SINTERING OF HIGH SPEED STEELS CONTAINING NIOBIUM

Edval Gonçalves de Araújo
Fulvio Siciliano Jr.
Francisco Ambrózio Filho
Danilo A. Almeida Filho
Vladimir Araujo de Sousa
Mauricio D. M. das Neves

IPEN - CNEN / BRAZIL
DEPARTAMENTO DE METALURGIA - EPUSP / BRAZIL

ABSTRACT

High speed steels are known by their high hardness and wear resistance. This kind of steel usually has in its composition the element vanadium, which forms the hard phase VC. High speed steels produced by powder metallurgy show some technical and economical advantages over those produced by casting technique. Moreover, their vanadium contents are higher. This paper presents results using Nb, which has behavior similar to that of V.

An experimental study was carried out by adding niobium carbide (NbC) to AISI-M2 high speed water atomized powders in order to provide steels with different contents of Nb and C.

The processing conditions for the chosen compositions were studied by measuring the material densities after sintering and by microstructure analysis.

It was found that an increase in the carbon content results in an increase in the sinterability but also leads to a rapid increase in the grain size. The NbC added by simple powder mixture decreases the sinterability of high speed steels, the carbide are heterogeneously distributed in the structure. The mixture process by mechanical alloying results in a microstructure with carbide particles homogeneously distributed and low porosity in the sintered steel.

I. Introduction

Carbide segregation and grain growth are problems associated with high speed steels produced by conventional techniques, mainly in large transversal section parts. This leads to a product of unadequate characteristics which affect its performance. Large dimensional reductions are required during the hot working process in order to obtain the required properties. These are low efficiency processes (~50%) and they involves high costs.

The powder metallurgy of high speed steels are becoming increasingly important. The production costs can be cut down by reducing the loses of processing, improving the mechanical properties, reducing the number of processing steps involved in the fabrication of the finished product and by saving energy. New composites for tool steels which cannot be produced by conventional methods such as foundry and hot work, may also benefit from this technique [1]. The production of cutting tools and wear resistant materials are among the various applications of sintered high speed steels.

The material performance is directly related to the secondary hardening potential the carbide content, which is in the range of 7 to 13% in volume for the high speed steels, and to the relative hardness of the present phases, as shown in figure 1 [2].

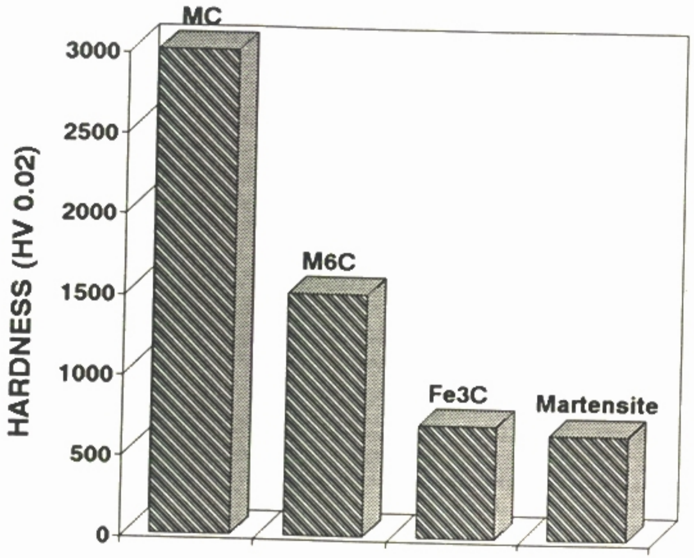

Fig. 1.Relative Hardness of the phases of high speed steels [2].

Concerning the microstructure, in order to obtain the best mechanical properties, the carbide particles must be fine and well distributed, and a continuous network around the grain should not be formed. The grain size and porosity level after sintering should be small. The heat treatment of sintered high speed steels, which confers the final properties to the finished product, are analogous to the one of the conventional steels [3,4,5].

