Chapter 6

Metal Matrix Composites Added of Nanostructured Tantalum Carbide

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Additional information is available at the end of the chapter

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1. Introduction

Metal matrix composites (MMC) reinforced with dispersed ceramic particles have received considerable interest over the years and are still in constant development in order to expand their applications in industry. It is a brilliant choice for applications that require mechanical strength and wear resistance. They combine a soft metal matrix with hard ceramic particles resistant to wear (Gordo, 200).

Different matrixes and reinforcements for MMC have been studied, and therefore studies have been conducted with various combinations of metal matrixes with reinforcement of ceramic powders aimed at obtaining composites with similar properties or superior to conventional steel tool. Carbides such as NbC, TaC, VC, and TiC have been combined with iron or steel powders to produce sintered composites. Niobium and tantalum carbide can be used for structural purposes and also for the production of refractory components, or as steel reinforcement by dispersed particles. Particularly, reinforced steel has been used in the automotive and textile industries and also in the manufacture of high-speed cutting and ore milling tools (Martinelli, *et al*, 2007). Advances in research (Silva, 2005; Silva, *et al*, 2005; and Silva, *et al*, 2012) and marketing of steel reinforced with NbC and TaC particles have also contributed to add value to manufactured products strategically using mineral sources produced in northeastern Brazil.

New techniques for the production of refractory metal carbides (WC, NbC, TaC, TaxNby) have been developed by synthesizing nanostructured carbides that provide improvement of diverse properties of materials compared to materials obtained by conventional methods (Medeiros, 2002 and Medeiro, et al, 2005). Uniform distribution and fine particle size of nanosized particles of the reinforcing phase added to steel provide homogeneous dispersion of these steel carbides into the matrix, thus providing uniformity of properties and allowing compounds to be used in a variety of applications.

In the production of metal composites reinforced with carbides, powder metallurgy offers some economic and technological advantages in relation to other competing processes such as low cost of raw material processing and relatively low temperatures involved in the process. As the microstructure of the sintered steel is the result of process parameters (time, milling speed, compaction pressure, sintering time and temperature, sintering in solid or liquid state) and also the characteristics of starting powders (size and particle size distribution, compressibility and chemical purity); any changes in these parameters also affect the sintering kinetics, with wide variations in their microstructure and consequently their performance in relation to specific application. The use of the technique Powder Metallurgy (PM) in the manufacture of MMC composites is increasing.

2. Synthesis of nanostructured carbides — TaC

Refractory, ultrathin and nanostructured metal carbides (TaC, NbC, MoC and WC) have been produced at low temperature from ammonium oxalate complexes via gas-solid reactions with suitable characteristics for various purposes: high surface area (38 to 58m^2/g), size of crystallites (18-20 nm), extremely porous particles and high acidity.

Recent discoveries aimed at facilitating the routes for obtaining these carbides with high surface area have led to a rapid growth in the application of these materials in catalysis, composite alloys (Medeiros, 2002; Oyama, 1992, and Ledoux, 1990) and metal-ceramic composites (Upadhyaya, 1998 and Matthews, 1994).

Nanosized refractory metal carbides, particularly TaC, are produced at low temperature and short reaction time through gas-solid reaction in fixed bed reactor using tris (oxalate) hydrated ammonium oxytantalate-$(NH4)_3TaO(C_2O_4)_3nH_2O$-precursor of fine grain size (ultrafine particles with high surface area). However, for successful precursor carburizing process, certain parameters such as temperature, composition of the gas mixture (H_2 and CH_4), methane concentration, reaction time, precursor cost and heating rate must be taken into account.

The system used for carrying out gas-solid reactions is composed of a horizontal tubular resistive furnace (alumina tube) with temperature controller, fixed bed reactor (slip casting), gas inlet and outlet system, valves and flow meters. The gas composition determines reduction and carburization, which is characteristic of atmosphere. The amount of CH_4 in the mixture cannot be too excessive, since much carbon would precipitate on the surface of particles, hindering the reaction and producing free carbon in the product, which hinders the diffusion process. In the case of low CH_4 concentration, carburization does not occur. The complete carbide reaction occurs at temperature below 1000°C with CH_4 concentration ranging from 3 to 5%. Under some conditions, carburization is completed within 2 hours (Medeiros, 2002).

http://dx.doi.org/10.5772/59187

In recent work (Lima, 2013) Fig.1, TaC synthesized revealed the formation of pure phase with characteristics different from those conventionally obtained for the product, with average crystallite size of the order of nanometers, approximately 12.05 nm, and surface area of 19 m^2/g. These characteristics allow its use not only as reinforcement for MMC composites and solid inserts but also as catalysts.

