energy and reduce costs (Delforge, et.al. 2007).

Stainless steels processed through powder metallurgy present a very interesting field of research, as it represents an attractive combination of mechanical strength and corrosion resistance associated with its biphasic (austenitic-ferritic) microstructure (Brytan, et. al. 2009). To improve the mechanical properties of steels produced by powder metallurgy, a metal-ceramic compound is used in which the addition of carbides as reinforcement is advantageous (Trueman, et.al., 1999). Metal carbides have important properties, such as high temperature stability, high hardness, good electrical and thermal conductivity (Gubernat & Zych, 20014; Kosolapova, 1971). Kuffner et.al, 2015 investigated that with the addition of niobium carbide in the high-energy grinding of an AISI 52100 steel there was an increase in the efficiency of the grinding process with greater particle size reduction.

The purpose of this work is to reuse the machining chip of a duplex stainless steel through the high energy mechanical milling with addition of vanadium carbide, this work verified the influence of metallic carbides on the properties of the final product, and the feasibility of using the technique by analyzing the properties of the metal-ceramic composite obtained.

## 2. EXPERIMENTAL PROCEDURE

The milling of chips with an addition of the 3% vanadium carbide was realized in the rotation of 350 rpm, mass/ball 1:20 ratio, milling time of 50 hours and inert atmosphere to avoid oxidation. In order to determine the size and distribution of particle were used an analyzer of particle size.

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. The back scatter electron (BSD) and EDX spectrum gave the information on the elemental composition of the material.

Before to the compaction and sintering process, the samples were subjected to a strain relief treatment at a temperature of 1050°C for 30 minutes. For the compaction process the powder was submitted initially the uniaxial pressing to the load 1.5 ton to molding the body and after was submitted an isostatic pressing at load 400 MPa. Then the specimens were sintered at 1250°C for one hour and cooled in the furnace.

The X-ray diffraction technique was used to identify the phases present in the microstructure. By means of an optical microscope, the image analysis was performed to evaluate the porosity of the sample. Density was also measured using the Archimedes principle.

## 3. RESULTS AND DISCUSSION

Figure 1 shows the initial characterization of UNS S31803 steel chips obtained by machining. It is observed that the obtained chips have an average size of 8000  $\mu\text{m}$  with regions of plastic deformation resulting from the shear caused by the machining.

Figure 2 shows the micrograph of the vanadium carbide, it is possible to observe that the particles presented granulometry varying from 5 to 10  $\mu\text{m}$ .

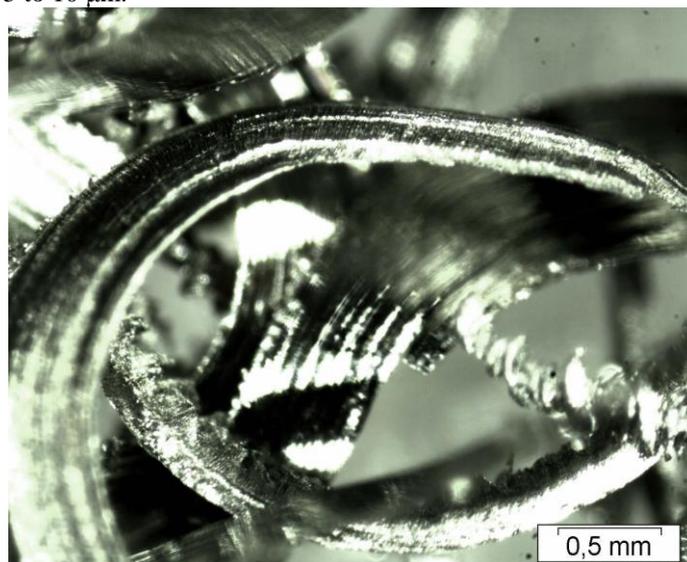


Figure 1. Stainless steel duplex UNS S31803 in the form of machining chips.



Figure 2. Micrograph of the vanadium carbide powders.

Figure 3 shows the morphology of the powder of the stainless steel UNS S31803 after being submitted to 50 hours of milling with an addition of 3% of VC. It can be observed in this figure that the material in the form of chips was transformed into particles with irregular morphology, with size ranging from 20 to 150 μm. The particle size obtained was 150 times smaller than the original chip size.

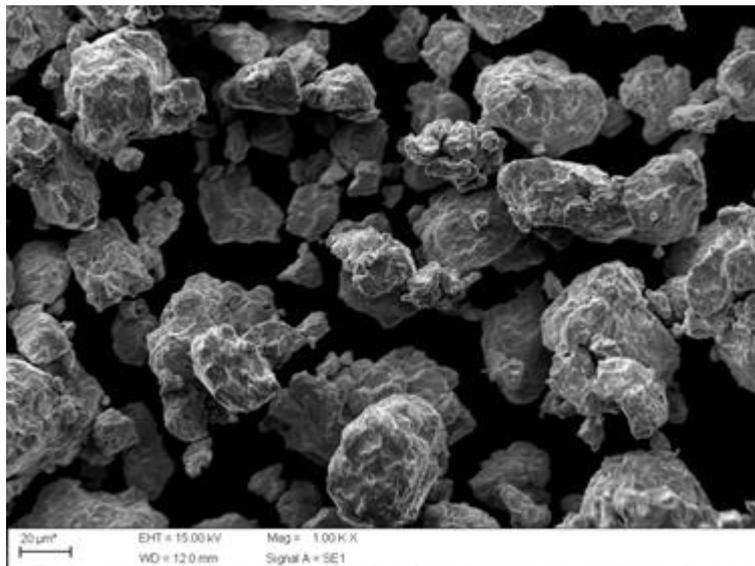


Figure 3. Particle morphology after high energy milling for 50 hours of UNS S31803 steel chips with addition of 3% vanadium carbide.