It must be emphasized that a corrective heat treatment might be undertaken for the cases when inferior product final properties resulted from the heat treatment process. However, if the sintering step was responsible for introducing lower properties, nothing can be done to improve it. As an example porosities of 1 and 3% in volume reduce toughness, in terms of transverse rupture strenght (TRS) by more than 10% and 25%, respectively. Excessive temperature or sintering time also produce a drop in TRS between 10 and 25% depending on the overheating [6].

II. The High Speed Steel Sintering

The product prepared by powder metallurgy is affected by variables such as the chemical and physical characteristics of the powder. The techniques and pressures of consolidation and the sintering parameters: temperature, time and atmosphere also affects the powder metallurgy product.

During the sintering of high speed steels there is a temperature range, ± 3°C for the M2 [7,8], at which the microstructure constituents are fine and uniformly distributed, for small time intervals. Below this temperature range the density levels are low, even for long sintering periods. For higher temperatures or for relatively long time in this temperature range, there is an excessive grain growth, as shown in figure 2. Besides, carbide coalescence occurs. These carbide particles generally end up at the grain boundaries [4,9].

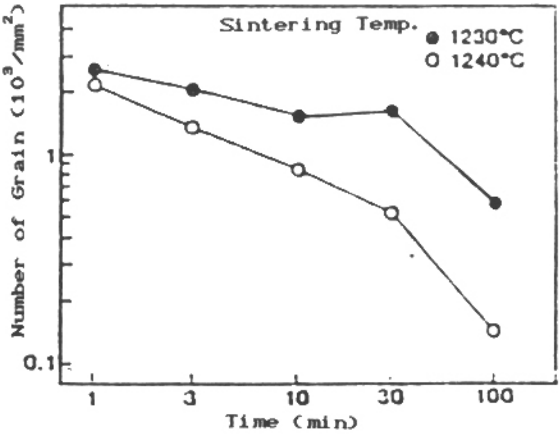

Fig. 2. Effect of sintering time on solid grain size [4].

The addition of graphite to the powder of high speed steel increases its sinterability due to the formation of eutectic phases of low melting point in the powder particles interface [6]. However, for large carbon contents there is a tendency for a decreasing density and the production of isolated pores of a relative large size in the sintered material [8].

The sintering mechanism is represented in figure 3. It is similar to others metallic systems where sintering occurs in the presence of a liquid phase [4].

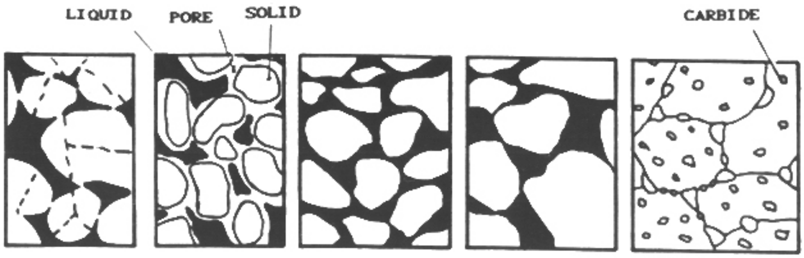

Fig.3. Mechanism of liquid phase sintering [4].

The particles are sintered in the solid state before the occurrence of the liquid phase, and form a skeleton. Metallographic evidences indicate that the carbide MC type are involved in the eutectic: austenite + carbide → liquid, but do not exclude the possibility of the reaction of MC carbide [10]. Thus, during the sintering a liquid film is formed which penetrates through the particles grain boundaries. The skeleton in consequently disintegrated in individual grains (supersolidus sintering), wich are rearranged and a rapid densification follows.

During the last sintering step, grain growth occurs probably by solution-reprecipitation, the transport of material taking place by difusion through the liquid [4]. During cooling, the liquid phase is decomposed in austenite and carbide. The carbide particles are mainly located in the grain boundaries and there is an aditional precipitation of carbide from the austenite which is characterized by fine carbide inside the grain.

III. The Effect of the addition of MC carbide

One purpose of primary carbide in the high speed steels is to sustain the austenitic grain boundary, impeding its growth during heat treatment. Regarding this the carbide type MC, which consist mainly of VC ou NbC, is more effective than the M_6C particles [11].

