MICROSTRUCTURAL CHARACTERIZATION OF CARBIDES IN A CAST HIGH SPEED STEEL USING DIFFERENT METALLOGRAPHIC TECHNIQUES

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Abstract

High speed steels are mostly manufactured by casting and they exhibit a characteristic dendritic microstructure with primary carbides. In this study, the morphology of carbides in a cast high speed steel is investigated using light and scanning electron microscopes. The morphology and structure of the carbides were revealed by means of different metallographic techniques such as chemical and electrolytic etching and color metallography. The combination of these metallographic techniques gives different approaches to how different primary carbide morphologies can be observed in the martensitic structure. Besides, microanalysis was carried out to determine the chemical composition of primary carbides using SEM/EDX.

Keywords : High speed steels, etching, microstructure, primary carbides, characterization.

1. INTRODUCTION

High speed steels (HSS) are mainly used as cutting tools. These steels can retain their hardness even at high temperatures resulting from cutting metals at high speeds. Besides carbon, HSS consists of other alloying elements such as Cr, W, Mo, V and Co. High speed steels are mostly manufactured by casting and they exhibit a characteristic dendritic microstructure. The main problem faced during casting process of HSS is the formation of coarse primary eutectic carbides in the form of a network within the interdendritic zones during solidification. Such eutectic structures cause segregation and this affects the mechanical properties adversely. Niobium and titanium may be added to HSS as carbide formers. Niobium is a strong primary MC former and the morphology of these primary carbides changes from eutectic to smooth, octahedral shape as the amount of niobium increases. The primary carbides and the volume fraction of eutectic carbides in the final microstructure affect the mechanical properties and consequently the cutting performance. All or two (MC+ M_2C or MC+ M_6C) of M_6C, M_2C and MC type eutectic carbides can be found in the casting microstructure of high speed steel. M in M_6C type carbide primarily refers to W and Fe elements and Mo, V and Cr also exist in the carbide structure. M_6C type eutectic carbide is present in W-based alloys, intensively. Low cooling rates and low carbon amount promote this formation. The structure of M_6C type carbides is mainly skeleton-like. The M in the M_2C composition refers to Mo and Cr elements and in W, V and Fe also exist in the carbide crystal. M_2C type eutectic carbide is present in Mo-based alloys, intensively. High cooling rates and high carbon amount promote this formation. The structure of M_2C type carbides is generally in the form of fine or coarse parallel plates. Normally, M_2C decomposes to MC and M_6C during hot forming. The MC carbide is harder than the others and therefore it exhibits superior wear properties. The morphology of Nb-rich MC carbides is controlled by the niobium activity in the melt [1-12].

In this study, carbide phases in niobium alloyed high speed steels are characterized by applying different metallographic methods.

2. EXPERIMENTAL STUDY

2.1 Materials

In this study two high speed steel samples with different niobium contents are used and their chemical compositions are given in **Tab. 1**. Microstructures of high speed steels are designed to improve the cutting performance determined by their wear, strength, hot hardness and toughness properties. They contain vanadium and niobium which are strong MC-type carbide forming elements. Small amount of tungsten, molybdenum, chromium and iron dissolve in such type of carbides. Another type of carbide observed in high speed steel is M_6C-type which is mostly rich in tungsten/vanadium. It can dissolve a small amount of chromium, vanadium, molybdenum and cobalt. The type, morphology, amount and distribution of carbides in the matrix is very important to achieve high mechanical performance.

Table 1. The chemical compositions of the experimental steels (%-wt).