Figure 4 shows the particle size analysis, the granulometric distribution is found in values of 20 and 200 μm. The particle size for the 50% distribution factor (D50) was 69.28 μm with 50 hours milling, this indicates that 50% of the particles present up to this size. In the same way, the values reported for D10 and D90 are 36.02 μm and 152.40 μm. The material submitted to milling with an addition of carbide has a particle size below 150 μm and the average size obtained was 67.42 μm.

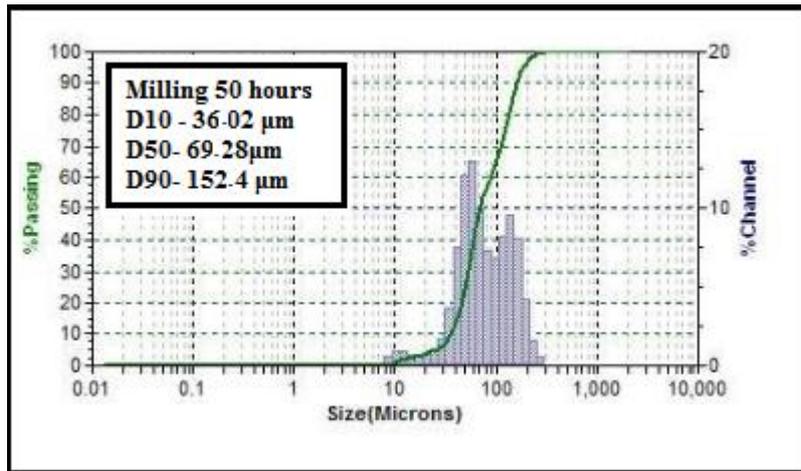


Figure 4. Stainless steel duplex UNS S31803 with 50 hours of milling with an addition of 3% of VC.

With the X-ray was possible to obtain the diffractogram of the material that shows the phases present in it, represented by Figure 5. It is possible to observe the presence of austenite and ferrite phases on the material "as received". It was also observed that the peaks intensity has a decrease, occurring a widening of the peak, due to the tendency to amorphized. It was observed through the diffractograms of the material submitted to the stress relief treatment at 1050 ° C, the appearance of the ferritic and austenitic phases. Due to the low percentage of vanadium carbide added in the process, being below the detection limit of the equipment, it was not observed the presence of the carbide.

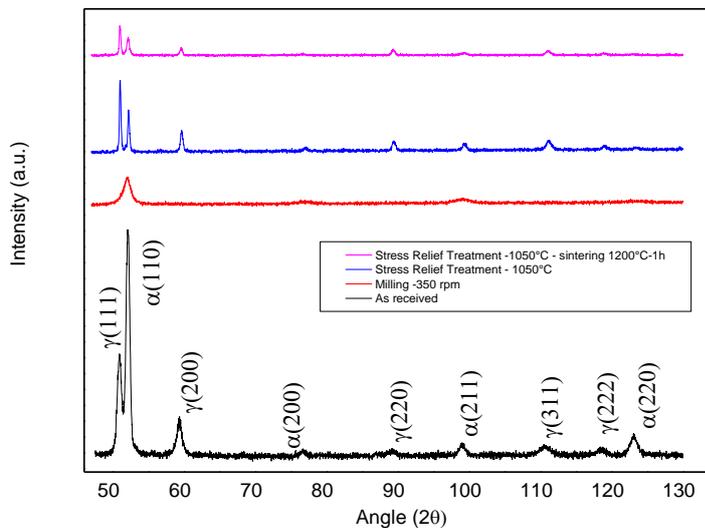


Figure 5. X-ray diffraction pattern after 50 hours of milling.

Figure 6 shows the micrograph of the sample of a stainless steel compacted in an isostatic press and then sintered at 1250 ° C. The porosity generated from the analysis of optical microscopy images was based on five images, in which the average porosity was 18.3% of the sintered samples. Figure 6 (a) shows the lowest porosity value found to be 12.57%, and Figure 6 (b) shows the highest value that is 21.53 %.

