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Effect of the Additives of Nanosized Nb and Ta Carbides on Microstructure and Properties of Sintered Stainless Steel

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1. Introduction

Many applications on modern technologies try to combine uncommon properties that can't be attended by conventional metallic alloys, ceramics and polymeric materials. On this way, engineers of aerospace, for example, are ever looking for structural materials that have low densities, be stronger, with wear resistance and that have high resistance to corrosion (Gordo et al., 2000). These characteristics combine amazing properties for just one material. Normally, very dense materials are harder with high resistance to wear. Thus, composite materials are designed to provide this mixing of controlled properties.

The principle of combined action of different properties from two or more distinct materials characterizes the composite materials. Then, it's the central principle of composite materials that is enhancing one or more properties my mixing materials in various ways. Thus a very elementary example of a composite would be mud mixed with straw; still a very widely used material in the construction of houses. These innovative materials open up unlimited possibilities for modern material science and development.

Basically there are three kinds of composite materials, they are: polymer matrix composite (PMC), for example fiberglasses embedded in a thermoplastic resin of polyethylene; ceramic matrix composite (CMC) with carbon fibers reinforcing SiC matrix; and metallic matrix composite (MMC) to reinforce a matrix of steel with refractory ceramic hard particles.

This last one kind of composite material will be discussed in this chapter with focus on effect of the dispersion of nanosized carbides (NbC and TaC) in the sintered microstructure of the stainless steel 316L. Powder metallurgy technique will be applied as well as mechanical alloying and the effects promoted by hard particles in a metal matrix. A practical case study will be shown.

2. Metal Matrix Composites (MMC)

Metal matrix composites are an attractive end goal for many materials engineers as an alternative to the traditional pure ceramics or pure metal material. The goal is to create a

material that has nearly the same high strength as the ceramics, but also high fracture toughness due to the contribution from the metal, this has lead to the development of a new family of metallo-ceramic composites. Classical examples are the precipitation and nucleation of carbides in steel for obtaining good strength while still maintaining most of the toughness from the metal, or the fabrication of hard metals such as a matrix cobalt containing a large percentage of grains of hard tungsten or titanium carbides or nitrides, which during the powder processing grow together and form a continuous skeleton (Breval, 1995).

In principle, wrought irons and most conventional steels could be treated as MMCs since they have a metallic matrix which is reinforced with a dispersed phase that could be oxides, sulphides, nitrides, carbides, etc. Even if we take a definition through "microstructural design", many conventional engineering alloys, such as some steels involving dispersions created at a moving alpha-gamma interface, really ought to be included. Although, composite material is a definition very embracing, it is quite tendentious to think that a directional solidification of a eutectic microstructure is within the framework of an MMC definition (Ralph, 1997).

The reinforcement of metals can have many different objectives. The reinforcement of light metals opens up the possibility of application of these materials in areas where weight reduction has first priority. The precondition here is the improvement of the component properties. The development objectives for light metal composite materials are:

- Increase in yield strength and tensile strength at room temperature and above while maintaining the minimum ductility or rather toughness,
- Increase in creep resistance at higher temperatures compared to that of conventional alloys,
- Increase in fatigue strength, especially at higher temperatures,
- Improvement of thermal shock resistance,
- Improvement of corrosion resistance,
- Increase in Young's modulus,
- Reduction of thermal elongation.

To summarize, an improvement in the weight specific properties can result, offering the possibilities of extending the application area, substitution of common materials and optimization of component properties. With functional materials there is another objective, the precondition of maintaining the appropriate function of the material. Objectives are for example:

- Increase in strength of conducting materials while maintaining the high conductivity,
- Improvement in low temperature creep resistance (reactionless materials),
- Improvement of burnout behavior (switching contact),
- Improvement of wear behavior (sliding contact),
- Increase in operating time of spot welding electrodes by reduction of burn outs,
- Production of layer composite materials for electronic components,
- Production of ductile composite superconductors,
- Production of magnetic materials with special properties.

For other applications different development objectives are given, which differ from those mentioned before. For example, in medical technology, mechanical properties, like extreme corrosion resistance and low degradation as well as biocompatibility are expected. Although increasing development activities have led to system solutions using metal composite materials, the use of especially innovative systems, particularly in the area of light metals, has not been realized. The reason for this is insufficient process stability and reliability, combined with production and processing problems and inadequate economic efficiency. Application areas, like traffic engineering, are very cost orientated and conservative and the industry is not willing to pay additional costs for the use of such materials (Kainer, 2006).

Reinforcements for metal matrix composites have a manifold demand profile, which is determined by production and processing and by the matrix system of the composite material. The following demands are generally applicable:

- Low density,
- Mechanical compatibility (a thermal expansion coefficient which is low but adapted to the matrix),
- Chemical compatibility,
- Thermal stability,
- High Young's modulus,
- High compression and tensile strength,
- Good processability,
- Economic efficiency.

On the other hand, the matrix has the role of distribute and transfer the tensile and to protect the surface of reinforcement. Reinforcements can be used as particles, whiskers or fibers. Unfortunately, high strength fibers and their processing methods are extremely expensive, and this limits their wide industrial application. Moreover, continuous fiber reinforced composites do not usually allow secondary forming process, which is used in the original shape in which they were manufactured. As a result of these limitations, over the last few years, new efforts on the research of non-continuous reinforced composites have been applied. Composites with non-continuous reinforcement do not have the same level of properties as continuously reinforced composites, but their cost is lower, their processing methods are more adaptable to conventional ones and their performance is acceptable (Torralba et al., 2003).