In the case of high speed steels produced by the conventional method, the niobium addition at contents above 3% wt, results in the formation of primary carbide. Theses carbides grow until the solidification of the eutectic, during this long period an overgrowth of carbide may take place, resulting in a drastic decrease of the wear resistance properties, the tougnesss and the cutting ability of the material. This limits the niobium content in billets of high speed steel in the range 1% to 1.5% in weight [11].

Karagoz and Fischmeister [11] explicited that only through powder metallurgy processes it is possible to use the whole potential of the niobium for the high speed steels.

The large volumetric addition of hard particles (above 20% in volume) such as carbides (NbC, TiC, VC, WC, SiC) [12], oxides (Al_2O_3) [13] or nitrides (CrN, NbN, TiN) [14] via powder metallurgy enhance the wear resistance of tool steels.

Figure 4 shows the best performance of niobium carbide relatively to the wear resistance for the matrix Fe-0.6%P-0.9%C with 10% in volumetric fraction for the various hard phases possible to the system [15].

Thumler and Gutsfeld [16] obtained, by mechanical alloying and sintering with liquid phase at 1280°C, a microstructure refinement proportional to the volumetric fraction of second phase particles (NbC or TiC) in a matrix of Fe-0.6%P-0.9%C. There was density increase of the sintered material due to NbC.

The mechanical alloying promotes the uniform distribution of carbide, hindering their transport to the grain boundary by the liquid phase and obstructing the grain growth during sintering.

The obtainment of a homogeneous microstructure of high carbide volumetric fraction, leads to an increase of the material wear resistance, as showed in figure 5, and also improves its mechanical properties [15].

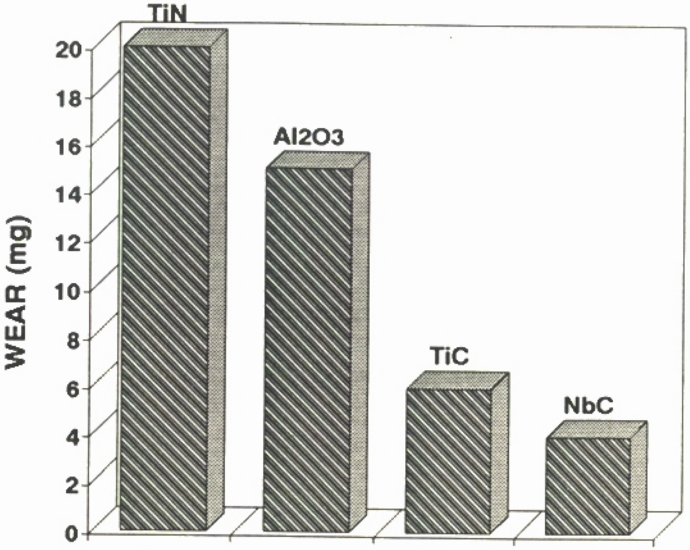

Fig. 4. Wear resistence for Fe-0.6%P-0.9%C matrix with 10% vol of hard phase [15].

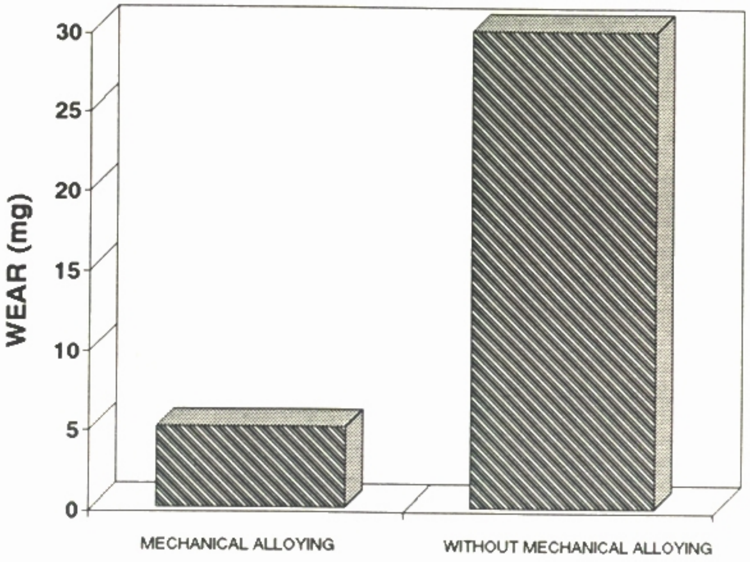

Fig. 5. Effect of processing via Mechanical Alloying in the wear for Fe-0.6%P-0.9%C-10% NbC alloy [15].

