Effect of Ti and Nb on the Formation of Carbides and the Mechanical Properties in As-cast AISI-M7 High-speed Steel

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In this research, the carbides of as-cast AISI M7 high-speed steel, in which different amounts of V and W were replaced by equivalent Ti and Nb, were investigated using light and electron microscope. EDX and WDX analysers were used in order to recognize the type and formulae of carbides. The volume fractions of each type of carbides are determined using especial etchant, so that the effects of Nb and Ti on the carbides types, shapes and volume percentages could be understand.

The results show that Nb and Ti increase the volume fraction of MC instead of M$_2$C or M$_6$C and decrease the volume fractions of total carbides. It is also found that, the volume fractions of M$_2$C decrease and M$_6$C increase with increasing Nb percentage.

KEY WORDS: cast carbides; M7 high-speed steel; VC; M$_2$C; M$_6$C; Ti; Nb; mechanical properties.

1. Introduction

High-speed steel (HSS) structures as well as those of other tool steels consists of ferrite matrix strengthened by ultra fine secondary alloying carbides which has high plastic deferability resistance. There is also some larger alloying carbide in HSS structure, precipitated during the solidification. These kinds of carbides can improve the abrasive wear resistance of cutting tools. Chemical composition, shape, size and distribution of these carbides have significant effect on tools properties. These major primary carbides in high-speed steels are known to be M$_6$C, M$_2$C, and MC type carbides.

M$_6$C or η carbides have general formula of A$_4$B$_2$C to A$_3$B$_3$C, where A=(Ti, In, V, Cr, Fe), and B=(Ta, Zr, Nb, W, Mo). The crystal structures of these carbides are FCC with crystal lattice parameter of 1.098 to 1.110 nm. There are 112 atoms in this crystal unit cell. It is reported that specific gravity of M$_6$C carbides is from 10.4 g/cm$^3$ in AISI-M2 steel to 12.0 g/cm$^3$ in AISI-T1 steel. Shape of eutectic M$_6$C in cast high-speed steel is fishbone or skeleton shape in which the central layers are present.

M$_2$C or η carbides have general formula of A$_2$B$_3$C to A$_3$B$_4$C, where A=(Ti, In, V, Cr, Fe), and B=(Ta, Zr, Nb, W, Mo). The crystal structures of these carbides are FCC with crystal lattice parameter of 1.098 to 1.110 nm. There are 112 atoms in this crystal unit cell. It is reported that specific gravity of M$_2$C carbides is from 10.4 g/cm$^2$ in AISI-M2 steel to 12.0 g/cm$^2$ in AISI-T1 steel. Shape of eutectic M$_2$C in cast high-speed steel is fishbone or skeleton shape in which the central layers are present.

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M$_2$C carbides have Mo$_2$C formula, in molybdenum high-speed steels, with HCP crystal in which the lattice parameters $a$ and $c$ are changed from 0.2960 to 0.3012 nm and from 0.4669 to 0.4735 nm respectively. Shape of eutectic M$_2$C in cast molybdenum HSS are lamellar or feathery type. M$_2$C carbides are thermodynamically unstable at high temperature and are decomposed to M$_6$C and MC, hence they are seldom remained in heat treated high-speed steel tools as primary carbides.

MC carbides are VC, NbC and TiC. Their crystal lattices are FCC and there are always lack of carbon in VC and NbC. It is reported that the MC lattices parameters are 0.4173, 0.4433 and 0.4320 nm for VC, NbC and TiC respectively. The MC carbides are the hardest among all kinds of carbides in HSS.

The specifications of the cast high-speed steel carbides determine the mechanical properties of steels. In HSS, the formation of eutectic carbides during solidification and their unsuitable distribution in cast structure deteriorate the mechanical properties. So the structural control of carbides by changing their types, and shapes can improve the mechanical properties. From this point of view, the author has studied introducing Ti and Nb to the cast high-speed steel and the effects of these elements on the mechanical properties. Many details about the improvement of mechanical properties and cutting performances of the amendment cast HSS have been given in the literatures.

The aim of this research is the evaluation of the effects of Ti and Nb on the shape, type, distribution and volume percentages of cast carbides in high-speed steel.

