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Microstructural Evaluation of AISI T-15 High Speed Steel

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Abstract A metallographic study using optical-electronic techniques was carried out on commercial high speed steel AISI T-15, obtained by hot isostatic pressing. The steel was studied in the as-received condition and after quenching from 1160 °C and 1210 °C, followed by tempering at 540°C and 560 °C. Selective metallographic etching was optimized to reveal austenitic grains and MC as well as M_6C type carbides. The average grain size of austenite and the volume fraction, size distribution and semi-quantitative chemical composition of the carbides were determined. The austenite grain size, determined by the intercept method of Snyder-Graff, the semi-automatic Zeiss Quantimet method and the Quantikov method using digitized images, all presented similar results. In carbide content determination, the Quantikov method proved to be simple and appropriate, compared to the Zeiss Quantimet method.

1. Introduction

High speed steels used in cutting tools are iron alloys containing carbides of tungsten, molybdenum, vanadium and chromium. During their solidification, different types of carbides can form, and the more important ones are: M_6C, M_2C and MC, where M is the metal. The presence of these carbides depends on the cooling rate and the concentration of the different elements in the steel [1]. The presence of these carbides influences considerably, the properties of these steels in two ways [1-4]. Firstly, partial dissolution of the carbides in the matrix, during austenizing, influences properties that depend on matrix composition, like hot hardness. Secondly, the carbides themselves influence the mechanical properties, especially, wear resistance.

Ghomashchi's studies [4] show that the M_2C type primary carbides are metastable and transform to M_6C and MC type carbides in the austenite temperature range.

The time-temperature combinations used in quenching and tempering treatments of these steels, influence the microstructure and consequently, the properties [5]. In this context, some important microstructural aspects such as austenite grain size and type, volume fraction as well as size distribution of the carbides require evaluation.

In this paper, selective metallographic etching procedures, for both austenite grains and carbides have been defined. The microstructures were studied using a variety of metallographic techniques, optical-electronic image analysis and energy dispersive X-ray microanalysis.

2. Materials and Experimental Procedures

In this investigation a commercial AISI T-15 type high speed steel from "Crucible Materials Corporation", and obtained by hot isostatic pressing was used. The temperature and pressure normally used are of the order of 1100 °C and 100 MPa, respectively.

The chemical composition of the steel in weight percent, as per the manufacturer, is shown in Table 1.

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Table 1. Chemical composition of T-15 steel (in weight %)

C	W	Mo	Cr	V	Co	Si	Mn	P	S
1.60	11.95	0.72	4.06	4.66	4.87	0.33	0.33	0.017	0.06

To evaluate the microstructure as a function of heat treatments, the specimens were quenched (austenitizing) at 1160 °C and 1210 °C. These were then triple tempered in a salt bath for 3 x 3600 seconds. Specimens austenitized at 1160 °C were tempered at 540 °C and those austenitized at 1210 °C, at 540 °C and 560 °C.

After the heat treatments, the specimens were prepared for metallography and polished down to 1μm with diamond paste.

The main analytical techniques used for microstructural characterization were: optical and scanning electron microscopy, energy dispersive X-ray microanalysis, Zeiss semi-automatic "Quantimet" image analysis, and "Quantikov" analysis, developed on Windows platform.

2.1.Determination of grain size

Initially, the specimens were studied by optical microscopy and by scanning electron microscopy, to evaluate well the microstructure of the grains, using the following three different methods for grain size determination:

-"Snyder Graff" intercept method [6]

-"Quantimet" method

-"Quantikov" method [7]

To determine the grain size by the "Snyder-Graf" method, the specimen is observed at 1000x magnification, and a circle with a perimeter of 127 mm is highlighted. The number of grains intercepted by the line is counted, and the average of 10 such measurements corresponds to the "Snyder-Graf" (SG) grain size. To convert the SG grain size into average intercept length, L, SG is multiplied by 7.874, to obtain the number of intercepts per millimeter, N_L, and the equation $L = 1/N_L \times 1000$ gives the value in micrometers (μm).

In the "Quantimet" method a semi-automatic particle size analyser, Carl Zeiss, coupled to an optical microscope enables the size distribution measurements to be carried out on etched specimens. The size determination is based on equivalent diameter of a circle with the same area as that of the object being measured.