IV. Objectives

The main aim of this work is to develop a high speed sintered steel of high density with a M2 steel matrix and additions of niobium carbide. The procedure adopted points towards the attainment of a uniform and fine distribution of the microstructure constituents.

V. Material and Methods

The specification of the powder used in the present work are showed in Table I. The high speed M2 steel powder was water atomized and subsequently vacuum annealed.

	ELEMENTS (WT %)						
MATERIALS	C	Cr	Mo	V	W	O	N
M2	0.91	4.06	4.60	1.76	5.80	0.058	0.027
	$C_t^{\bullet}$	$C_l^{\bullet}$	Nb				
NbC	11.3	0.30	85.7				
Graphite	$C_t^{\bullet}$ 98.5						

M2 :	Theorical density	(g/cm^3):	8.32
	Aparent density	(g/cm^3):	2.65
	Vickers Hardness	(90g):	220
	Fischer Grain Size	(μm):	175
	Average Grain Size	(μm):	80.5
	Hall flow rate	(sec/50g):	32
NbC :	Aparent density	(g/cm^3):	3.4
	Fischer Grain Size	(μm):	2.6
	Theorical density	(g/cm^3):	7.78
Graphite:	Sieve Size	(μm):	< 38

• C_t = Total Carbon C_l = Free Carbon

Table I. Chemical and physical properties of M2 and NbC powders

The simple mixture, using 100 g of M2/C or M2/NbC, were carried out in a blender for 30 minutes. The specimens consisted of cylinders of 11.06 mm diameter and 3 mm height. These were compacted with zinc stearate lubrication only in the matrix.

The mechanical alloying of M2/15%NbC was undertaken in a Netzch ball mill attritor type MOD PE075, over heptane for 1 hour. The mixed powder was dried and annealed in hydrogen atmosphere for 1 hour at 900°C, subsequently it was cooled at 100°C/h up to 600°C, and at 180°C/min up to room temperature.

The sintering was carried out in a vacuum furnace. The specimen M2/15%NbC was also sintered in a hydrogen atmosphere of commercial grade inside a dilatometer.

Figure 6 shows the process variable studied to establish the effect of the carbon content and the volumetric fraction of NbC on the microstructure.

The optical microscope Neophot 30 with the Quantimet 520 image analyser - Cambridge Instruments and the scanning electron microscope with EDS were used for the microstructural characterization. The structures were developed by the selective etch with 45 ml of Nital 5% + 45ml of Picral 4% + 5 ml of concentrade HCl [17].

The green densities of the sintered material were geometrically measured. The sintered grain size was measured in the image analyser according to the standard ASTM E112.