The selection of suitable matrix alloys is mainly determined by the intended application of the composite material. With the development of light metal composite materials that are mostly easy to process, conventional light metal alloys are applied as matrix materials. In the area of powder metallurgy special alloys can be applied due to the advantage of fast solidification during the powder production. Those systems are free from segregation problems that arise in conventional solidification. Also the application of systems with oversaturated or metastable structures is possible (Kainer, 2006).

Normally, the process to produce MMC involves at least two steps: consolidation and synthesis (i.e. introduction of the reinforcement into the matrix), following by a modeling operation. There are a lot of techniques to consolidate; MMC reinforced with discontinuous fibers are susceptible to modeling through mechanical forming (i.e. forging, extrusion and lamination).

3. Powder Metallurgy (P/M) used to obtain composite materials

Between lots of ways to produce composite materials, powder metallurgy (P/M) is an easy way to manufacture composite materials with metallic matrix and offer some advantages compared with ingot metallurgy or diffusion welding. The main advantage is the low manufacture temperature that avoids strong interfacial reaction, minimizing the undesired reactions between the matrix and the reinforcement. In other cases, P/M allows production of some materials which cannot be obtained by any other alternative route (for example, SiC reinforcing Ti alloys). Composites that use particles or whiskers as reinforcement can be obtained easier by P/M than by other routes. Another advantage of P/M is the homogeneous distribution of reinforcements. This uniformity not only improves the structural properties but also the reproducibility level in properties (Torralba et al., 2003).

In short, powder metallurgy is the process of blending fine powdered materials, pressing them into a desired shape (compacting), and then heating the compressed material in a controlled atmosphere to bond the material (sintering). Sintering is a term applied to bond the material and it describes the process in which aggregated or compacted powders acquire a solid structure, coherent through internal displacements of mass by diffusion process of atoms activated by temperature (Gomes, 1995). P/M process generally consists of four basic steps: (1) powder manufacture, (2) powder blending, (3) compacting and (4) sintering. Compacting is generally performed at room temperature, and the elevated-temperature process of sintering is usually conducted at atmospheric pressure. Hot Isostatic Pressure (HIP) is another way to press and to sinter at the same time. This technique avoids some troubles in the pressing step. Optional secondary processing often follows to obtain special properties or enhanced precision such as thermal treatments. Each step has specific parameters that must be studied and controlled to reach the performance desired.

Among some techniques to manufacture powders, such as liquid metal atomization, chemical and thermal decomposition reactions, electrolyte deposition, sol-gel process and milling, this last one will be shortly discussed in the next section.

4. Mechanical alloying and milling

Two kinds of millingcould be distinguished - conventional low energy milling (e.g. in a ball mill) and high energy milling. In the last case, the most used devices are planetary ball mill, SPEX ball mill and attritor ball mill. High energy milling (HEM) and mechanical alloying (MA) are terms utilized to name the synthesis of alloys and compounds by means of intensive milling that causes mixing at an intimate level and deformation of the materials microstructure (Costa et al. as cited by Benjamin, 1976 and Suryanarayana, 2001). MA is a solid-state powder processing technique involving repeated welding, fracturing, and re-welding of powder particles in a high-energy ball mill. Originally developed to produce oxide-dispersion strengthened (ODS) nickel- and iron-base superalloys for applications in the aerospace industry, MA has now been shown to be capable of synthesizing a variety of equilibrium and non-equilibrium alloy phases starting from blended elemental or prealloyed powders. The non-equilibrium phases synthesized include supersaturated solid solutions, metastable crystalline and quasicrystalline phases, nanostructures, and amorphous alloys. Recent advances in these areas and also on disordering of ordered

intermetallics and mechanochemical synthesis of materials have been critically reviewed after discussing the process and process variables involved in MA (Suryanarayana, 2001). The process of MA consists of loading the powder mix and the grinding medium (generally hardened steel or tungsten carbide balls) in a container sealed usually under a protective atmosphere (to avoid/minimize oxidation and nitridation during milling) and milling for the desired length of time. There are some parameters that must be controlled in the milling process, such as ball to powder ratio, length of milling time, atmosphere and ball sizes (Suryanarayana, 2001).

Nowadays, mechanical alloying has been successfully applied to prepare Ni-based, Fe-based, and Al-based oxide dispersion strengthened (ODS) alloys and carbide dispersion strengthened (CDS) alloys. During preparation of these materials, the strengthened phase is directly put into the matrix powders as a raw material. After a long time of ball milling, mechanical alloying can be achieved to some extent, but it is unlikely to produce the optimum combination between the strengthened phase, and the matrix. Element powders which can form oxide or carbide are mixed with the matrix powders and then subjected to ball milling, the supersaturated solid solution will form first, from which the dispersion strengthened alloy can be fabricated by a precipitation of second phase during heat treatment, or by mechanical-chemical reaction. This is a new method to produce CDS and ODS alloys by mechanical alloying, which can be used to optimize the strengthened action of the second phase to the maximum (Wang et al., 2001). During mechanical alloying, the mechanical energy transfers from the balls to the alloy powders, which results in the severe plastic deformation and the introduction of dislocations, lattice distortion and vacancies. When hard particles are incorporated into the matrix they promote an effect of hardening.