The "Quantikov" method was designed to integrate the capabilities of a "Windows" platform for microstructural image analysis, aimed at automatizing the process to quantify microparticles from the digitized images obtained with a "scanner". This capability permits the grain boundaries and/or carbides, transported manually by the researcher from the respective micrographs to the transparencies, to be recorded by a "scanner" and analysed. The **quanti**fication of microparticles by applying the "Salti**kov**" method, gave rise to the name "Quantikov".

In all methods, about 250 grains were measured, corresponding to 5 different regions in each specimen to obtain statistically, representative results.

2.2. Determination of Carbides

Optical microscopy and scanning electron microscopy were again used to determine the number of carbides per unit area. In optical microscopy, magnifications of the order of 1500 and 2000x were used. In scanning electron microscopy magnifications of the order of 1500, 2000 and 7000x were used. As in the earlier case, five different areas were evaluated by the "Quantikov" and "Quantimet" methods. Use of the backscattered electron image (composition image) made it possible to distinguish the M_6C (white) from the MC (grey) carbides, and this enabled subsequent image analysis by the "Quantikov" method.

Microanalysis by energy dispersive analysis of X-rays was carried out in a "Jeol" scanning electron microscope coupled to a "Noran" analysis system. During semi-quantitative identification of the carbides, care was taken and only carbides in the size range of 3μm were analysed, to avoid the interference of the matrix.

3. Results and Discussion

3.1. Selected etchants

The etchants listed below were tested, depending on the microstructural component to be revealed:

-Etchant (a) - nital with 5% nitric acid in ethyl alcohol,
-Etchant (b) - chromic acid solution, obtained by dissolving 20g chromium trioxide in 100ml of distilled water,
-Etchant (c) - a solution of 10% sodium hydroxide and 1% tartaric acid in 100ml of distilled water.

Etchant (a) was used to reveal the whole microstructure (grains and carbides) without differentiating any specific microconstituent, Fig. 1. This etchant was also used for the first etch, in a double etching process to differentiate the carbides, with etch times between 3-4 minutes, Fig. 2. Finally, this etchant was used at a temperature of approximately 70°C, to reveal all the carbides present, without differentiation, Fig. 3 (a)

Etchant (b) was used as an electrolytic etchant at 2-3V, to reveal preferentially the MC type carbides, making them dark while maintaing unaltered, the M_6C type carbides, Fig. 4.

Etchant (c) was used as an electrolytic etchant at approximately 50mV, and was used to selectively etch the M_6C type carbides, Fig. 5.

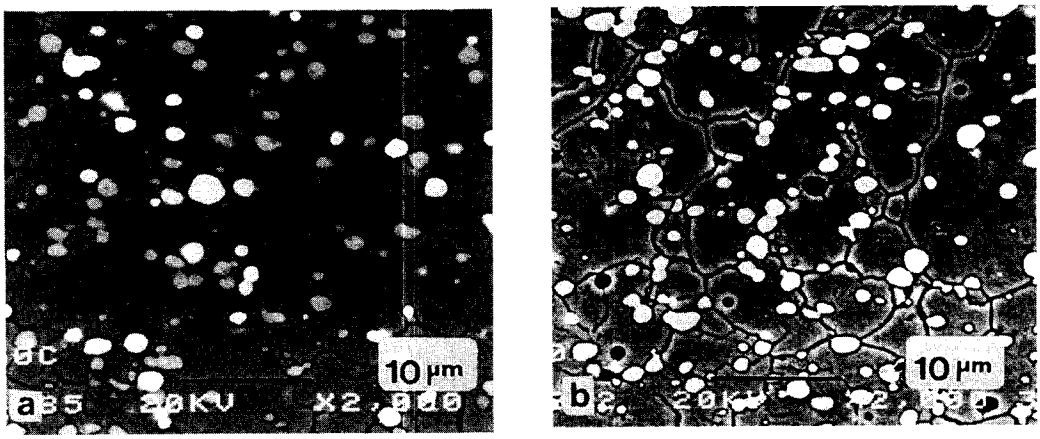

Fig.1: Scanning electron micrographs of AISI T-15 steel after quenching: a) 1160 °C; b)1210 °C. Etchant: nital 5 %.