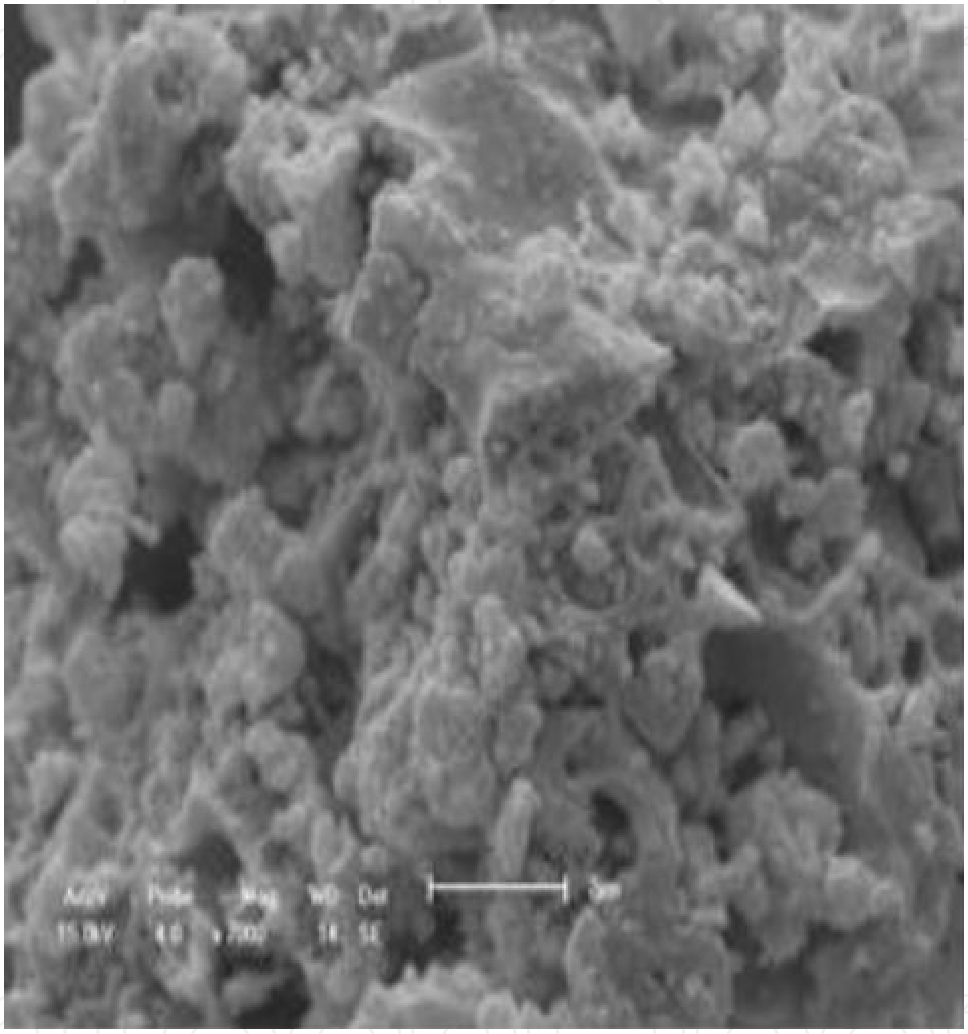

Figure 1. Electron microscopy of tantalum carbide (Lima, 2013). Magnification of 7000x.

3. Eurofer steel — Complexity microstructure and property

Eurofer steel, also called Eurofer 97 due to the year 1997 when its chemical composition was defined, is the end product of a synthesis of four steels of reduced activity that were studied in the European Union (MANET, OPTIMAX, BATMAN and OPTIFER I and II) (Lucon, *et al*, 2006). Although, a huge progress in the improvement of this steel has been developed in recent years (Lindal, 2005), studies with Euroferno with regard to optimization of the reduced activity, increased fluency and decrease in the ductile-brittle transition temperature after radioactive damage remain under constant growth, evolving thus to EUROFER 2 and 3. The aim is that EUROFER 3 becomes a residue of low level of radiation after 80 or 100 years when applied to the DEMO demonstration reactor (Daum and Fischer, 2000; Huang *et al*, 2007).

The mechanical and corrosion resistance properties of martensitic stainless steels can be seriously impaired particularly in function to the precipitation of complex phases, generally rich in chromium, operating temperature, or even during processing; thus, the thermal cycles to which these steels are submitted should be conducted under absolute control.

To provide the steel mechanical strength, hardness and toughness needed, the most common heat treatments consist of tempering followed by single or double thermal treatment. The main parameters involved in this case are: heating and cooling rate, austenitization temperature and time and tempering thermal cycles (Mariano, 2007).