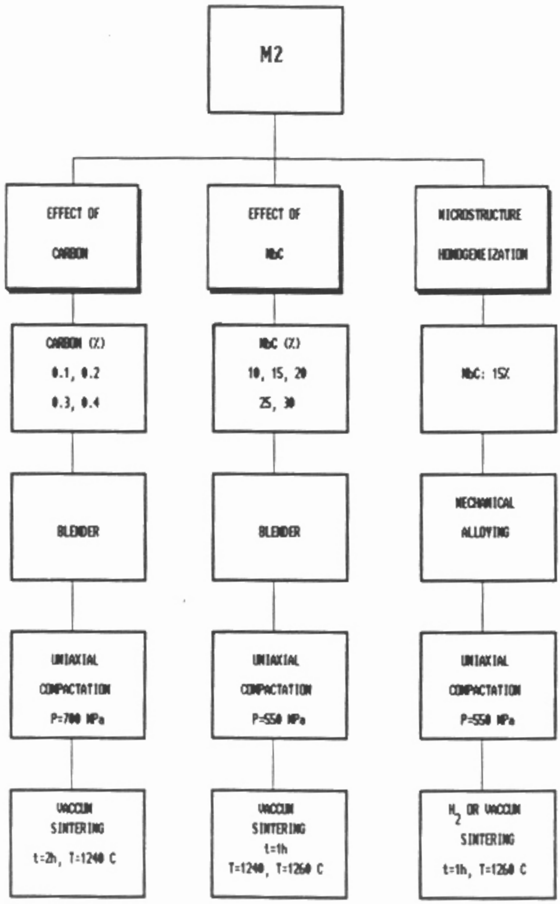

Fig. 6. Variable studied in the high speed steel sintering.

VI. Results and Discussion

The microstructural characterization of the powder used in this work is show in figure 7. Figure 7a shows the optical micrography of the high speed M2 steel powder particle. Figure 7b is a scanning electron micrograph showing cubic niobium carbides.

After sintering, the microstructure of the M2 material consisted of a martensitic matrix with a dispersion of MC and M_6C carbides, occorring mainly in the grain boundaries [7]. The M_6C carbides, light contrast phase showed in figure 8, are iron, tungsten and molibdenium rich, with the presence of some chromium and vanadium, as shown in figure 9a. The carbides type MC showed in figure 8 are basicaly vanadium carbides with a low iron, tungsten and molibdenium solubility as illustrated in figure 9b.

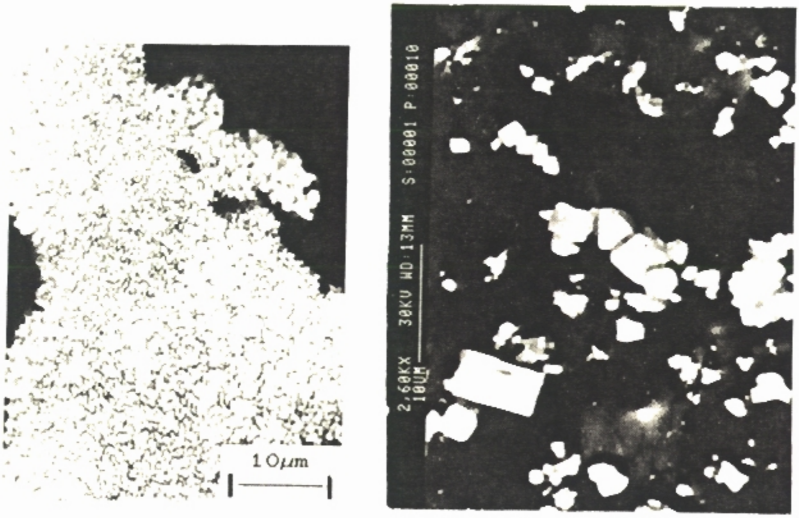

Fig. 7. (a) Microstructure of the high speed steel M2 powder. (b) Microstructure of NbC powder.

VI.1 - The Carbon Level Effect

The effective sintered densification can be achieved by a raise in the carbon level [3]. Part of the added carbon reacts with the dissolved oxygen present in the powder or from the sintering atmosphere [6].

The sintering of the high speed steel M2 can be explained by the following facts:

1. For each 1% of carbon added there is a decrease of 111°C in the solidus temperature, and it also lowers the system reaction temperature as shown in figure 10 [2].
2. There is the formation of approximately 15% of liquid, at the temperature range 1240°C to 1245°C for the M2 [2], as shown in figure 11.

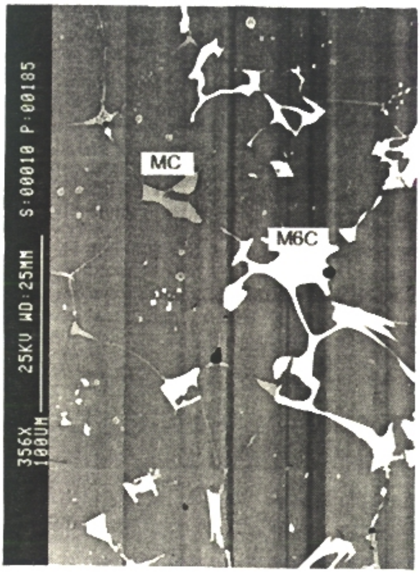

Fig. 8. Microstructure of sintered high speed steel M2 (MEV).