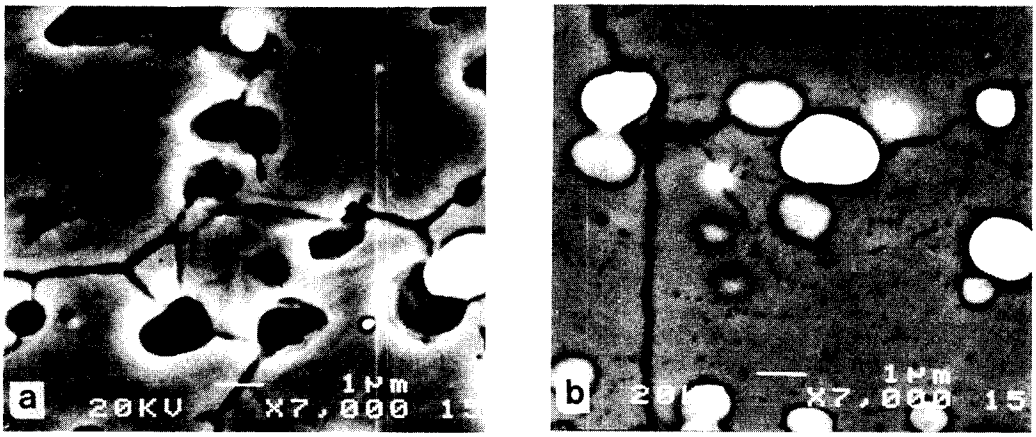

Fig. 2: Scanning electron micrographs of AISI T-15 steel after quenching: a) 1160 °C; double etching: 5 % nital followed by electrolytic etch with chromic acid solution; b) 1210 °C; double etching: 5 % nital followed by electrolytic etch in a solution of 10 % NaOH and 1 % tartaric acid.

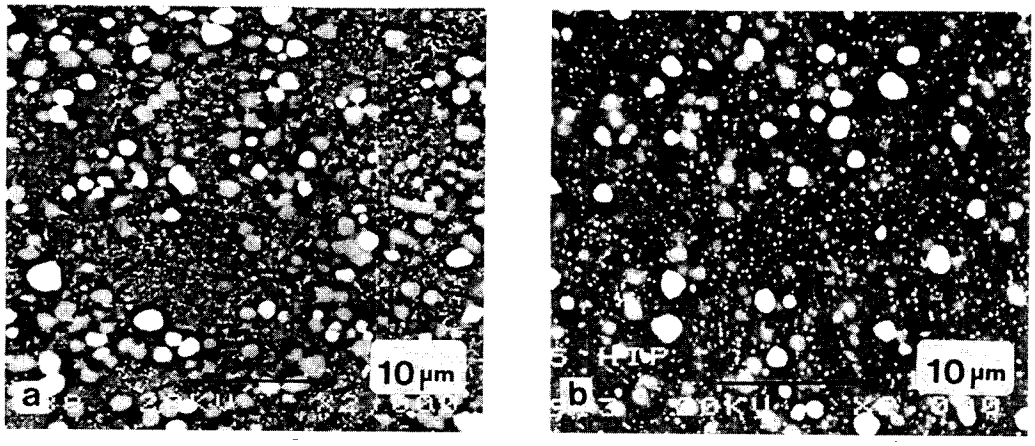

Fig. 3: Scanning electron micrographs of AISI T-15 steel: a) as received. Etchant: 5 % nital (70 °C); b) backscattered electron image (unetched specimen).

3.2. Average austenite grain size

A number of authors have reported that the austenite grain size varied directly with the average size of the primary carbides and that the presence of the carbides along the grain boundaries hindered austenite grain growth. This behavior can be observed in both the specimens quenched from 1160 °C and 1210 °C, Fig. 1 (a and b). The metallographic etch did not reveal all the grain boundaries. Some grains with incomplete boundaries can be seen in Fig. 1. In this investigation, only grains with well defined boundaries were considered.

After the metallographic etch with nital (etchant a), the average size of the original austenite grains in specimens quenched from 1160 °C and 1210 °C was measured, and the results are shown in Table 2.