In the specific case of Eurofer steel, the improvement of its chemical composition aiming at reduced activity was achieved by replacing niobium by tantalum and molybdenum by tungsten. Nb, Mo, Ni, Cu, Al and Co were restricted to ppm values. Calculations aided by computer simulation programs indicate that Eurofer is a very promising reduced-activity alloy.

The mechanical properties of ferritic-martensitic steels restricted their use to temperatures above 550°C. Subsequently, the addition of fine and homogeneously dispersed particles allowed their use at higher temperatures (650°C) to give origin to ferritic-martensitic alloys hardened by oxide dispersion (ODS) (Lindau, 2005). To date, the most promising of this series is the ODS-EUROFER alloy. This reduced-activity alloy was developed by the Karlsruhe Research Center (Forschungszentrum Karlsruhe, FZK), in cooperation with France and Russia aiming nuclear applications.

EUROFER steel (9Cr-1W) can be used in turbines for power generation, pressure vessels, nuclear reactors or applications where the material is submitted to temperatures between 250 and 550°C. One way to improve the properties of steel, so that it works at higher temperature or become more stable is to add second-phase particles into the matrix. These particles can be in the form of oxides, carbides, nitrides, or even in solid solution when certain chemicals are added to the material.

4. Metal matrix composite — MMC with ceramic reinforcement

Metal matrix composites significantly evolved from the 20's. This composite was first used in the aerospace industry. More recently, various areas of industry have increasingly studied this type of material, for example, automotive, electronic, and nuclear, among others. MMCs attract many researchers and engineers as a good alternative to develop materials with high technological potential.

Generally, the aim is to create material with the hardness of ceramics, combined with metal that has high toughness compared to ceramics. Thus, they form a family of materials called

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metal-ceramic materials. Nucleation and precipitation of carbides in steel to obtain good mechanical strength coupled with high toughness or the manufacture of hard metals containing high amount of carbides and / or nitrides, in which, when processed by powder metallurgy form a continuous ceramic skeleton, are classical examples of these materials (Breval, 1995).

In principle, forged steel and most conventional steels can be treated as metal matrix composites since this forged matrix has a dispersed phase. The dispersed phase may be composed of oxide, sulfide, nitride, carbide, etc. In addition, many metal alloys should be included as MMC if the microstructural definition is considered. Although the definition of a composite material is very comprehensive, there is a strong tendency to believe that the solidification direction of eutectic microstructure is within the possible definitions of MMC (Ralph, *et al*, 1997).

Different applications are found when a metal is reinforced. The reinforcement of light metals, for example, opens the possibility of their use in areas where weight reduction is a priority. The main objectives of reinforcing steel (not limited to only these) are:

- Increase in elasticity modulus and tensile strength at room temperature and above it, while maintaining minimum ductility or greater toughness;
- Increase the fluency resistance at high temperatures compared with conventional alloys;
- Increase fatigue resistance, especially at high temperatures;
- Improve the thermal-shock resistance;
- Increase corrosion resistance.

Therefore, composites are versatile in their applications because they combine the distinctive properties of each material that composes them.

Metal matrix composites can be obtained with continuous fiber reinforcement and the use of particulate reinforcements. However, particulate reinforcements have significant advantages because the cost of manufacture of such composite is reduced and conventional metallurgical processes such as powder metallurgy and casting, followed by post-processing steps such as lamination, forging and extrusion can be used. Depending on the type of particulate reinforcement, the composites obtained may have higher use temperature compared to the matrix material, improved thermal stability and wear resistance, such as Al $_{(matrix)}$ / Si $_{(fiber)}$ composites. Therefore, research efforts have been directed to obtain metal matrix composites and particulate reinforcement (Levy and Pardini, 2006).

5. High-energy milling — Reinforcement by dispersing nanoparticles into the matrix

High-energy milling is a technique fo the processing of powders in solid state involving repeated cycles of deformation, cold welding, fragmentation and cold re-welding of powder particles held in a high-energy ball mill (Suryanarayana, 1998; Suryanarayana, 2001; Koch, 1998). This method uses high-energy milling to form composite powders typically for long milling times (Gomes *et al*, 2001; Costa *el al*, 2002.).

In high-energy milling, the constant ball-powder-ball collisions result in deformations and fractures that define the dispersion of components, homogenization, phases and the final powder microstructure. The nature of these processes depends on the mechanical behavior of the powder components, their equilibrium phase and stress state during milling (Suryanarayana, 1998 and 2001). High-energy milling can be performed with three different categories of metal powders or alloys, namely: ductile-ductile components, ductile-brittle components and brittle-brittle components.