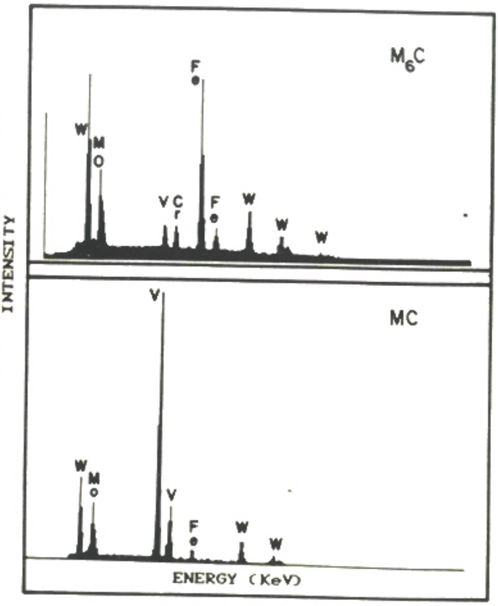

Fig. 9. EDS analisys of the high speed steel M2 carbides.

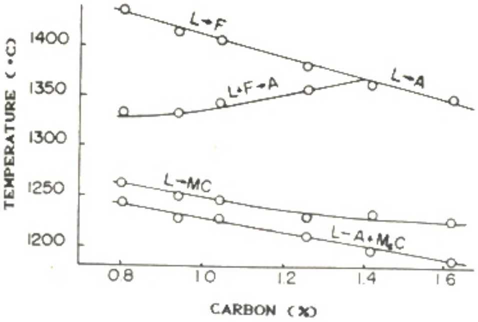

Fig. 10. Reaction temperature versus carbon level for high speed steel [2].

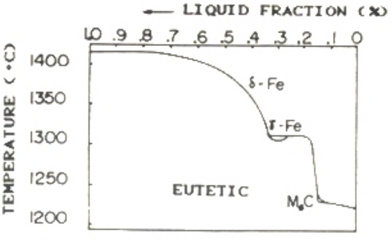

Fig. 11. Volumetric liquid fraction versus temperature [2].

Figure 12 shows a porosity free structure from 0.2% of carbon excess. Grain growth and carbide coalescence also occur. This is due to the enhancement of the diffusion mechanism by the great amount of liquid phase formed. Takajo [4] reported that the liquid amount to cause full density in the M2 in approximately 5%.

Figure 13 shows the grain growth as a function of the carbon level. There is some evidence that for carbon level below 0.1% the formation of liquid phase is not enough for densification. Above 0.2%C there is a linear grain growth. This supports the fact that the grain growth is a direct function of the liquid phase amount.

VI. 2 - The Niobium Carbide Effect

Some iron ternary systems produce low melting point phase reactions, many of which with carbides. For the high speed steel M2, it is more likely that the liquid phase be formed from the preferential dissolution of intergranular M_6C carbides [7]. Though, if niobium carbides, a low matrix solubility phase, are added by a simple mixture, this NbC is heterogeneously distributed in the structure and there is a decrease in the sinterability by using more then 10% NbC, as shown in figure 14. A raise in the sintering temperature promotes a better densification as shown in figure 15.