2. Experimental Procedures

In AISI M7 steel, 1/4, 1/2, 3/4 and all of W were replaced by equivalent Ti and Nb, therefore four Nb-steels called Nb1–Nb4 and four Ti-steels called Ti1–Ti4 were prepared. The 1/4, 1/2 and 3/4 of V were also replaced by equivalent Ti to make Ti5–Ti7 steels, and Nb to make Nb5–Nb7 steels. The whole amounts of V were not replaced because of the significant role of V in the formation of secondary MC carbides during tempering. The fundamental of this replacement is based on the carbide forming roles of the elements, i.e. Ti, Nb and V make MC carbides and W makes Fe$_7$W$_2$C or Fe$_7$W$_3$C in high-speed steels. So, based
on the atomic weights of these elements, 1 wt% of V must be equal to 1.82 wt% of Nb or 0.94 wt% of Ti, and 1 wt% of W must be equal to 0.22–0.34 wt% of Nb or 0.12–0.17 wt% of Ti, due to the lower amounts of W in Fe₄W₂C, and upper amount of it in Fe₃W₃C. Table 1 shows the composition of these cast steels. The samples with the section sizes of 14 × 14 mm and 10 × 10 mm, were prepared by the method of investment casting using an air-melting induction furnace. The pouring temperature was 1600°C and mould temperature was equal to room temperature, so the samples cooling rates were estimated approximately 25°C/s and 100°C/s for 14 × 14 and 10 × 10 mm section sizes samples, from the measurement of dendrite secondary arm spacing. Light and electron microscopy tests were done at the centre point of the cross section of each sample, using etchant solutions shown in Table 2. All samples were as-cast and no heat treatments were done.

Quantitative metallography was carried out according to ASTM E562 method using especial etchant for each carbide shown in Table 2. The standard deviation for carbides volume percentage was about 0.5%. EDX point analyser as well as X-ray mapping was used to recognize the type of carbides using comparison the results with those published in literatures. WDX analyser was used quantitatively to determine the carbides formula. It was calibrated with pure elements and its results were corrected with ZAF (atomic numbers, absorption and fluorescence effect). Each analysis was carried out at the magnification over than 10000. Each result of WDX shown in Table 3 is the average of analysis of 5 different samples.

For further study of the mechanical properties, all as-cast 10 × 10 section sizes samples were heated to 1200°C after annealing followed by oil quenching then double tempering for 2 h at 525°C. The temperature accuracy for austenitizing and tempering furnaces are 5°C and 3°C. Bend tests carried out according to ISO7438 were used to study the strength and toughness of 8.8 × 30 mm specimens. Standard deviations were 0.3 HRC for hardness, 40 MPa for bend strength and 0.2 for max. bend strain.

3. Results and Discussions

3.1. Carbides Types

There are three types of eutectic carbides in these cast steel.

3.1.1. Lamellar M₂C Eutectic

This type of carbide, for example as is shown in Fig. 1(a) (point A) for Nb3 steel, has thick and parallel layers. X-ray mapping of Mo, W, V, and Fe shown in Fig. 1(b), indicates the high concentration of Mo in this carbide. It can be recognized that, there are some amounts of W and V in M₂C. Results of quantitative WDX analysis, shown in Table 3, indicates that this type of carbide is Mo₄C which dissolves some amounts of Fe, W, V and Cr. This kind of carbide is dominant eutectic carbide in all cast steels used in this experiment. It is agree with the result that M₂C is the main eutectic carbide in cast Mo high-speed steels. It must be noted that the only eutectic type carbide, which observed in Ti-alloyed steels, is M₃C.

3.1.2. Rod Like M₂C Eutectic

There is another kind of eutectic carbide in Nb-alloyed
steels especially at high amount of Nb. The layers in this carbide become short and thin so that they are similar to rod shape. Figure 2 shows such carbide in Nb6 steel which chemical formulae, shown in Table 3, are the same as lamellar type. Addition of Nb seems to assist the formation of this type of carbides.

3.1.3. Skeleton M6C Eutectic

This kind of carbide is seldom seen in the cast steel structures, because these steels are in the Mo series. But in this experiment there is some M6C especially in Nb-alloyed steels at high weight percent of Nb. Figure 3 shows M6C in Nb7 steel. The central branch, which is the characteristics of this type of carbide, can be seen at the point A in Fig. 3. WDX analysis results (Table 3) indicates that this type of carbide is Fe3Mo3C which dissolves Nb, V, W and Cr. Increasing Nb seems to be the main reason of the formation of this carbide.21)

3.2. Other Carbides

There are some single carbides in addition to the eutectic carbides mentioned above, in the cast steels investigated in this study, which are as follows:

3.2.1. Vanadium Carbide

This type of carbide seems to be precipitated at the end of solidification just before the formation of eutectic phase,22) hence they have approximately large dispersed single shape and are always formed near the eutectic carbides. Figure 4(a) shows VC, point A, beside M6C (point B) in martensite matrix (point C) of Nb2 steel. X-Ray mapping of W, Mo, V and Fe, shown in Fig. 4(b), shows the ex-