The influence of particle dispersion with the formation of a second phase, slowing the metal surface movement and its sintering, has been proposed by Kuczynsky and Lavendell, who considered that moving particles act as barriers to the surface advancement. Later, it was discussed that particles are swallowed by the surface movement exerting force and preventing its movement (Sbrockey and Johnson, 1980).

The dispersion of large amounts of ceramic particles by powder metallurgy aims to improve their tribological properties and thus their mechanical properties; therefore, 12% SiC powder with average size of 3 μm was added to 316L steel powders of 5 μm, mixing for 8 hours, uniaxially compressed at 100 MPa and sintered at temperatures of 1100°C for one hour in inert atmosphere, resulting in complete fusion of samples (Patankar, *et al*, 2000).

Another study that used 3% W TaC dispersed in the metal matrix of atomized 316L austenitic stainless steel and processed by powder metallurgy showed higher density values and a significant increase in hardness compared to material without reinforcement (Oliveira, 2008).

6. Sintering

Sintering is a non-equilibrium thermodynamic process in which a system of particles (powder or compacted aggregate) acquires a coherent solid structure by reducing the specific surface area. The result is the formation of grain boundaries and growth of necks and inter-particle bond, typically leading the system to densification and volumetric shrinkage (Gomes, 1995).

Solid-phase sintering occurs at temperature where none of the system elements reaches their melting point. This is accomplished with material transport (atomic diffusion, vapor transport, viscous flow, etc) (Costa, 2004).

In the second case, liquid-phase sintering leads to the formation of a liquid phase capable of dissolving a percentage of particles. This yields a diffusion pathway analogous to the grain boundary in the solid-phase sintering, causing a rapid initial density increase and then, dissolution of solid particles in the liquid and precipitation on the neck region occur (Gomes, 1995).

The sintering of metal matrix composites can be divided into two categories: a) solid-phase sintering (including powder metallurgy) and b) liquid-phase sintering (Kocjak, *et al*, 1993).

http://dx.doi.org/10.5772/59187

This is a complex step in the powder technology route and is influenced by several parameters such as: solubility, self-diffusivity and inter-diffusivity, mutual solubility and wettability, etc., which act simultaneously on the process and systems are so diverse that it has not yet been possible to develop a single sintering model capable of meeting the most varied and possible systems. Therefore, the solution is to develop a model for each system to be studied.

7. Case study: Developing a Metal Matrix Composite (MMC) — Eurofer 97 steel reinforced with tantalum carbides — TaCs

EUROFER 97 steel is a promising alloy for use in nuclear reactors or in applications where the material is submitted to working temperatures up to 550°C due to its lower strength under fluency. Factors that influence the slip of boundaries are the grain morphology, angle and speed of grain boundaries. The speed can be reduced with the presence of a dispersed phase in the material, provided it is thin and evenly distributed.

The state of Rio Grande do Norte is a major producer of refractory metals (W, Ta, Nb), ceramic minerals (diatomite, kaolin, feldspar, mica, barite, clays, etc.) and other minerals containing rare earths and semi-precious stones. However, this natural wealth that places Rio Grande do Norte among the top five producers of minerals in the country has not reversed in progress and development for the region, mainly due to lack of technology to aggregate these resources to local raw materials.

This study presents the development of a new metal matrix composite (MMC), which has the following starting materials: ferritic / martensitic EUROFER 97 stainless steel and tantalum carbides – TaC as reinforcement, one of them synthesized in laboratory (UFRN) and the other supplied by Aldrich, the first with average crystallite size of 13.78 nm and the second with crystallite size of 40.66. TaC nanometric particles were inserted into the metallic matrix through the processing steps of powder metallurgy seeking to improve the properties of the final product.

Initial sintering studies with EUROFER 97 steel reinforced by TaC nanosized particles dispersed into the matrix of MMC composites showed satisfactory values with respect to the improvement of the mechanical properties regardless of processing, as sintered materials with similar microhardness values and even greater that 100% the value of 333.2 HV for bar-shaped pure steel was obtained (Oliveira, 2013).

7.1. Experimental procedure

EUROFER steel was received in the form of bar and has ferritic / martensitic microstructure as can be seen in Figure 2, as well as the presence of grain boundaries with considerable sizes, unable to be viewed in full due to their size indicated by arrows. Microhardness measurements were made and the average value was 333.2 HV and according to the literature, the theoretical density ranges from 8.0 to 8.1 g/cm^3.