5. Mechanism of reinforcement

The characteristics of metal matrix composite materials are determined by their microstructure and internal interfaces, which are affected by their production and thermal mechanical prehistory. The microstructure covers the structure of the matrix and the reinforced phase. The chemical composition, grain and/or sub-grain size, texture, precipitation behavior and lattice defects are of importance to the matrix. The second phase is characterized by its volume percentage, its kind, size, distribution and orientation. Locally varying internal tension - due to the different thermal expansion behavior of the two phases - is an additional influencing factor. With knowledge of the characteristics of the components, the volume percentages, the distribution and orientation, it might be possible to estimate the characteristics of metallic composite materials. The approximations usually proceed from ideal conditions, i.e. optimal boundary surface formation, ideal distribution (very small number of contacts of the reinforcements among themselves) and no influence of the component on the matrix (comparable structures and precipitation behavior). However, in reality a strong interaction arises between the components involved, so that these models can only indicate the potential of a material. The different micro-, macro- and meso-scaled models proceed from different conditions and are differently developed (Kainer, 2006 as cited by Clyner & Withers, 1993 and McDaniels et al., 1965).

6. Effect of ceramic particles in a metal matrix

Ceramic particles are some of the most widely used materials for reinforcing metal matrix. In this way, the aging process can accelerate in aging alloys and the hardness can be improved considerably with direct impact and wear behavior (Torralba, 2003). Plastic deformation of a metallic material is associated to sliding of crystalline planes of crystalline structure and, therefore, there is a hardening and increase of mechanical resistance of the material when obstacles are placed to this movement. The particles of the reinforcement distributed uniformly into the grain serve as the obstacles to displacement of the discordances. The mechanisms of hardening occur when this discordance goes through the reinforcements or surrounds these reinforcements to continue its movement. In this sense, these mechanisms promote the hardening of the material during the plastic deformation. Obviously, the closer the particles are, the better tensile strength is improved. This is due the difficulty in movement of the discordances through or around the particles.

Compared with their unreinforced alloys, steel matrix composites offer higher hardness and wear resistance and their elastic modulus is the highest among all machinable materials and materials which can be hardened. On the other hand, as in all metal matrix composites, reductions in the fracture properties (ductility, toughness) are to be expected. Finally, the incorporation of dispersed ceramics particles inside the matrix can improve sintering, corrosion and oxidation properties of composites.

7. Case study – Effect of the dispersion of nanosized carbides (NbC-TaC) in the sintered microstructure of the stainless steel 316L

Composites containing different hard phases have already been studied (Tjong and Lau, 1999). Hard phases added to the steel matrix were mainly refractory carbides such as NbC, TaC and VC (Gordo et al., 2000). WC-Co (Ban and Shaw, 2002), Cr_2Al, $TiCr_2$, SiC and VC (Abenojar et al., 2002 and Abenojar et al., 2003) were also investigated as a potential reinforcement of the steel matrixes. This case study proposes a practical analysis to improve density and hardness of hard particles, used as additive. Recently a possibility to produce very homogeneous compacts with relative density among 95% without exaggerated grain growth was reported. These results were attributed to the incorporation of nanosized carbides' particles into stainless steel matrixes (Gomes et al., 2007).

7.1 Experimental procedure

This case study was carried out with starting powders of water-atomized stainless steel 316L manufactured by Höganäs. Pure sample and samples with addition of up to 3% of NbC and with 3% of TaC produced in the UFRN (Federal University of Rio Grande do Norte, Brazil) laboratory, previously characterized were mechanically milled in a conventional ball mill for 24 hours and axially cold pressed in a cylindrical steel die at 700 MPa. Sintering was carried out in vacuum. Samples were heated up to 1290°C with heating rate 20°C/min and isothermally held for 30 and 60 minutes. Sintered samples were characterized by x-ray diffraction, scanning electron microscopy and density and hardness were measured.

7.2 Results

Figures 1, 2, 3, 4, 5 and 6 show the x-ray diffraction patterns and micrographs of NbC and TaC used in this work. Only carbides characteristics peaks were identified. The carbides produced by UFRN show wider peaks than supplied by others due to the crystal refinement in accordance with previous work (Souza et al., 1999 and Medeiros, 2002). The carbides synthesized by UFRN are finer than those supplied by other manufacture. The small crystallite size and lattice strain values, provided by Rietveld's Method presented in the table 1, confirm it. Both carbides synthesized in UFRN, NbC (MCS = 16.67nm) and TaC (MCS = 13,78nm) present similar medium crystallite size (MCS).

Powder	**Size (nm)**								**Lattice Strain (%)**
	NbC (2θ)								
	34.90	40.49	58.40	69.90	73.40	87.48	97.70	MCS (nm)	
NbC-U	17.65	17.64	17.10	16.43	16.22	15.27	16.38	16.67	0.0009
NbC-C	75.77	75.65	77.22	79.44	80.61	84.73	80.35	79.11	0.000145
	TaC (2θ)								**Lattice Strain (%)**
	34.88	**40.48**	**58.60**	**70.06**	**73.66**	**87.61**	**98.03**	**MCS (nm)**	
TaC-U	15.91	15.47	14.14	13.39	13.18	12.43	11.97	13.78	0.00156
TaC-C	42.68	41.62	40.19	39.79	39.73	40.04	40.61	40.66	0.000102
MCS = Crystallite medium size; NbC-U and TaC-U = carbides synthesized by UFRN; NbC-C and TaC-C = carbides supplied by Johnson Matthey Company and Sigma Aldrich.									