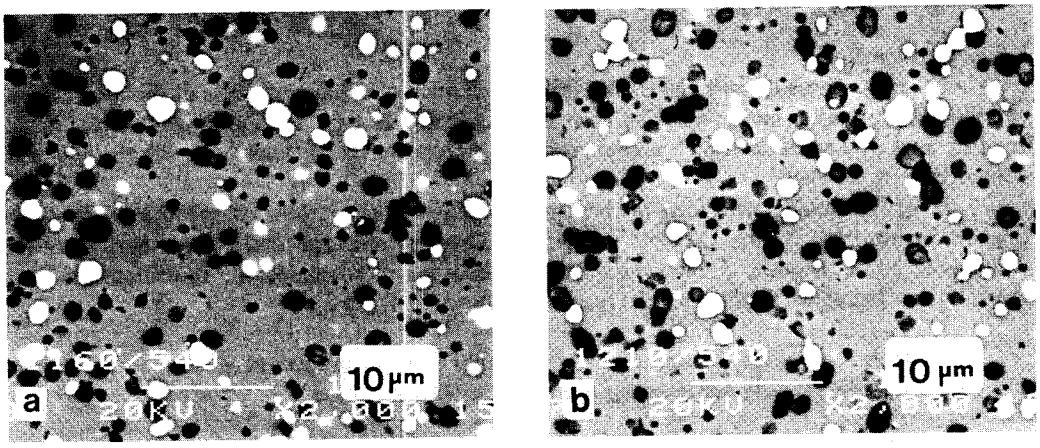

Fig. 4: Scanning electron micrographs of AISI T-15 steel after quenching and tempering: a) 1160/540 °C; b) 1210/540 °C. Electrolytically etched with chromic acid.

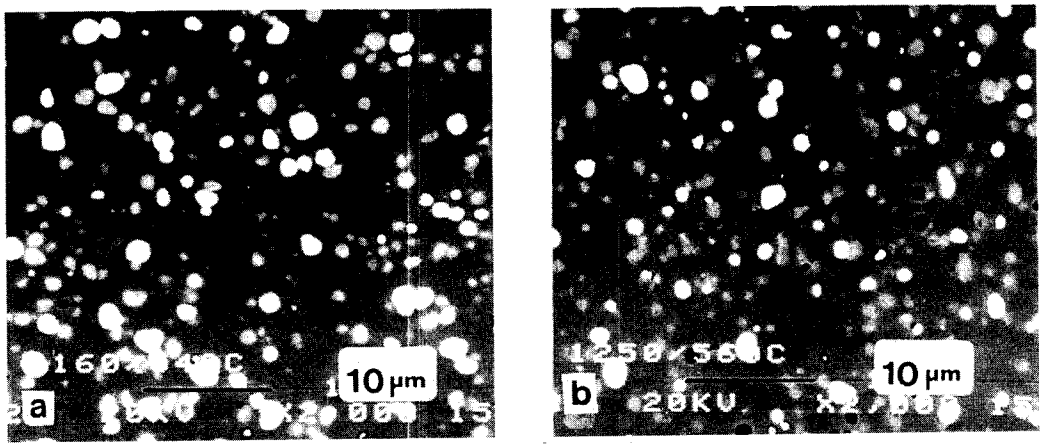

Fig. 5: Scanning electron micrographs of AISI T-15 steel after quenching and tempering: a) 1160/540 °C; b) 1210/540 °C. Electrolytically etched in solution containing 10 % sodium hydroxide and 1 % tartaric acid.

Table 2. Average size of austenite grains(µm)

	Methods		
Quench. temp. (°C)	Snyder-Graff	Quantikov	Quantimet
1160	7.0 ± 1.2	6.3 ± 0.4	6.6 ± 0.5
1210	7.9 ± 1.1	6.3 ± 1.0	6.4 ± 0.3

It can be seen that the three methods used for measuring austenite grain size, presented results close to one another, and besides this, an increase in quench temperature did not result in an increase in average austenite grain size.

3.3. Carbides

3.3.1. Total carbides

A large volume of carbides is always found in microstructures of steels of this type. These carbides, known as primary carbides and generally of the type M_6C and MC, form during solidification of the steel and their amounts as well as relative proportions vary with the chemical composition of the steel.