C	W	MO	V	Nb	Cr	Co
1.10	3.00	5.00	1.50	1.00	5.00	5.00
1.31	1.34	3.69	2.10	2.66	3.28	< 0.01

2.2 Metallographic and microscopic investigations

The specimens were metallographically prepared by grinding with 120, 600 and 1000 mesh SiC paper and followed by polishing with diamond paste. The samples were etched using different etchants and techniques to determine the carbides in the matrix. Firstly, the samples were investigated in polished condition and then etched by 3% nital solution. Secondly the polished samples were sputter coated using copper cathode to obtain interference layer on their surfaces. The samples were tilted during sputtering to obtain a layer with varying thickness. Finally, the samples were subjected to **e**lectrolytic etching in two different electrolytes, 10% NaOH and perchloric acid solutions. The samples were investigated in each step by light microscope (LM, Zeiss Axiotech 100) and scanning electron microscope (SEM, Jeol JSM 6060). Microanalysis was carried out to determine the chemical composition of primary carbides using SEM/EDX (Jeol JSM 840A).

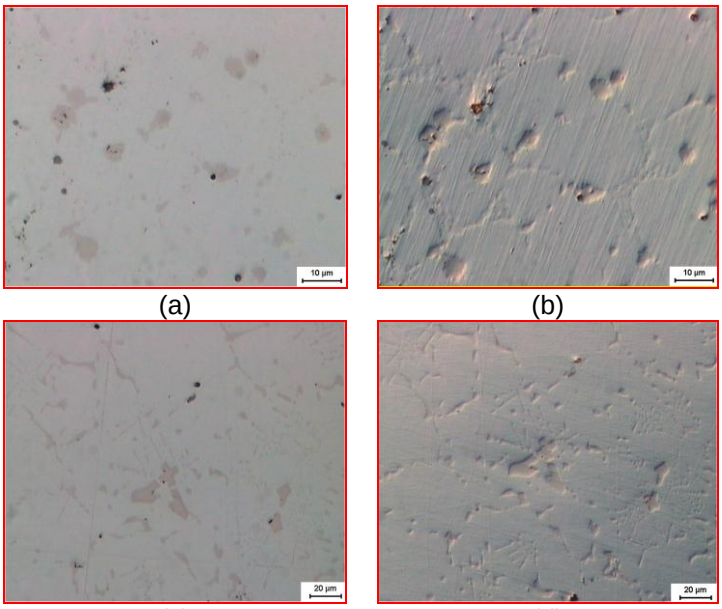
3. RESULTS

3.1 Microstructural investigation in as-polished condition

Fig. 1 gives microstructures of high speed steels in as polished condition. In brightfield imaging carbides in both steels appear in different contrast than the matrix. Differential interference contrast imaging enhances this difference. In the brightfield imaging, the light from the surface is reflected directly to the optic system. The contrast difference in as polished condition is obtained due to the difference in the reflection coefficients of the phases. On the other hand, slight difference in height due to the difference in the hardness of the phases causes varying reflections from the surface in as polished condition. The light reflected from different heights causes phase shift. The human eye cannot detect these phase shifts in the image. It can only distinguished by placing a phase plate in the back focal plane. The difference in hardness between carbides and the matrix will help to observe the carbides (**Fig. 1**). In **Fig. 1a** the MC-type carbides can be distinguished only, whereas in **Fig. 1b** the M_6C-type carbides at the grain boundaries also become visible by using differential interference contrast. The MC carbide structures in the steel containing 2.66 % Nb are shown in **Fig. 1c** and **d**. The contrast is enhanced by using differential interference contrast as seen in **Fig. 1d**, the rod-shaped structures distributed in the matrix are eutectic MC-type carbides.

Scanning electron microscope (SEM) images of the steel having 2.66% Nb in as polished condition are given in **Fig. 2**. In electron microscopy different contrasts can be obtained by using secondary and backscattered electrons. Generally, secondary electrons are used for topographic contrast whereas backscattered electrons are used for composition contrast. In composition contrast, regions with the same atomic number

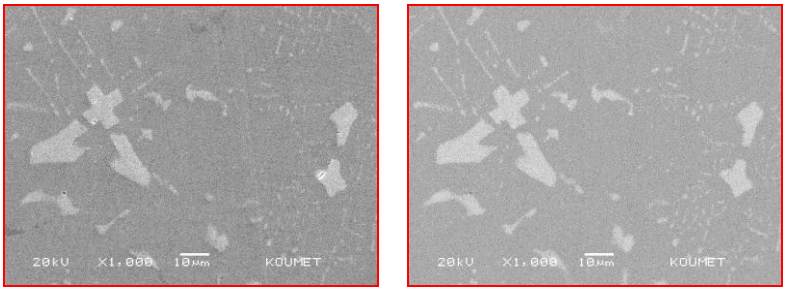
will give the same contrast. Nb-rich primary MC carbides give the same contrast as the Nb-rich eutectic carbides in the microstructures shown in **Fig. 2b**. Microstructures of the same steel after chemical etching, imaged using the same contrast methods, are given in **Fig. 3**.