Table 1. Crystallite size and lattice strain obtained for the carbides.

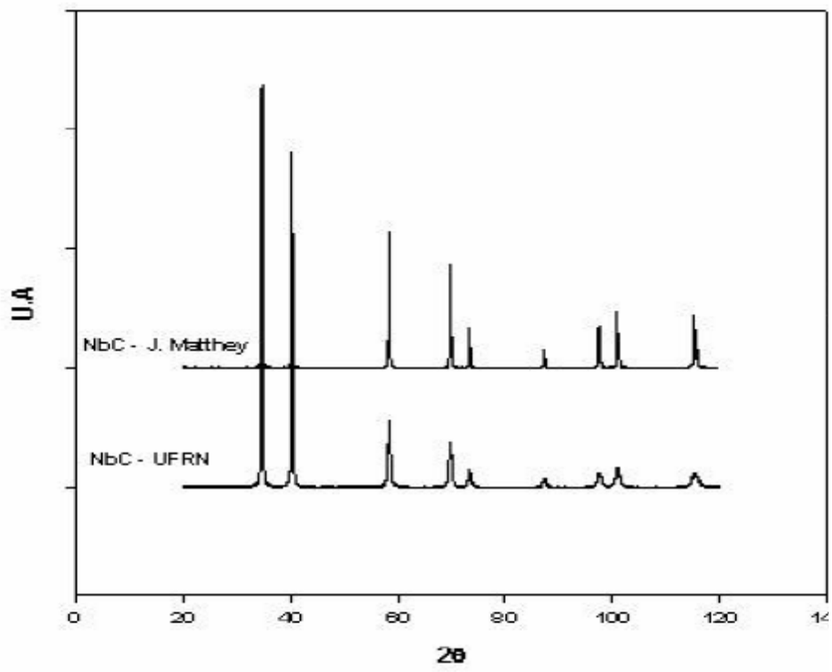

Fig. 1. X-ray diffraction patterns of Niobium Carbide produced at UFRN and from Johnson Matthey Company.

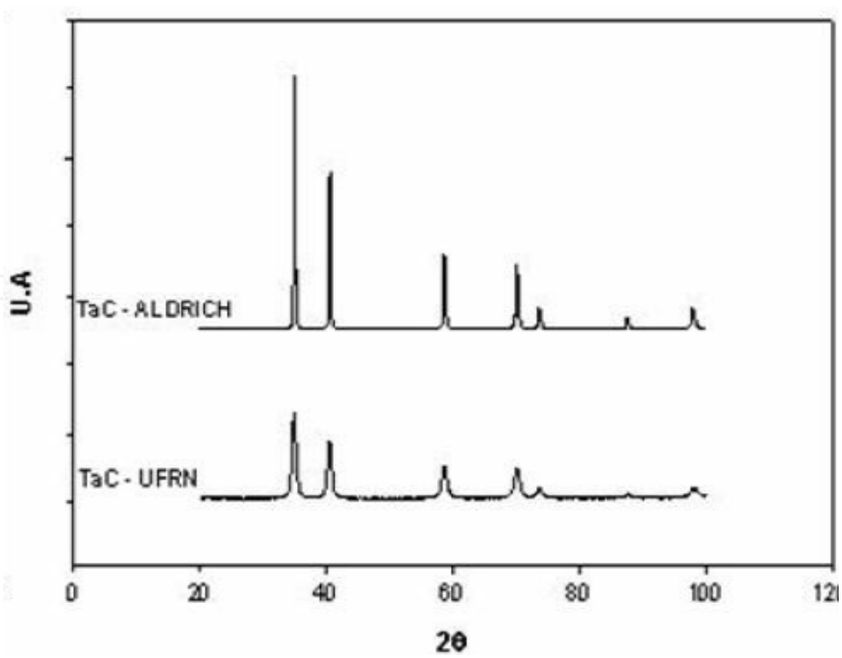

Fig. 2. X-ray diffraction patterns of Tantalum Carbide produced at UFRN and from Sigma Aldrich.

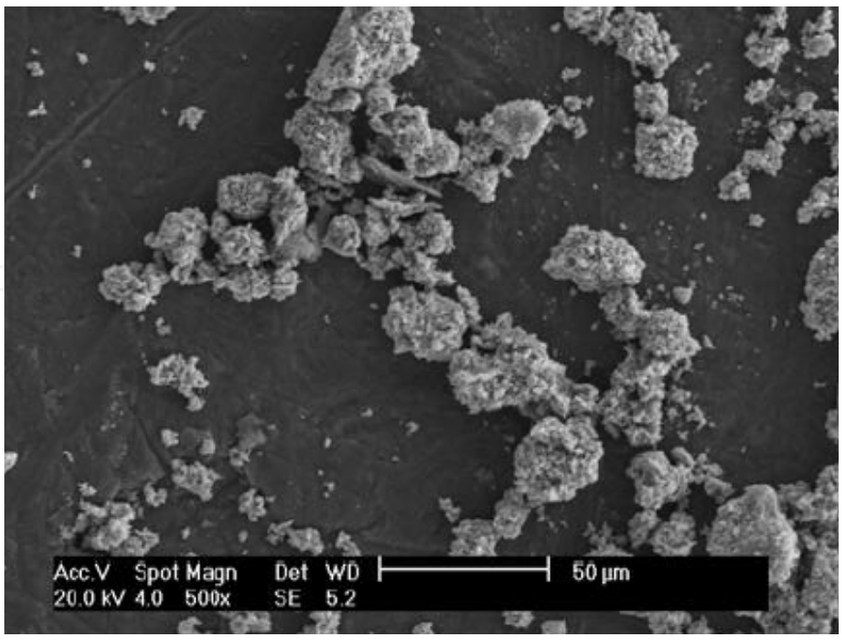

Fig. 3. Scanning electron micrographs of NbC as supplied from UFRN – 500x.