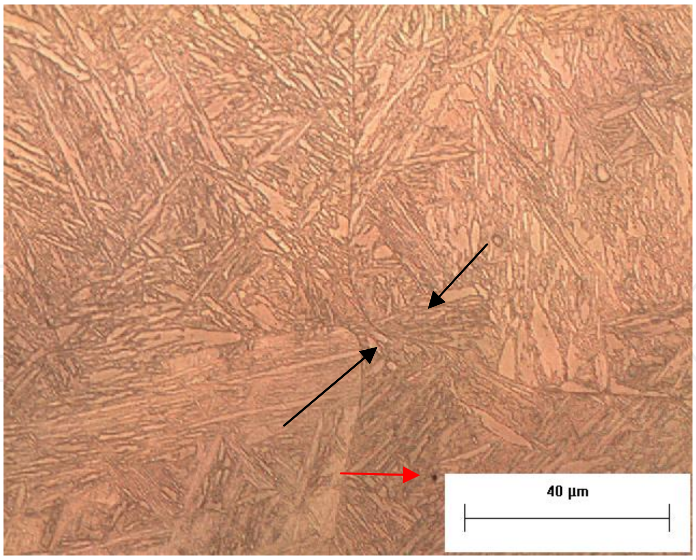

Figure 2. MO micrograph (500x) of EUROFER steel as received, attacked with 2% Vilela.

After characterization of the bar-shaped starting material, machining was performed, where chip was removed, and from this, the grinding process began. Firstly, pure steel was performed, and then steel with the addition of 3% UFRN TaC and 3% commercial TaC. The powders were milled for 5 hours in high-energy mill. Each of the resultant particulate samples were characterized by x-ray diffraction and SEM and then cold compacted under uniaxial pressure of 600MPa in a cylindrical array of 5 mm in diameter. Subsequently, the compressed powders were sintered in a vacuum oven at temperature of 1250°C isotherm for 60 minutes. Sintered samples were cooled to room temperature. The structure of sintered samples was observed by optical and scanning electron microscopy and analyzed by microhardness tests.

7.2. Results

Figure 3 shows the microstructure and the X-ray pattern of pure EUROFER steel milled for 5 hours. The electron micrograph of EUROFER 97 shows uneven particles with rough surface, which can be compared to the morphology of powder atomized with water, Fig (3a). The X-ray diffraction pattern, Figure (3b) shows only peaks associated to Fe with CCC structure, which is the same structure of the ferrite phase. This is a strong indication that the milling conditions used significantly influenced the phase transformation occurred during sintering of EUROFER steel. During milling, the starting percentage of martensite was transformed into ferrite from the effects of powder processing.

Figure (4a) shows the electron micrograph of EUROFER 97 steel composite powder with addition of elemental TaC (UFRN) after 5 hours of milling. It was observed that there is a similarity in images-SEM of pure steel powder and composite powder, Figure (4a) and Figure (4a). The presence of carbides (light spots) is noticeable, which are distributed and inserted in a dispersed form on the surface of the metal matrix (dark gray surface). Figure (4b) shows the XRD pattern of X-ray of EUROFER 97 steel composite powder with elemental TaC (UFRN)

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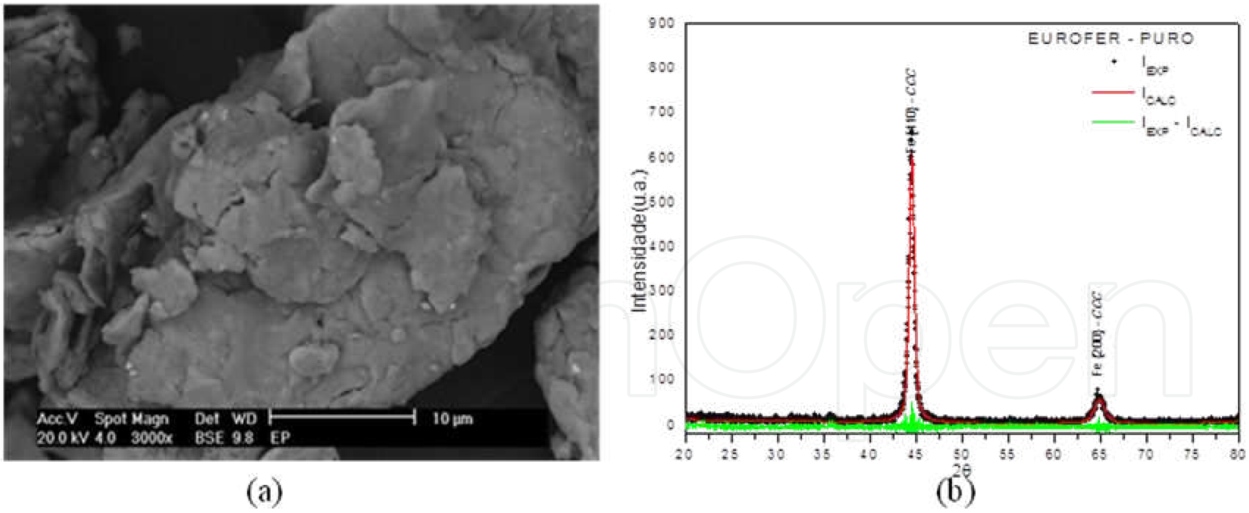


Figure 3. Pure EUROFER 97 steel powder milled for 5 hours, (a) SEM-magnification 3000x and (b) XRD.

milled for 5 hours, which presents peaks characteristics of steel and TaC, and their respective phases. The diffractogram shows only the ferrite phase, without martensite, for iron after 5 hours of milling.