(a) (b)

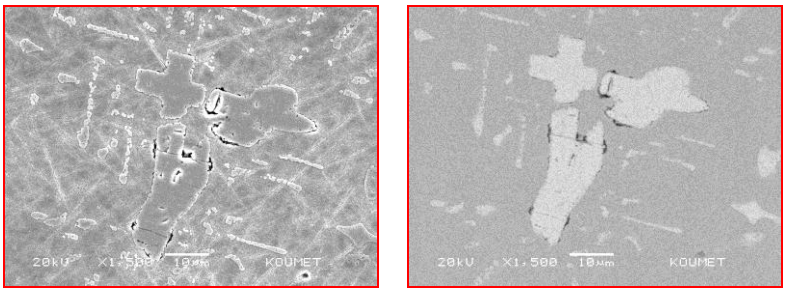
(c) (d)

Figure 1. Microstructures of the high speed steels in as polished condition; (a, c) bright field image, (b, d) differential interference contrast image, (a, b) steel having 1 %Nb, (c, d) steel having 2.66 %Nb.



(a) (b)

Figure 2. SEM images of the steel having 2.66% Nb in as polished condition; taken with (a) secondary electrons (b) backscattered electrons.



(a) (b)

Figure 3. SEM images of the steel having 2.66% Nb after chemical etching; taken with (a) secondary electrons (b) backscattered electrons.

3.2 Microstructural investigation after chemical etching

In metallography, a suitable etching reagent is applied to the polished surface of a sample to reveal the microstructure properties. Etching reveals the microstructure by selective dissolution of the structure. Microstructures of the high speed steels after etching by 3% nital solution are given in **Fig. 4**. M_6C type

carbides at the grain boundaries are seen in the matrix of the steel having lower Nb content (**Fig. 4a**). The matrix consists of a typical tempered martensitic structure. Imaging with differential interference contrast technique enhanced the image compared to the brightfield contrast (**Fig. 4b**). The carbide-matrix interface is clearly elucidated by chemical etching and it is seen that the matrix is in an overtempered condition and far from being martensitic. Along with overtempering, the primary and eutectic carbides start becoming globular due to the effect of soft annealing, as seen from **Fig. 4b**.

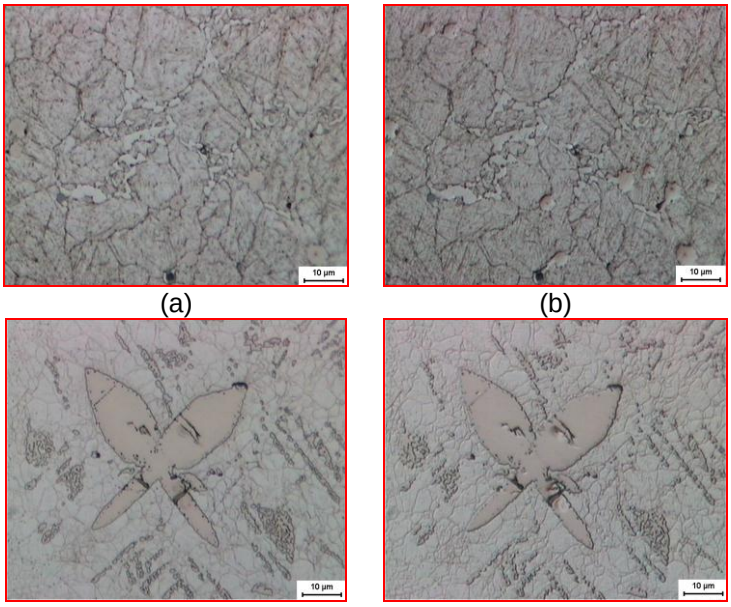


Figure 4. Microstructures of the high speed steels after chemical etching; (a, c) bright field image, (b, d) differential interference contrast image, (a, b) steel having 1 %Nb, (c, d) steel having 2.66 % Nb.