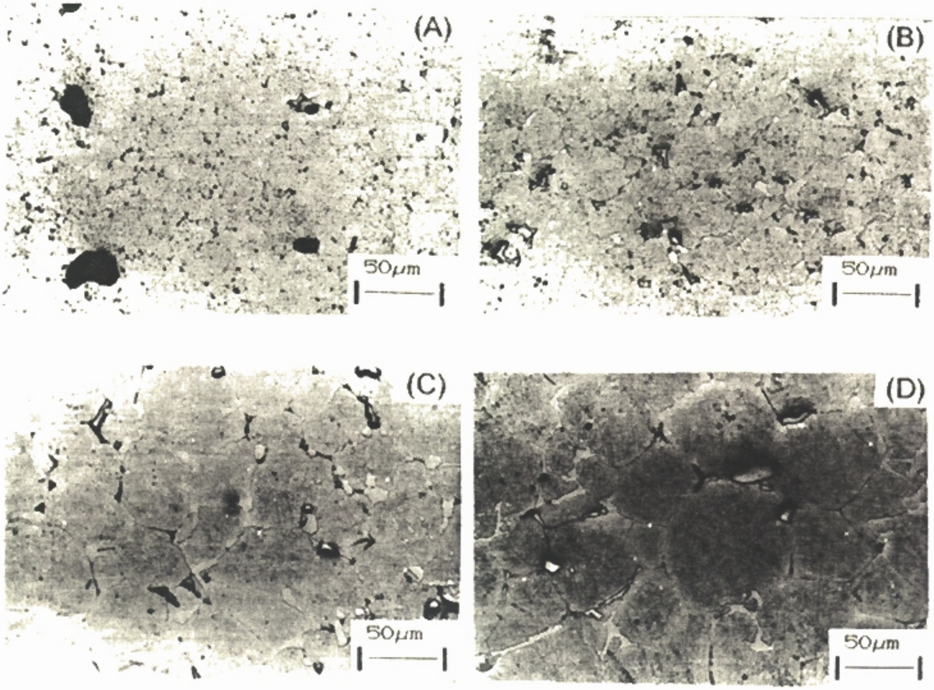

Fig. 12. High speed steel M2 microstructure for carbon additions: (a) 0.1%C, (b) 0.2%C, (c) 0.3%C e (d) 0.4%C.

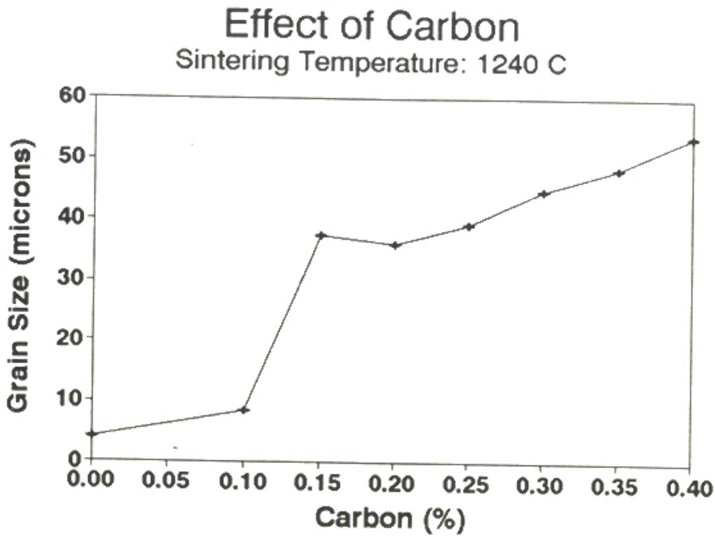

Fig. 13. Effect of carbon addition on grain size for high speed steel M2.

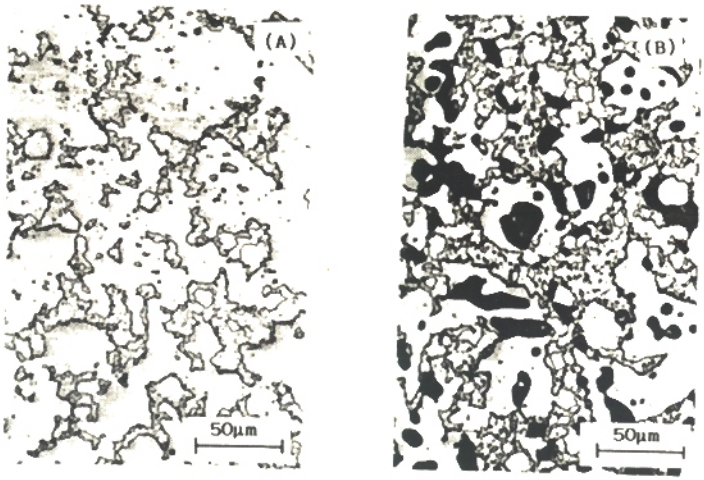

Fig. 14. Microstructure of (a) M2-10% NbC (blender) and (b) M2-30% NbC (blender).

