

Sintering of AISI M₂ high speed steel with the addition of NbC

Alexandre Wentzcovitch¹, Francisco Ambrozio Filho¹, Luis Carlos Elias da Silva², Maurício David Martins das Neves²

¹Centro Universitário da FEI

²Instituto de Pesquisas Energéticas e Nucleares – IPEN-CNEN/SP

alexandrew_@hotmail.com; ambrozio@fei.edu.br; lcesilva@ipen.br; mdneves@ipen.br

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Abstract. *The influence of adding 6 wt% (NbC) niobium carbide on the sintering temperature and microstructure of high speed steel - AISI M₂ (0.87% C, 5.00% Mo, 6.00% W, 4.00% Cr, 2.00% V and Fe bal.) powder was studied. The starting material was obtained by vacuum melting followed by atomization in water. The samples were axially cold compacted in a cylindrical matrix and then vacuum sintered at 1250 and 1350 °C. Dilatometry and differential scanning calorimetry measurements indicated an increase in sintering temperature with addition of niobium to the AISI M₂ steel. Optical and scanning electron microscope observations revealed a microstructure with uniformly distributed niobium carbides.*

Introduction

High-speed steels have been widely used in the manufacture of cutting tools and wear-resistant materials [1]. Several techniques have been used to improve the properties of sintered high speed steels and these include: addition of alloying elements to increase carbide formation [2, 3], addition of ceramic reinforcements and use of high-energy milling [4].

Niobium is an alloying element that can be added to high speed steels to form stable carbides and provide reinforcement to the matrix. The niobium added to the steel combines with carbon to form a MC-type carbide, which can increase the hardness and wear resistance of the high speed steel and prevent austenite grain growth during sintering and heat treatment. Another factor in favor of adding niobium is possibility to replace other alloying elements like vanadium [2].

Sintering of niobium containing high speed steels should be performed with liquid phase formation. Hence, it should be carried out above the solidus temperature and below the liquidus temperature (supersolidus liquid phase sintering) to increase densification. Sintering below the solidus temperature is inefficient and the sintered product has lower density besides containing a large number of pores.

Studies related to niobium containing AISI M₂ high-speed steel have indicated the formation of primary MC type carbide that consisted largely of a mixture of niobium and vanadium carbides [1,3,5]. Other studies have shown increased wear resistance and cutting performance of niobium containing steels compared to niobium free AISI M₂ steel [5].

The objective of this investigation was to study the influence of adding 6 wt % NbC to AISI M₂ high-speed steel powder on its sintering temperature.

Materials and Methods

The starting material used in this study was obtained by vacuum melting an alloy with composition similar to AISI M₂ high speed steel but with the addition of 6 wt% (in weight) NbC. The chemical composition of the cast ingot and the steel AISI M₂ are shown in Table 1. The ingot was water atomized at the Institute for Technological Research (IPT). Atomization of 18 kg of steel was carried out using a nozzle 10 mm in diameter, pressure of 1200 psi and at 1610 °C.

Table 1: Chemical composition of the cast alloy and AISI M₂ steel.

Element wt%	Fe	C	Nb	Mo	W	Cr	V
Ingot	74.7	1.34	5.7	4.39	5.22	5.03	1.74
AISI M₂	Bal.	0.87	-	5.00	6.00	4.00	2.00

The powder was sieved to separate particles less than 150 μm (100 mesh). Subsequently the apparent density and flow characteristics of the powders were determined, the latter by the 'Hall Funnel' method. ASTM B-121 standard test method was used to determine the apparent density of free-flowing powders. The geometric density (*d*) was determined from mass (*m*) / volume (*v*).

The hydrogen loss test was performed with a sample of atomized powder. This consisted of leaving a powder sample in hydrogen atmosphere for 60 minutes at 800 °C followed by determination of mass change.

Differential Scanning Calorimetric analysis (DSC) and dilatometry were carried out using a Setsystem apparatus at the University Center of FEI. The DSC test was performed up to 1400 °C while the dilatometry test was carried out till 1300 °C for 1 hour.

The atomized powders of AISI M₂ high speed steel with niobium was axially compacted at 700 MPa. The powders could be compacted without annealing or addition of a binder. The green density was estimated from the mass / volume ratio of the sample. Sintering of the samples was carried out in vacuum of 1.10⁻⁵ torr for 1 hour at 1250 °C and 1350 °C. Sintering was performed at IPEN. The geometric densities of the sintered samples were calculated.