Selective etching with nital (etchant a), at 70°C, of the as received specimens and those quenched from 1160 °C and 1210 °C, permitted the average size and total volume fraction of the carbides to be determined, without differentiating between the carbides.The measurements were made by the Quantikov method and the results are shown in Table 3.

Table 3. Average size and volume fraction of carbides in specimens, as received, and after quenching from 1160 °C and 1210 °C

Specimens	Average size (μm)	Volume fraction (%)
as received	0.6 ± 0.5	31
1160 °C	1.4 ± 0.6	13
1210 °C	1.0 ± 0.5	13

The 31% volume fraction in the as received specimen, indicates that the material is annealed. All the other volume fraction values obtained are in agreement with results presented in reference [3]. No significant differences were observed in the carbide volume fraction, between those heat treated at 1160 °C and 1210 °C, where in a lesser amount of carbides were expected in those heat treated at 1210 °C, because of the higher solubility. In terms of average carbide size, the expected results were obtained, that is, the average size was higher for those quenched from the lower temperature.

3.3.2. MC and M_6C type carbides

Also in this case, five distinct areas were studied for improved statistical confidence. The "Quantimet" method could not be used to determine the carbides, as the density values of the MC type carbides were very close to that of the matrix, and therefore did not reveal sufficient contrast for the phase to be identified [2].

Since the MC type carbides also impede grain growth, it is essential to determine their content with respect to the total volume fraction of carbides. Thus, the "Quantikov" method was used to determine the carbide content.

This method enabled the unetched specimens to be studied, using the backscattered electron image (composition image) which differentiated between the M_6C (white) and MC (grey) carbides. Fig. 3b shows the back scattered electron image of the as received specimen.

After selective metallographic etching of specimens with different quench and temper treatments, the average size and volume fraction of the MC and M_6C carbides in these specimens were measured. The results are shown in Table 4.

Table 4. Average size and volume fraction of the MC and M_6C carbides in specimens quenched and tempered from different temperatures.

Temperature Quench. / Temp.	Type of carbide	Average Size (μm)	Volume fraction (%)
1160/540°C	MC	0.7 ± 0.4	6.3
	M_6C	1.0 ± 0.4	5.0
			Total :11.3
1210/540°C	MC	0.8 ± 0.4	7.8
	M_6C	1.2 ± 0.4	5.6
			Total :13,4
1210/560°C	MC	0.7 ± 0.4	7.7
	M_6C	1.0 ± 0.5	5.5
			Total :13.3

3.4. Chemical composition of the MC and M_6C type carbides

Besides identifying the carbides by selective etching, they were analysed and their chemical compositions determined semi-quantitatively. The results are shown in Table 5.

Table 5. Semi-quantitative chemical composition of the MC and M_6C carbides (in weight %)

Quenching temperature	Type of carbide	W	Mo	Cr	V	Fe
1160 °C	MC	43.0	3.8	4.7	40.2	8.4
	M_6C	65.3	3.7	3.4	2.8	24.8
1210 °C	MC	34.5	3.0	5.5	39.4	17.4
	M_6C	62.7	4.2	3.8	3.0	26.3

4. Conclusions

4.1. The chemicals selected for metallographic etching gave the best results, and are therefore considered to be ideal for studying the microstructure of AISI T-15 high speed steels.
4.2. Regarding austenite grain size determination, the three methods gave satisfactory results and could therefore be used depending on their availability.
4.3. The semi-automatic image analysis technique Zeiss "Quantimet" was inadequate for determining carbides, as the MC type carbides have the same density as the matrix, making their identification difficult because of lack of contrast.
4.4. In the backscattered electron image, the M_6C and MC type carbides could be clearly distinguished, thus enabling subsequent study using the "Quantikov" method.
4.5. The volume fraction of the MC and M_6C carbides remained unaltered with increase in tempering temperature of the specimens quenched from 1210°C.
4.6. The main carbides are of the MC type, distributed as a relatively fine dispersion, intragranular, and thicker at the grain boundaries, especially at the triple points. The M_6C carbide frequently appears along with the MC carbides.

5. References

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