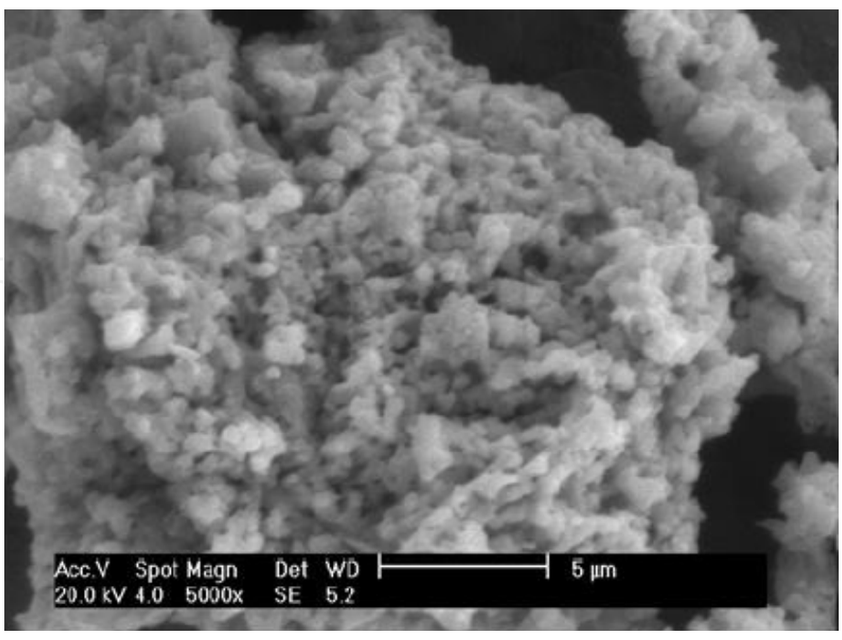

Fig. 4. Scanning electron micrograph of NbC as supplied from UFRN – 5000x.

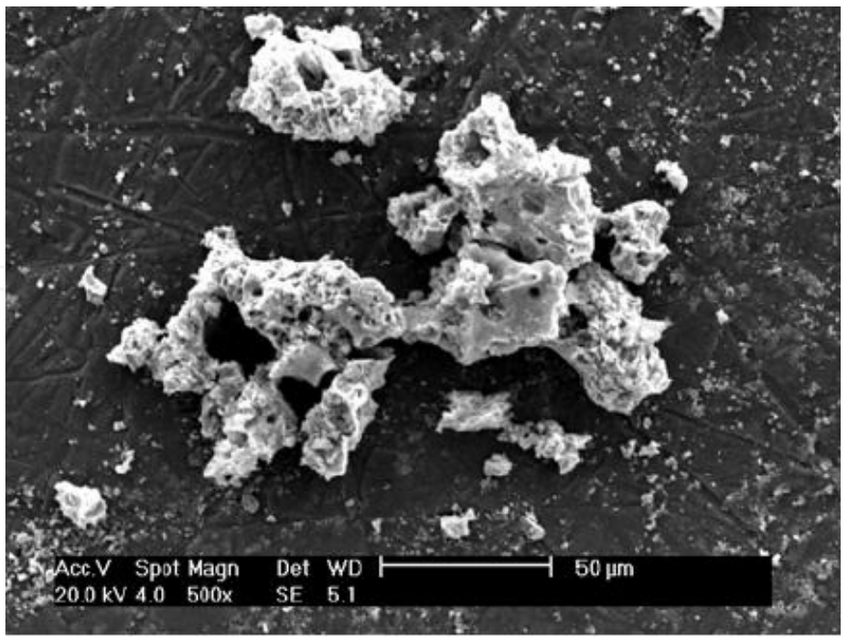

Fig. 5. Scanning electron micrographs of TaC as supplied from UFRN – 500x.