Standard metallographic techniques were used to prepare samples for microstructure characterization and Vickers hardness measurements, the latter with a Shimadzu HMV-2 at the University Center of FEI. Figure 1 shows a flowchart of the methodology used to prepare and characterize the Nb containing steels.

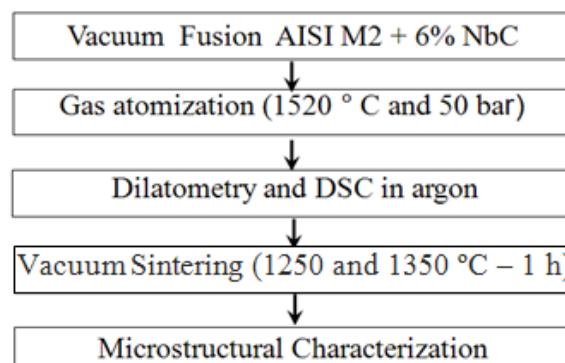


Figure 1: Flowchart of the manufacturing process.

The composition of the matrix and the carbide were determined semi-quantitatively with the help of a scanning electron microscope equipped with an energy dispersive spectrometer (EDS). The microstructure examination was carried out at IPEN using a Philips XL30 SEM and an Olympus BX 60M optical microscope. Table 2 shows the main properties of the atomized powder.

Table 2: Properties of water atomized AISI M₂ + NbC powder.

Property	
Theoretical density [g/cm ³]	8.321
Particle size [μm]	< 150 (-100 # mesh)
Hardness – HV 0.1	558.2 ± 24.2
Apparent density – [g/cm ³]	3.098 ± 0.008
Flowability [s/50 g]	27.39 ± 0.16
Hydrogen loss [%]	1.51

Results and Discussion

Figure 2 shows the equilibrium diagram of M₂ steel + 6% NbC as determined with Thermo-Calc. The term FCC_A1 # 2 is the MC type carbide (niobium or vanadium), BCC_A2 the ferrite phase and FCC_A1 # 1 the austenitic phase. The first solid phase is the niobium rich primary MC type carbide.

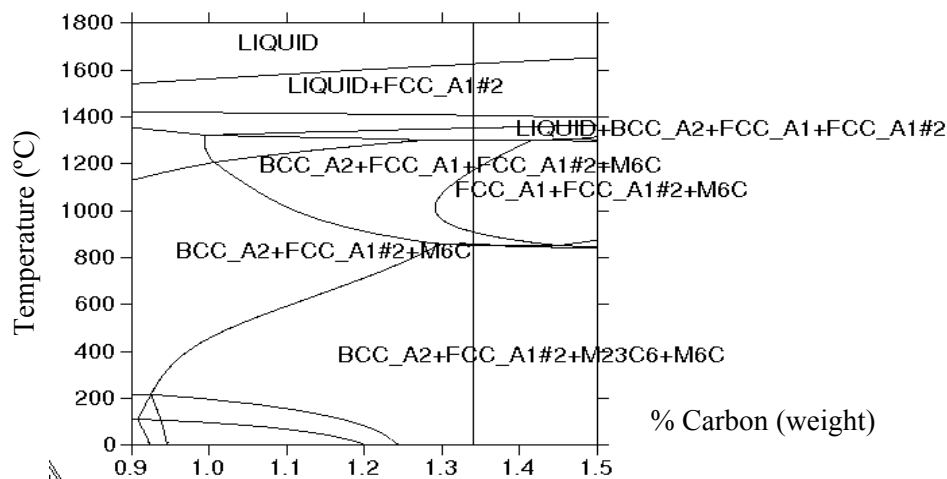


Figure 2: Phase diagram obtained with a ThermoCalc software.

Figure 3 shows the curves obtained by DSC during heating (fig. 3a) and cooling (fig. 3b). During the heating stage the beginning of a ferrite to austenite transformation can be observed around 725 °C. Above this temperature, the curve is diffuse due to dissolution of carbides. Close to 1240 °C, melting started and the amount of liquid increased gradually due to melting of the austenitic phase and the carbides. In the cooling curve, solidification of the liquid phase started with the MC carbide after appearance of a solid matrix (austenitic phase), formation of M₆C carbide and then nucleation of the martensite phase.

The dilatometry test results are shown in Figure 4. A pellet 6 mm in diameter and 2 mm high was used. The dilatometry curve at 330 °C indicates accommodation of the particles. Above this temperature the sample dilated and at 1150 °C an inflection in the curve can be observed that is due to contraction of the sample, indicating the beginning of sintering. Residence at 1300 °C for 1 hour revealed dimensional changes in the height of the sample. There is a contraction of about 4% in the temperature of 1300 °C.