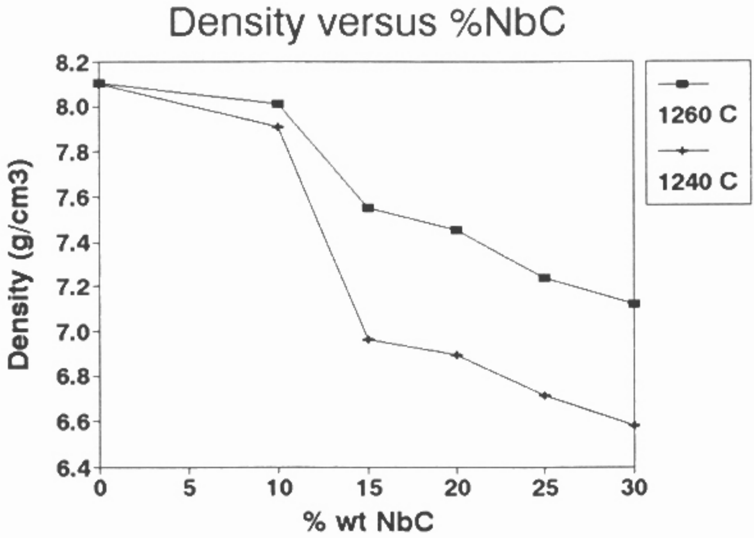

Fig. 15. Density versus %NbC for 1240°C and 1260°C.

A homogeneous microstructure and lower porosity was obtained by mixing the powders via mechanicall alloying, as shown by the micrographies in figure 16.

The mechanical alloying process besides of deaglomerating the NbC powder, it also promotes a uniform distribution of carbides in the matrix. This implies in:

1. a decrease of the starting liquid phase formation temperature due to the action of 0.3% of free carbon from the NbC.
2. preventing grain growth due to grain boundary pinning by the NbC. Regarding the sintering atmosphere, it seems that vacuum is more efficient than hydrogen, decreasing the porosity level as can be verified in the microstructures shown in figure 16.

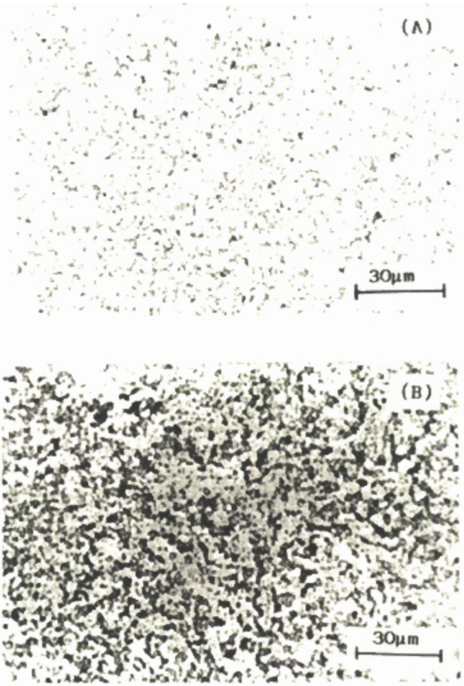

Fig.16. Microstructure of M2-15%NbC (Mechanical Alloying)
(a) Vacuum, (b) Hydrogen

VII. Conclusions

1. The carbon addition promotes a better sinterability of the high speed steel M2. However, grain growth speed up due to a liquid phase amount increase;
2. The NbC addition by simple mixture reduces the densification and it is not effective in carbide distribution through the matrix.
3. As far as mechanical alloying is concerned:
3.1- This process showed to be efficient in terms of microstructure homegenization.
3.2- This method allowed the introduction of high amounts of NbC, and consequently the obtention of high densification at the normal sintering temperature of the high speed steel M2.
3.3- For the composite material M2/15%NbC, the sintering showed to be effective under hydrogen atmosphere and under vacuum.

VIII. Acknowledgments

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