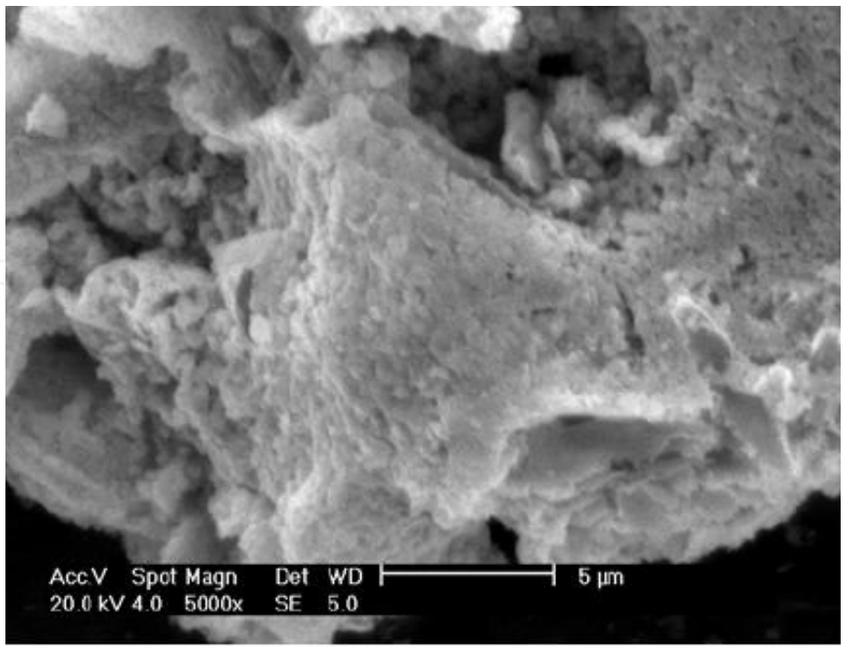

Fig. 6. Scanning electron micrographs of TaC as supplied from UFRN – 5000x.

Figures 7 and 8 show the micrographs of composite powders milled mechanically 24 hours, with addition (3%) of NbC (Fig. 7) and TaC (Fig. 8). The arrows indicate the carbide particles.

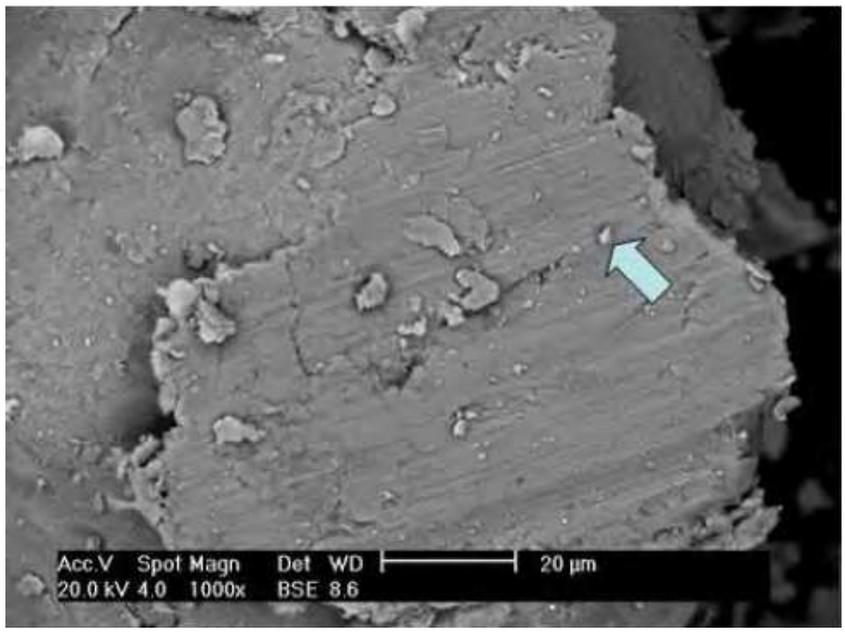

Fig. 7. Scanning electron micrographs of stainless steel powders reinforced with 3% of NbC from UFRN – 1000x.

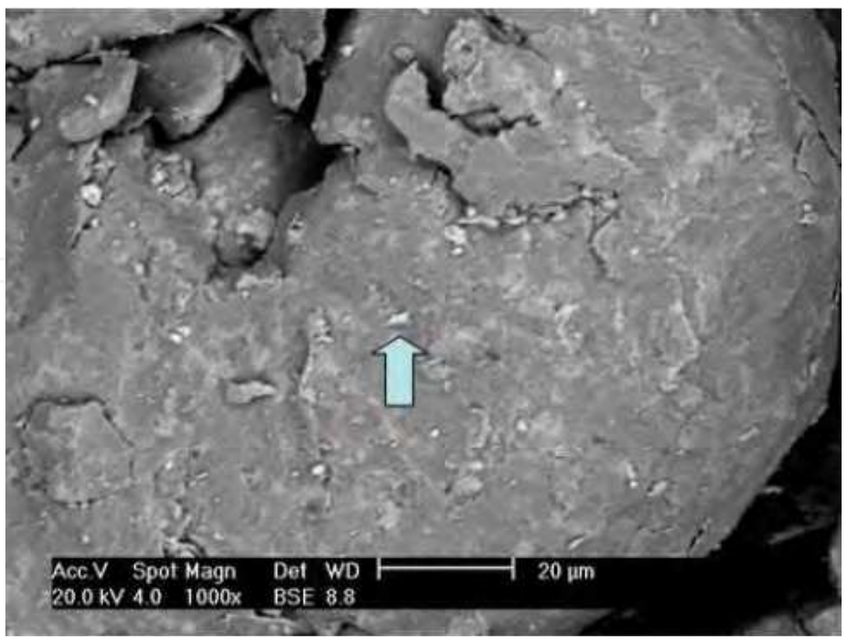

Fig. 8. Scanning electron micrographs of stainless steel powders reinforced with 3% of TaC from UFRN – 1000x.

Figures 9 and 10 present the scanning electron micrographs of samples with NbC-U and sintered for 30 minutes and 60 minutes, respectively.