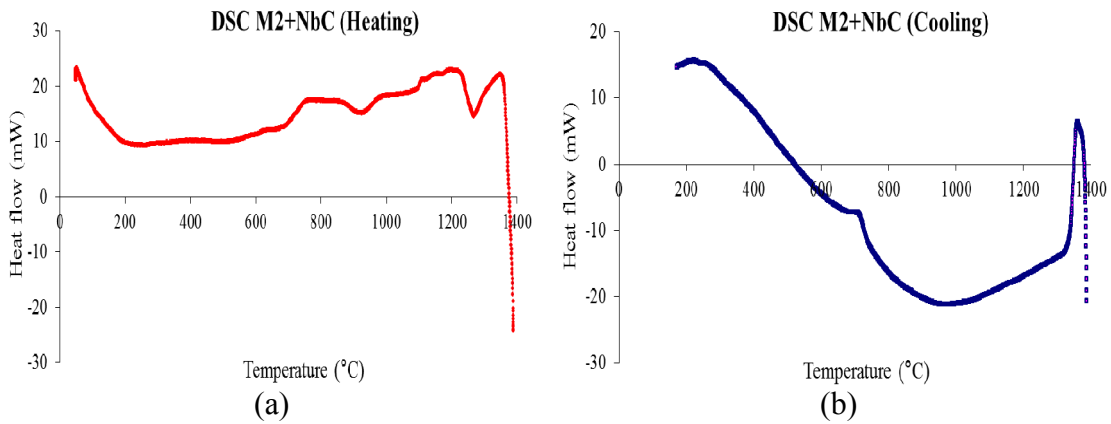


Figure 3: Differential scanning calorimetry (DSC) curves (a) heating and (b) cooling.

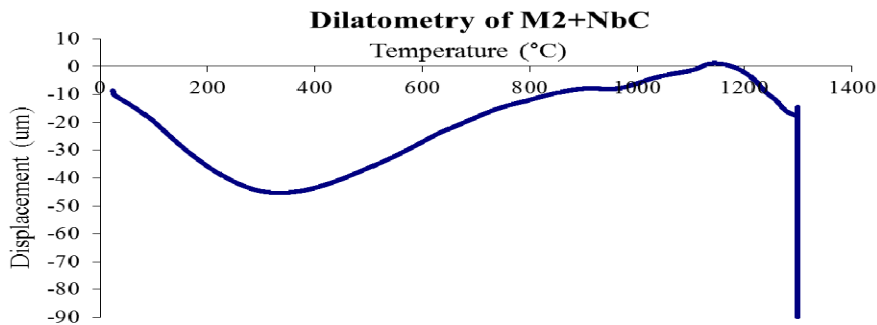


Figure 4: Dilatometry curve.

The change in mass of the sample during heating in the dilatometry test is shown in figure 5. The mass loss of the sample was 0.5%. This loss was due to deoxidation of the particles caused by the presence of carbon. Maximum mass loss occurred above 1100 °C and is related to the onset of the sintering process and deoxidation of the surface. The same trend occurred up to 1400 °C, as shown in Figure 5. Deoxidation of the surface of the particles occurred as a result of carbon diffusing from the solid solution to the surface, reduction of the surface oxides and generation CO gas.

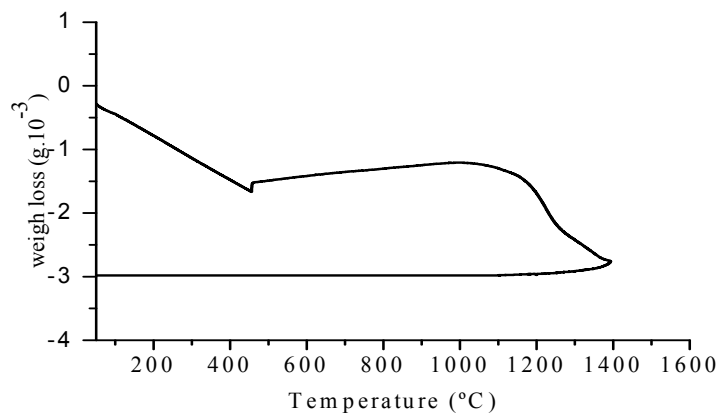


Figure 5: Weight loss during heating in the dilatometry test X temperature.