3.3 Color metallography and microstructural characterization

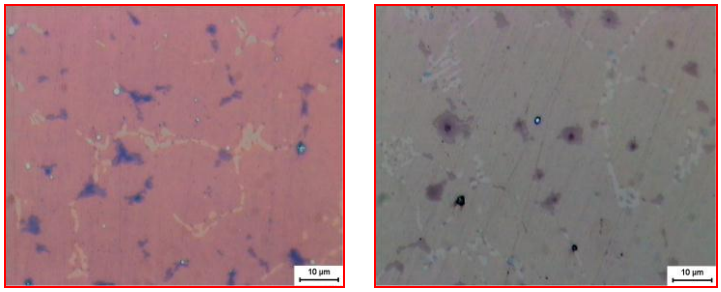
Microstructures obtained by sputtering the polished surface of the steel having %1 Nb are given in **Fig. 5**. The sample is tilted during sputtering and a film layer in varying thickness is obtained on the sample surface. M_6C carbides along grain boundaries are in bright contrast at the relatively thick side of the film layer whereas the MC carbides in the grains and partially along the grain boundaries are in dark contrast as is seen in **Fig. 5a**. Similarly, M_6C carbides appear in brightest contrast and the MC carbides appear in dark contrast at the thin side of the film layer (**Fig. 5b**).

3.4 Electrolytic etching and microstructural characterization

Components/phases in materials exhibit different anodic behaviors in acidic environments and as a result they can be distinguished from each other during microscopic investigations. Electrolytic etching consists of a simple anode-cathode system with suitable electrolyte that reveals the polishing/etching behavior as a function of the applied voltage. The microstructures of the steel having 2.66% Nb are given in **Fig. 6**, after electrolytic etching with NaOH solution. The carbides distributed in the matrix are seen in very dark contrasts. The Nb-rich carbides in the microstructure are dissolved due to the interaction with the electrolyte and they appear in dark contrast because of the low reflection of light.

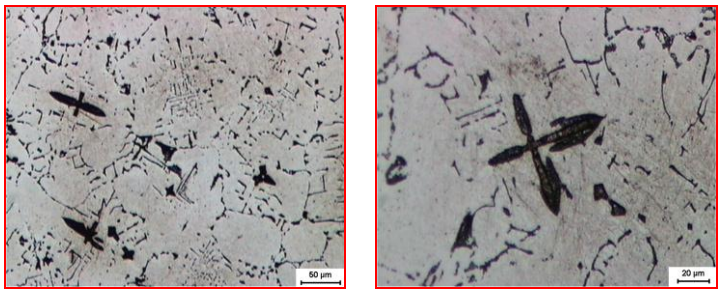
The same steel was electrolytically etched with perchloric acid solution as well. The microstructures taken by SEM are shown in **Fig. 7**. Dissolution of the matrix increased by increasing the etching time due to its anodic behavior. Thus, the matrix-carbide interfaces became more pronounced and carbide morphologies were revealed more clearly (**Fig. 7a-c**). As mentioned earlier, the MC carbides grow on certain habitus planes. In contrast to the eutectic carbides the primary MC carbides preserve their polyhedral shape during hot forming. As can be seen from the surfaces of primary MC carbides given in **Fig. 7c**, the surface growth is not

smooth but rather rough and exhibits a sponge-like morphology. Such a formation is a result of the surface mobility at high temperatures. Carbide forming elements (Fe, Cr etc.) diffuse from the carbide to the matrix during homogenization annealing process. So the surface of the carbide gains a sponge-like appearance as a result of these surface motions.