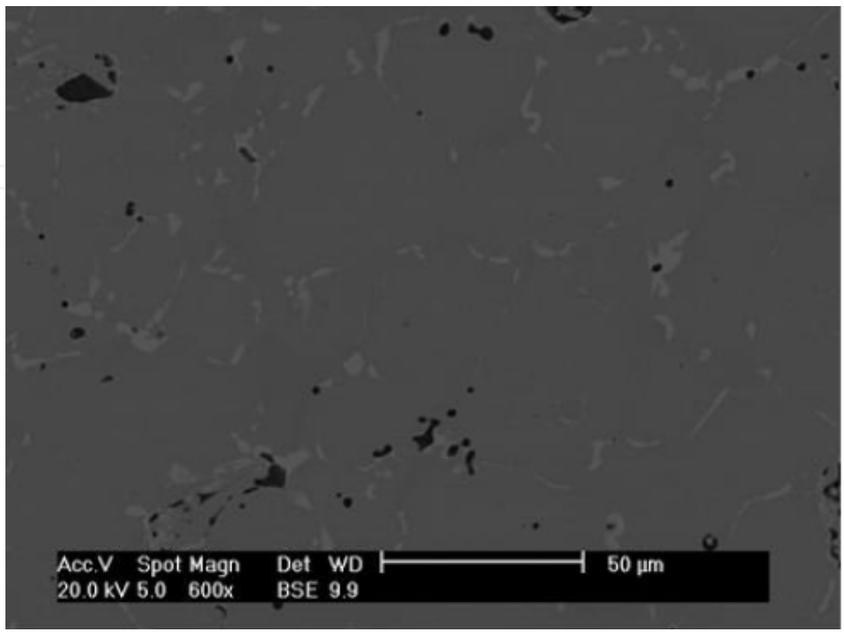

Fig. 9. Scanning electron micrographs of 316L stainless steel reinforced with 3% of NbC from UFRN sintered at 1290°C with isotherm of 30 minutes.

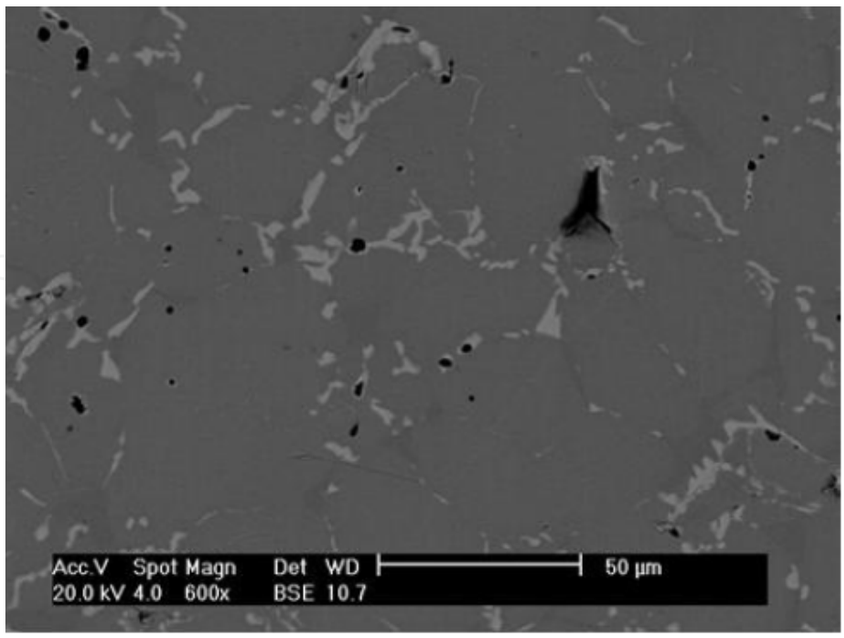

Fig. 10. Scanning electron micrographs of 316L stainless steel reinforced with 3% of NbC from UFRN sintered at 1290°C with isotherm of 60 minutes.

Figures 11 and 12 present the scanning electron micrographs of samples with TaC-U and sintered for 30 minutes and 60 minutes, respectively.

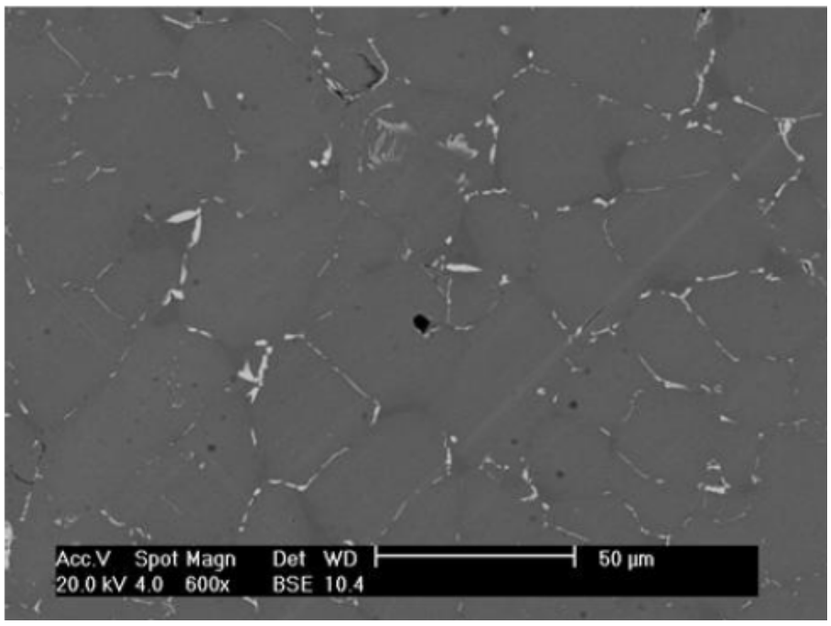

Fig. 11. Scanning electron micrographs of 316L stainless steel reinforced with 3% of TaC from UFRN sintered at 1290°C with isotherm of 30 minutes.