The theoretical density of AISI M₂ was 99.0 % at sintering temperature of 1250 °C, while that of the AISI M₂ + NbC steel was 85.0% at the same temperature. At 1350 °C the theoretical density of AISI M₂ + NbC was 95.0%. The presence of Nb in AISI M₂ high speed steel increases the sintering temperature. The density of this sintered steel is lower compared to that of AISI M₂, as shown in Table 2.

Table 2: Density of sintered steel and theoretical density (T.D.) as a function of sintering temperature.

Temperature [°C]	Green Density [g/cm ³]	sintered density [g/cm ³]	T. D. [%]
1250°C-AISI M ₂	6.71±0.04	8.01±0.10	99.0
1250°C-AISI M ₂ +NbC	5.60±0.06	7.21±0.15	85.0
1250°C-AISI M ₂ +NbC	5.59±0.05	7.97±0.20	95.0

Optical and scanning electron micrographs are shown in figures 6 and 7 respectively. AISI M₂ is free of pores at 1250 °C as shown in figure 6a. Figure 6b shows pores and a small amount of liquid phase indicating inadequate sintering of AISI M₂+NbC at 1250 °C. Figures 6c, 6d, 7a and 7b (1350 °C) reveal the presence of the liquid phase at the grain boundaries mainly, a pore free microstructure and a homogeneous distribution of carbides. The distribution of carbides was homogeneous but in some regions, the concentration of carbides was higher.

The sintering of high speed steels with addition of niobium occurs by supersolidus sintering. Sintering kinetics are enhanced in the presence of the liquid phase formed during sintering. Hoyle [1] reported that 5% is the amount of liquid phase necessary to achieve the theoretical density in AISI M₂ and metallographic evidence suggests that this liquid phase is obtained from the eutectic reaction: austenite + carbides (MC and M₆C) → liquid. Similar behavior was found in the alloy AISI M₂ + 6NbC.

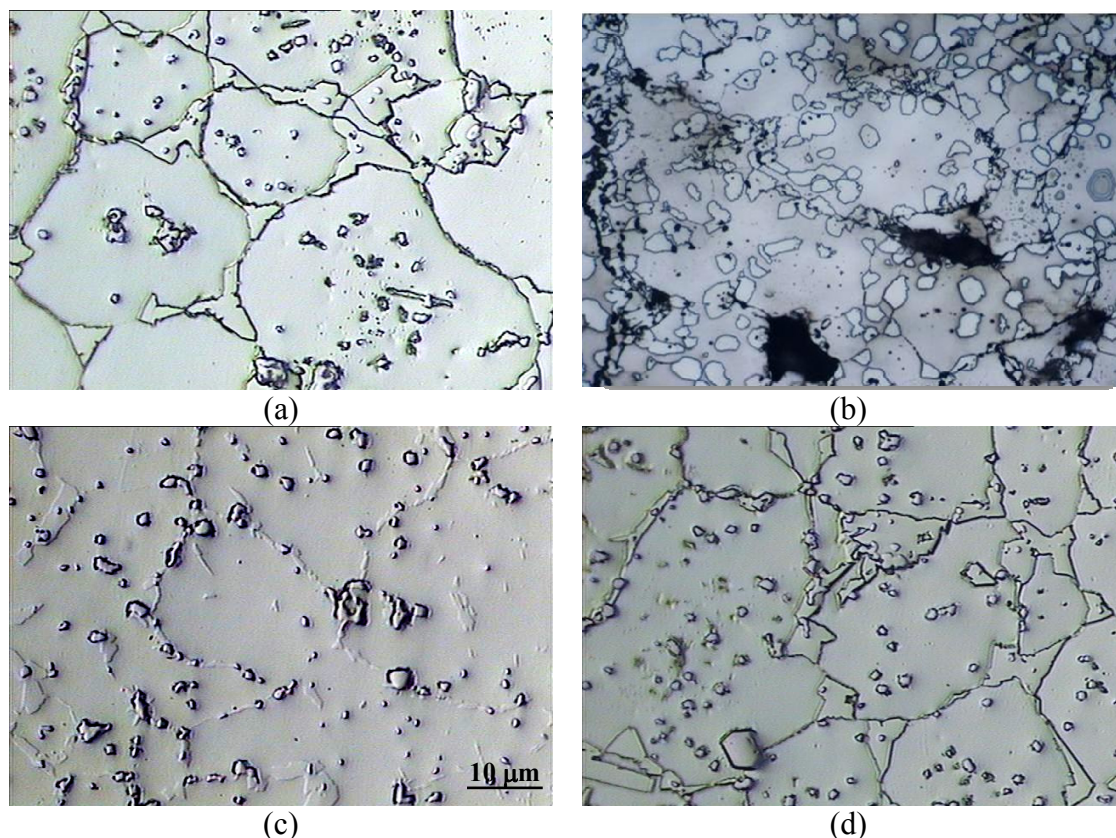


Figure 6: Optical micrographs of: (a) AISI M₂-1250 °C, (b) AISI M₂+NbC -1250 °C, (c) AISI M₂+NbC-1350 °C (no etching) and (d) AISI M₂+NbC-1350 °C.