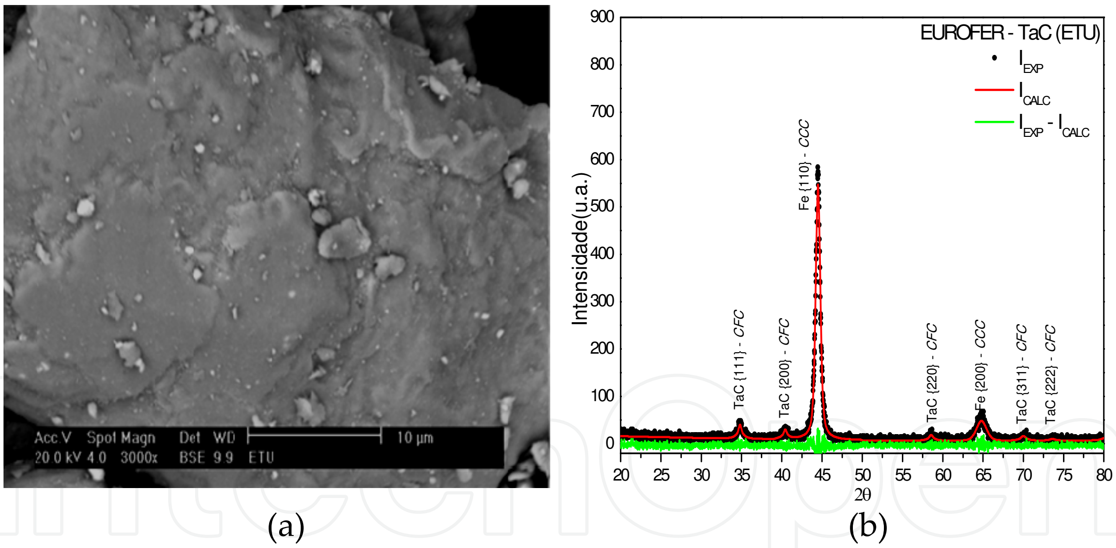
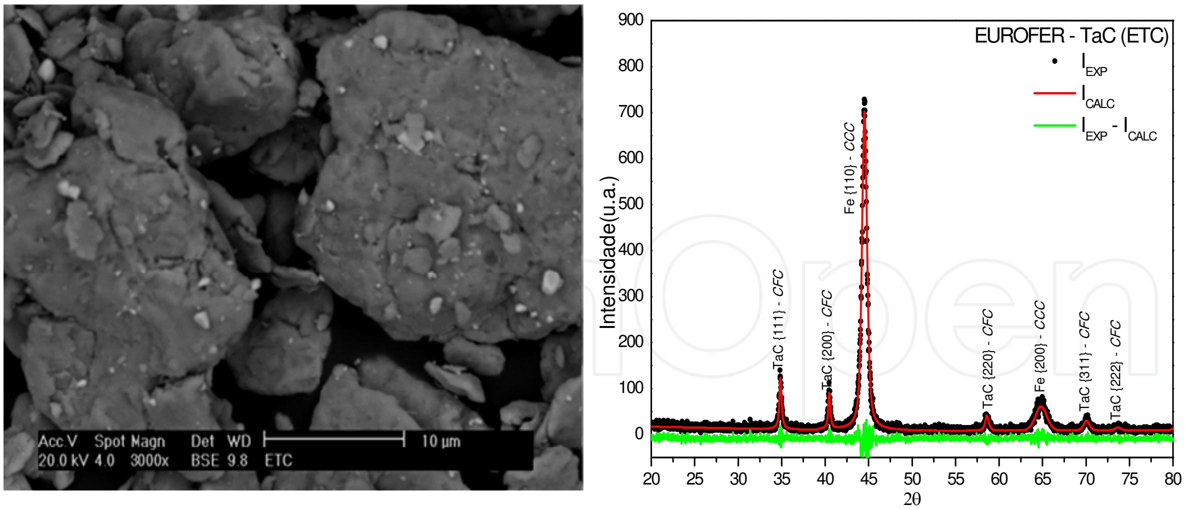


Figure 4. EUROFER 97 steel powder with TaC UFRN milled for 5 hours, (a) SEM-magnification 3000x and (b) XRD.