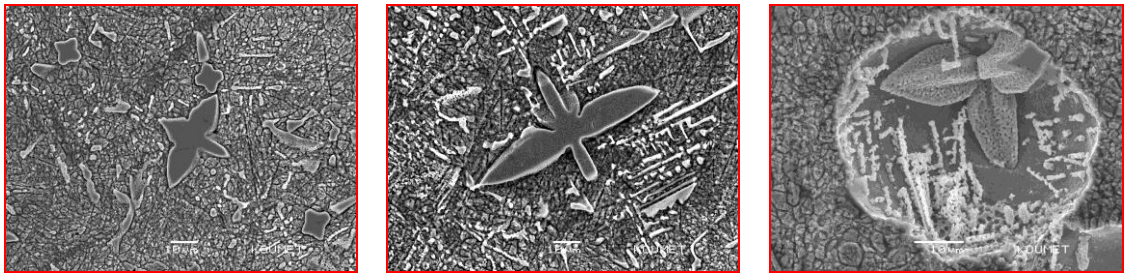
(a) (b)

Figure 5. Microstructures of the high speed steel having 1 % Nb after sputter coating the polished surface; (a) at relatively thick side of the film layer, (b) at relatively thin side of the film layer.



(a) (b)

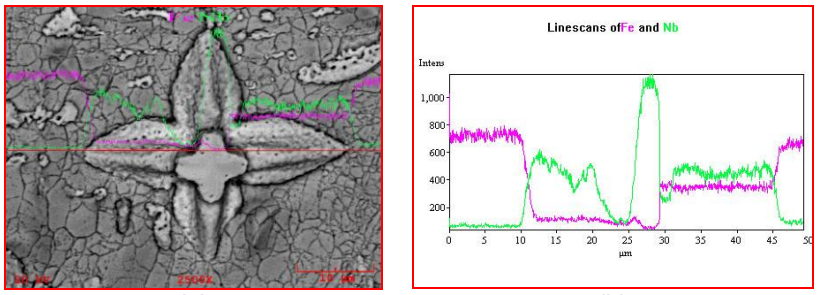
Figure 6. Microstructures of the steel having 2.66% Nb after electrolytic etching with NaOH solution.



(a) (b) (c)

Figure 7. SEM images of the steel having 2.66% Nb after electrolytic etching with perchloric acid solution; etching time (a) 60 s, (b) 180 s and (c) 480 s.

Microanalysis was carried out by SEM/EDX to determine the elemental distribution of niobium and iron in the primary carbides (**Fig. 8**). A line scan is applied to a primary carbide in the steel alloyed with 2.66 % niobium. The carbide is rich in niobium whereas iron solubility is very low as is seen from microanalysis data.



(a) (b)

Figure 8. (a) SEM image and (b) line scan of steel having 2.66% Nb after electrolytic etching with perchloric acid solution.

4. CONCLUSIONS

The aim of the study was to investigate the morphology and distribution of the carbide phases in high speed steels alloyed with 1 and 2.66 % Nb using different metallographic methods. Results may be summarized as follows:

(i) In both of the steels, carbides appear in different contrast from the matrix in as polished condition. In light microscope investigations, differential interference contrast revealed the morphology and distribution of the carbides at the grain boundaries and within the grains better, compared to the bright field imaging. M_6C carbides along the grain boundaries and faceted MC carbides distributed as massive precipitates were observed in the steel containing lower niobium.

(ii) Matrix phase became more apparent by etching with nital. However the carbides could not be distinguished from each other. Therefore, color metallography was utilized to improve the contrast. An interference layer is formed by sputtering method on the polished and etched surfaces. As the thickness of this layer on the polished surfaces increased, carbide phases became more pronounced.

(iii) It is observed that the carbide phase dissolved in the basic solution (NaOH) whereas the matrix phase is dissolved in acidic solution (perchloric acid). Line scan performed using energy dispersive X-ray spectrometer revealed that the primary carbide in the matrix was indeed a Nb-rich phase.

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