At the beginning of sintering of AISI M₂+6 NbC, solid-state diffusion takes place. With increasing temperature until the interval between the solidus and liquidus lines there is formation of a liquid film that penetrates the particle and grain boundaries, breaking

down the structure into individual grains. These grains rearrange themselves due to capillary phenomena favoring densification. In the final stages, the atoms go into solution and then precipitate at regions with lower chemical potential. Material transport occurs by diffusion of the liquid that evolves and causes pores to close. During cooling, the liquid phase decomposes into austenite and MC carbides. The carbides are located mainly at grain boundaries. This is followed by additional precipitation of carbides M_6C and MC from the austenite and these are as fine carbides within the grains [1, 2]. Figure 7 reveals the large carbides with higher niobium content at the grain boundary and the smaller carbides, also with higher Nb content within the grain.

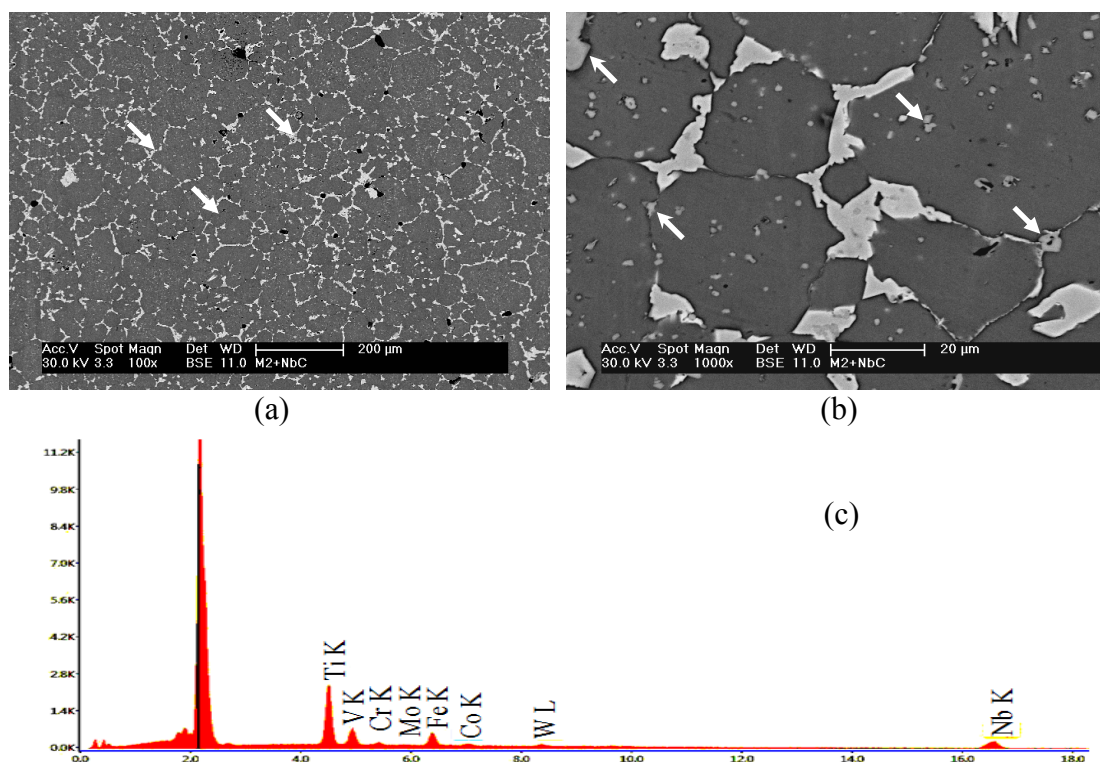


Figure 7: (a) and (b) Scanning electron micrographs and (c) EDS of NbC.

Conclusions

- The sintering temperature of the alloy AISI $M_2+6\%$ NbC increased to 1350 ° C compared to the Nb free steel.
- The microstructures revealed homogeneous distribution of carbides including those with higher niobium content.
- Large carbides, including those with higher niobium content are located at the grain boundaries.

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