Figure (5a) shows the SEM image and figure (5b) shows the diffraction standard of X-ray of EUROFER 97 steel composite powder with commercial TaC milled for 5 hours in a high-energy planetary mill. Initially, the SEM microstructure shows heterogeneous particle size and shape and the non-uniform dispersion of carbides into the metal matrix compared to particulate composites of steel with UFRN TaC Figure (4a). XRD pattern shows peaks of Fe with TaC and CCC structure with greater intensity and height for carbide when compared with XRD pattern of the EUROFER 97 composite powder with UFRN TaC, Figure (4b).



(a) (b)

Figure 5. EUROFER 97 steel powder with commercial TaC milled for 5 hours, (a) SEM-magnification 3000x and (b) XRD.

Figure 6 shows the micrographs (SEM) of samples sintered at temperature of 1250°C for 60 minutes. Figure (6a) shows sintered pure EUROFER steel with microstructure that indicates that it is in the final stage because pores are small and rounded, typical of this stage. Figures (6b) and (6c) show the dispersion of TaC particles in ETU and ETC composites, in which it is observed that TaC particles (white portion) are dispersed in the grain boundaries of the metal matrix (gray portion); in the case of ETU sample still with the presence of many pores, a large cluster of TaC particles randomly dispersed in the metal matrix between the boundaries and within the pores of the sintered sample was observed, hindering the closing of pores and the non densification of the material.

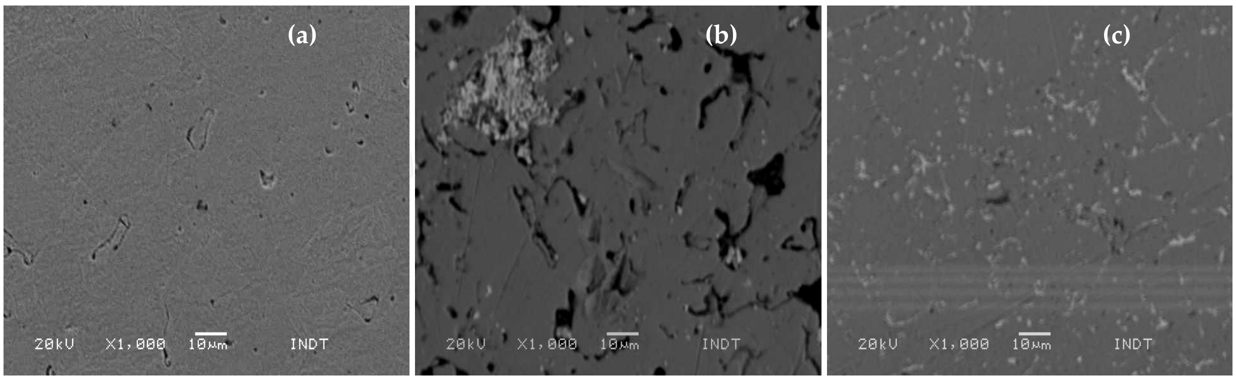


Figure 6. Micrographs (SEM-1000x) of samples sintered at 1250°C for 60 minutes.; (a) Pure EUROFER (EP), (b) EUROFER with UFRN TaC (ETU) and (c) EUROFER with commercial TaC (ETC).

Micrographs (OM) of Figure 7 show sintered pure steel (Fig. 7a), sintered steel with UFRN TaC (Fig7b) and sintered steel with commercial TaC (Fig. 7c). Regarding porosity, there are small and well rounded pores, indicating the final sintering stage. The idealized sinteriza-

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tion models proposed by various authors are specific to each stage. Figure 7a shows in the micrograph of sintered EUROFER pure steel sample, the size of grain boundaries and the presence of two phases, ferrite (lighter portion) and martensitic (darker portion); Figure 7b shows sintered ETU still with the presence of many pores compared to the two other sintered samples under the same conditions; however, pores in the segmented form in particles and also rounded pores, indicating sintering at stage from intermediate to the final stage. Microstructure with diffuse phases, dark and clear portions, was observed. Figure 7c shows densified samples with grain boundaries with regular shapes and small sizes; however, the presence of a single phase characteristic of ferritic phase can influence the mechanical properties of the material.