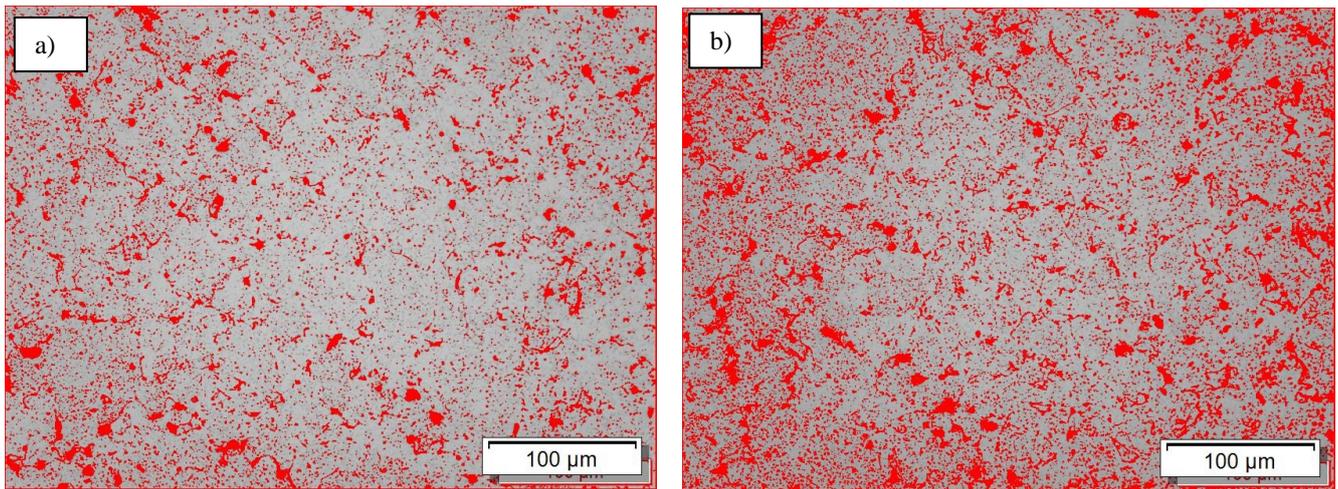


Figure 6.a) Microstructure sintered stainless steel at 1250 ° C for 1 hour, region of lower porosity. Figure 6.b) Region of greater porosity. Regions in red indicate porosity. Images obtained by optical microscopy.

Figure 7 shows the EDS spectrum for the associated image obtained by BSD. The spectrum illustrates the presence of all constituent elements of duplex stainless steel such as iron, chromium, nickel. It has been observed that the vanadium particles are located in some specific and porous regions.

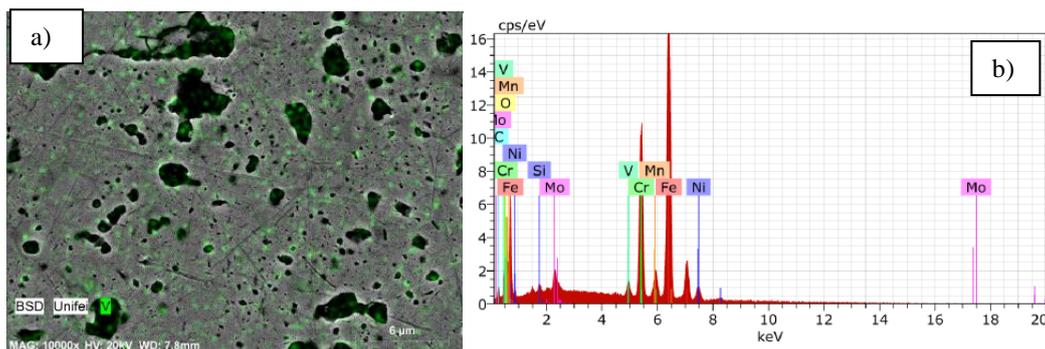


Figura 7. (a) Distribution of vanadium particles (green) on stainless steel particles. Figure 7. (b) EDX spectrum analysis of stainless steel with addition of 3% VC.

Table 1 shows the microhardness for samples of the material received and the sintered material at 1250°C for 1 hour. The decrease between the values of the microhardness of the sintered sample in relation to the sample in the condition of the material as received is probably due to the presence of pores in the sintered sample and the low atomic diffusion occurring during the sintering at 1250 ° C. The microhardness values obtained were 87% in relation to the sample as received

Table 1. Microhardness values of sintered material and material as received.

Condition	Microhardness (HV)
Sintered at 1250°C- 1 hour	232.02±7.98
Received material	284.16±3.76

#### 4. CONCLUSION

The present research is about the reuse of UNS31803 steel chips through high energy mechanical milling process. This study was intended to analyze the efficiency of the addition of vanadium carbide and the use of an isostatic pressing in the powder metallurgy process. The study of the sintered material showed a porosity of approximately 18% and a density around 84% of the bulk. The final product was characterized by a microstructural composed of ferrite, austenite and martensite. Therefore, the high energy milling process can be used as an alternative for the reuse of the chip of a duplex stainless steel.

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#### 6. RESPONSIBILITY NOTICE

The authors Michele Stanziola Kynchala, Claudiney de Sales Pereira Mendonça, Bruno Gonçalves Andrade, Valesca Donizeti de Oliveira, Mirian de Lourdes Noronha Mota Mello, Gilbert Silva are the only responsible for the printed material included in this paper.