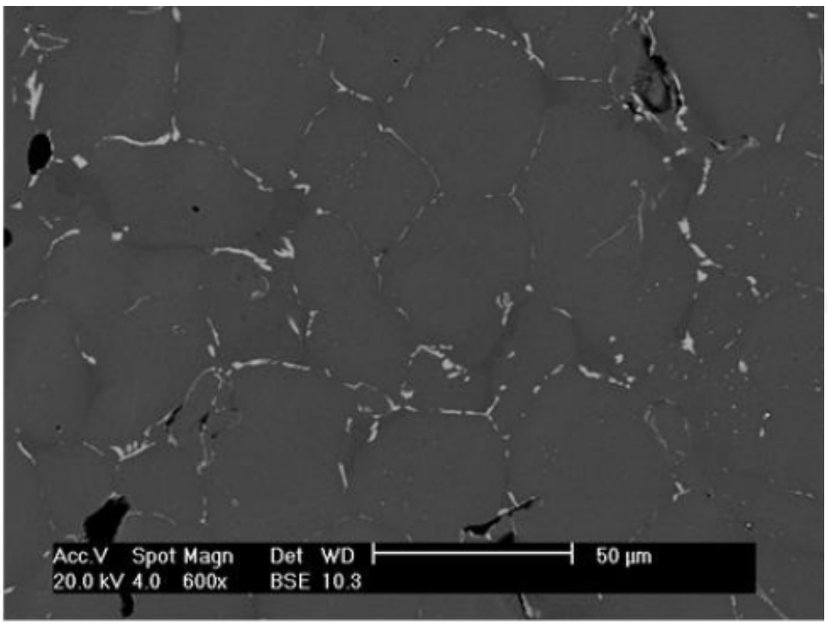

Fig. 12. Scanning electron micrographs of 316L stainless steel reinforced with 3% of TaC from UFRN sintered at 1290°C with isotherm of 60 minutes.

The table 2 presents values of densities and hardness. The carbides have influence on densification. Samples reinforced with NbC show higher densification. However, the great influence on the hardness values which increase from 76HV to 140 HV with addition of nanosized particles of NbC was observed.

Sample	Sintering time (min)	Density		Hardness (1000gf)
		(g/cm³)	(%)	
316L pure	30	7.10±0.07	89.0	76.0±5.2
316L pure	60	7.23±0.02	91.0	71.0±5.0
316L+NbC-U	30	7.53±0.05	94.6	133.0±8.3
316L+NbC-U	60	7.57±0.05	95.0	140±8.2
316L+TaC-U	30	7.57±0.07	93.0	94.0±9.8
316L+TaC-U	60	7.58±0.06	93.0	115.0±9.9

Table 2. Hardness and densities values from sintered samples.

7.3 Conclusions

The effects of nanosized particles of refractory carbides (NbC and TaC) on the sintering mechanism, denseness and hardness of stainless steel were investigated. The action of hardening mechanisms owed differences in fine particles size (10-20nm) results in great increase in the hardness values of the samples reinforced with carbides, especially for NbC.

The reinforced samples present very homogeneous composite raising the relative density to 93% for TaC and 95% for NbC. Great influence on the hardness that increase from 76.0 HV up to 115HV and even to 140HV, for the samples with nanosized reinforcement, was observed, owing to dispersion and precipitation of fine carbides in metallic matrix. The addition of carbides increases significantly the hardness of the stainless steel due to grain size reduction of the metallic matrix during sintering. Thus, the hardness does not increase only as a function of the density but mostly due to nanosized particles segregation on grain boundaries in the sintered microstructure.

8. Acknowledgment

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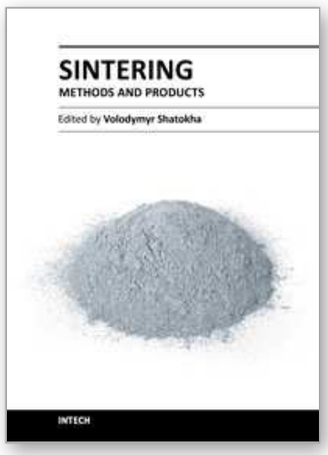

Sintering - Methods and Products

Edited by Dr. Volodymyr Shatokha

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This book is addressed to a large and multidisciplinary audience of researchers and students dealing with or interested in sintering. Though commonly known as a method for production of objects from fines or powders, sintering is a very complex physicochemical phenomenon. It is complex because it involves a number of phenomena exhibiting themselves in various heterogeneous material systems, in a wide temperature range, and in different physical states. It is multidisciplinary research area because understanding of sintering requires a broad knowledge - from solid state physics and fluid dynamics to thermodynamics and kinetics of chemical reactions. Finally, sintering is not only a phenomenon. As a material processing method, sintering embraces the wide group of technologies used to obtain such different products as for example iron ore agglomerate and luminescent powders. As a matter of fact, this publication is a rare opportunity to connect the researchers involved in different domains of sintering in a single book.

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