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**Table 3.** Chemical composition of carbides in cast steels.

| Carbide shape          | Steel number | Atomic % | Chemical formula                                      | Carbide Type |
|------------------------|--------------|----------|------------------------------------------------------|--------------|
| Lamellar eutectic      | Ti7          | Fe 21.35 | Mo 37.62 | V 3.45 | Cr 3.12 | W 2.75 | Ti 1.07 | Nb -- | C 30.66 | (Fe0.29,Mo0.12,V0.03,Cr0.10,W0.06,Ti0.02) C |
| Fixed carbide          | Ti7          | Fe 1.18  | Mo 6.78 | V 4.64 | Cr 3.65 | W 0.85 | Ti 35.55 | Nb -- | C 47.35 | (Fe0.02,Mo0.14,V0.04,Cr0.08,W0.07,Ti0.79) C |
| Blochly carbide        | Ti2          | Fe 2.15  | Mo 13.40 | V 28.40 | Cr 2.64 | W 1.08 | Ti 11.41 | Nb -- | C 40.91 | (Fe0.05,Mo0.33,V0.09,Co0.06,W0.05,Ti0.28) C |
| Lamellar eutectic      | Nb3          | Fe 19.42 | Mo 23.30 | V 6.80 | Cr 11.33 | W 4.85 | Ti 2.27 | Nb 32.36 | C 13.79 | (Fe0.46,Mo0.12,V0.21,Co0.31,W0.13,Nb0.07) C |
| Skeleton eutectic      | Nb7          | Fe 42.07 | Mo 33.10 | V 4.35 | Cr 4.69 | W 1.38 | Ti 1.52 | Nb 32.15 | C 13.79 | (Fe0.46,Mo0.12,V0.21,Co0.31,W0.13,Nb0.07) C |
| Rod-like eutectic      | Nb6          | Fe 21.54 | Mo 25.08 | V 5.47 | Cr 9.97 | W 3.22 | Ti 2.57 | Nb 32.15 | C 13.79 | (Fe0.46,Mo0.12,V0.21,Co0.31,W0.13,Nb0.07) C |
| Blochly carbide        | Nb2          | Fe 2.18  | Mo 12.52 | V 30.74 | Cr 2.14 | W 2.83 | Ti -- | Nb 8.24 | C 40.35 | (Fe0.05,Mo0.30,V0.34,Co0.01,W0.01,Nb0.20) C |
| String-like carbide    | Nb7          | Fe 6.10  | Mo 3.20  | V 8.02 | Cr 4.51 | W 1.43 | Ti -- | Nb 32.94 | C 43.8 | (Fe0.14,Mo0.07,V0.18,Co0.10,W0.02,Nb0.72) C |

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Fig. 2. SEM structure of Nb6 steel showing, A - rod-like M6C eutectic in B - martensite matrix.

Fig. 3. SEM structure of Nb7 steel showing, skeleton M6C eutectic.

Fig. 4. (a) SEM structure of Nb2 steel showing, A - VC, B - lamellar M6C eutectic in C - martensite matrix, (b) X-ray mapping of (W, Mo/V, Fe) in (a).
istance of V in this carbide, however there are some Mo and W in it. WDX analysis results of these carbides (Table 3) indicate that this type of carbide is VC, which dissolves some Mo, Ti in Ti-alloyed steel (Ti2), and some Nb in Nb-alloyed steels (Nb2). However, the shape of this carbide will change with the increasing of the amount of V, because it is reported that at high vanadium percentages the quasieutectic of VC and austenite will form.

3.2.2. Niobium Carbide

These types of carbides, which are present in Nb-alloyed steels, are like strings which join together at one point. These long strings form at wide temperature range.15) Figure 5(a) shows such carbides (point A) in martensite matrix of Nb7 steel. X-ray mapping (Fig. 5(b)) of this figure indicates that Nb is the main metallic element in this carbide as well as some amounts of V in it. WDX analysis results of this carbide (Table 3) also shows that this type of carbide is NbC, which dissolve some amounts of V and Mo. It is also reported that at high amount of Nb a quasieutectic type of NbC and austenite will form.

3.2.3. Titanium Carbide

This carbide, which is found in Ti-alloyed steels, has angular shape and is dispersed as single particles in the microstructures. TiC, like NbC, precipitate from the melt at the beginning of solidification.25) Figure 6(a) shows these carbides (point A), and also M2C (point B) in the martensite matrix of Ti7 steel. It can be seen from X-ray mapping of Mo, Ti, V and Fe (Fig. 6(b)) that, however Ti may be the main metallic element in this carbide but Mo and V have some solubility in it. WDX-analysis results (Table 3) indicate that this carbide is TiC which dissolve some V and Mo.