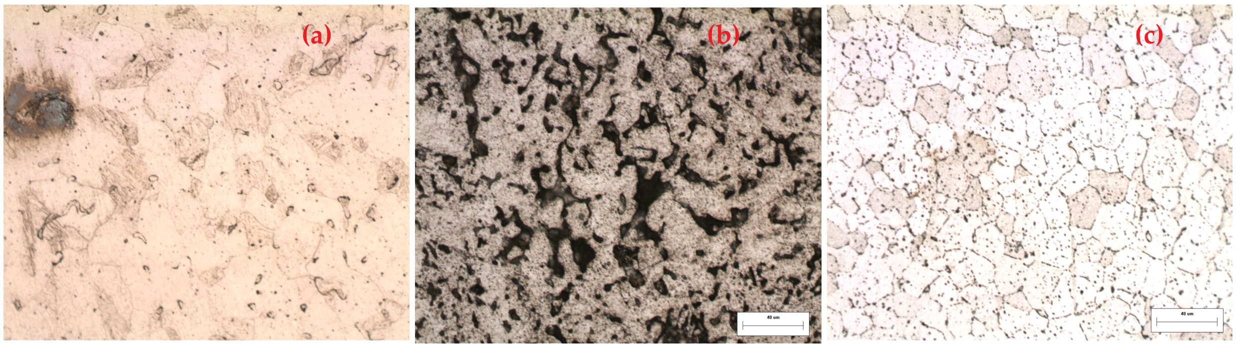

Figure 7. Micrographs (SEM-500x) of samples sintered at 1250°C for 60 minutes; (a) Pure EUROFER (EP), (b) EUROFER with UFRN TaC (ETU) and (c) EUROFER with commercial TaC (ETC).

Table 1 shows the microhardness values obtained for pure Eurofer 97 and samples added of elemental TaC sintered at temperature of 1250°C and time of 60 min.

It was observed that the ETU sample obtained the highest hardness value compared to all other samples, which can be related to the size of very fine particle, described in experimental procedure, not even when dealing with a well-densified sample.

The large difference in the microhardness values among pure steel samples can be observed, and for this, the processing of powder metallurgy (milling and sintering) was sufficient to improve the mechanical properties of steel samples with UFRN TaC compared to steel samples with commercial TaC, both at the same sintering temperature and sintering time. These differences in the microhardness values can be probably associated with the dispersal of coarse and / or fine particles in the metal matrix, and this is due to the synthesis of carbides; even with low energy input, UFRN TaC produced particles with small crystallite size and more homogeneous compared to that provided by Aldrich, or may even be related to phase transformation, i.e., in sintered samples with commercial TaC, the prevailing phase is ferrite, which has low hardness, whereas in those with UFRN TaC, the presence of other phases, i.e., the metal matrix composite remains ferritic / martensitic as the initial steel and the presence of fine particles dispersed or embedded in the metal matrix of the EUROFER steel.

Samples	Microhardness (HV)
EP	636.8 ± 35.6
ETU	700.6 ± 17.0
ETC	191.2 ± 20.3

EP= Pure EUROFER;
ETU=EUROFER + UFRN TaC;
ETC=EUROFER + commercial TaC

Table 1. Microhardness results of composites milled for 5 hours and sintered at temperature of 1250°C for 60

8. Conclusions

The process of TaC powder synthesis generated microstructure of refined grains with rounded morphology of particles, and crystallite size (13.78 nm) that may have influenced densification and also the microhardness of sintered samples.

Tantalum carbide-TaC with nanosized crystallites, strongly agglomerated, forming particles smaller than EUROFER 97 steel particles, were dispersed by milling, remaining compacted among particles that compose the metal matrix, thus obtained material with refined microstructure, smaller grain sizes and good mechanical properties.

SEM images show sintered pure steel samples and steel composites with UFRN TaC, in which microstructure typical of sintered material with initial, intermediate, final sintering stages were observed. In addition, the dispersion of tantalum carbides in the EUROFER steel metal matrix was observed.

Optical microscopy showed a change in grain size and in the microstructure of the different sintered materials. For samples with the addition of TaC, grain size decreased or remained stable compared with sintered pure steel. Sintering at temperature of 1250°C provided high densification in composite reinforced with Aldrich TaC but the microhardness result was low, which can be related to a phase change, i.e. steel became fully ferritic.

The powder metallurgy processing was sufficient to improve the mechanical properties of EUROFER steel, with small difference in relation to microhardness results between EUROFER steel and composite reinforced with UFRN TaC. In the case of composite reinforced with commercial TaC, there is a considerable difference, which can be related to the phase transformation and / or presence of coarse particles compared with UFRN TaC powder particles, which are thinner.

New EUROFER 97 steel composite with TaC was developed and characterized according to appropriate procedures for the manufacturing processes used in this work and with altered mechanical properties and future features such as: special applications in nuclear reactor, cutting materials and many others.

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