3.3. Quantitative Measurements of Volume Percentages of Carbides

3.3.1. MC Carbides

Figure 7, which is drawn from the results of quantitative metallographic measurements of all cast steels, shows the effect of V weight percentages on volume percentages of VC in two series of Nb And Ti-alloyed high-speed steels. This measurement was done with the aid of etchant No. 3 in Table 2. It can be seen that the amount of VC increases with increasing vanadium percentage. But the amounts of VC in Ti-steels are always less than those in Nb-steels. In fact Ti has greater affinity than Nb to form carbide, hence there are less carbon for produce VC in Ti-steels. On
Table 3 shows that the solubility of Nb in $M_2C$ and $M_6C$ is larger than that of Ti, so that in Nb-steels, there are less free Nb to attract carbon to form NbC, and the remainder carbon can increase the volume of VC.

As can be seen in Fig. 8, increasing the percentages of Nb or Ti naturally leads to increase the percentages of NbC or TiC in Ti-steels or Nb-steels. There are higher amounts of TiC than those of NbC at the same weight percentage of Ti or Nb in cast steels. Not only what has been mentioned for Fig. 7 can cause it, but also it must be due to the lower specific gravity of Ti (47.9 g/mol) than of Nb (92.9 g/mol). It is interesting to note that the volumes of NbC rise rapidly at high amount of Nb. It means that the amounts of NbC are much higher than that of TiC at the weight percentages of 2.7 Nb and 1.4 Ti in respectively Nb7 and Ti7 steels. It seems that the quantitative measurement, which is obtained by using light microscope, has low accuracy for measurement the high amounts of NbC, because the quasieutectic structure of austenite and NbC, formed in Nb-steels containing high amount of Nb, are so fine to study by light microscope. Hence this error can cause obtaining the high amount of NbC in Fig. 8. Factually the results of NbC volume percentages in Nb6 and Nb7 steels shown in Fig. 8 must contain both volumes of NbC and austenite, which are existed between the strings of Nb carbides.

The volume of MC, i.e. NbC+VC or TiC+VC, versus the amounts of Nb or Ti are drawn in Fig. 9. As the main aim of this work is to replace some alloying elements in AISIM7 HSS by the equivalent Ti and Nb, and not to add Ti and Nb to the standard composition of this steel, so that the results of Fig. 9 can be mentioned clearly in Figs. 10 and 11. These figures show the volumes of MC carbides versus the ratio of Nb/V or Ti/V (wt%/wt%), replacement of V by Nb in Nb-alloyed steels and Ti in Ti-alloyed steels, and the ratio of Nb/(W+Mo) or Ti/(W+Mo) (wt%/wt%), replacement of W by Nb in Nb-alloyed steels and Ti in Ti-alloyed steels (according to what has been mentioned in Sec. 2), respectively. As Mo has the important roll in these Mo-based HSS and the effects of Mo are the same as those of W, so the amounts of Mo were added to the amounts of W at the denominator of the fraction in the X axis of Fig. 11. As can be seen in Fig. 10, increasing the ratio of Ti/V or Nb/V rises the volume MC carbides. In fact the solubility of V in $M_2C$ and $M_6C$ carbides are more than that of Nb and Ti (Table 3), so that at the presence of Nb and Ti, there are higher amounts of these two elements to form NbC and TiC than that of V to form VC. In such a case the volume of MC will naturally increase. These results are the same as those of Fig. 11. In fact W is $M_6C$ carbide former\(^{17}\) and Nb and Ti are MC carbide formers in high-speed steels, hence increasing the amounts of Ti and Nb and decreasing the amounts of W+Mo leads to form high volumes of MC carbides instead of $M_6C$ carbides. It can be seen in Fig. 10 that, increasing Nb and Ti with decreasing V leads to increase the volume of MC, but it seems that, this decreasing must naturally decrease the volume of MC because V is...
MC carbide former element. But it must be noticed that, according to the fundamental of replacement of elements in AISIM7 in this research, which was mentioned in the "experimental procedures", decreasing the amount of V is equal to increase the amounts of Nb and Ti in Nb5 to Nb7 and Ti5 to Ti7 steels in Table 1.

3.3.2. Eutectic Carbides (M_{2}C and M_{6}C)

Figure 12 shows the effects of Nb or Ti on the volume of eutectic carbides using etchant No. 2 in Table 2. It has been known that Nb or Ti reacts with some carbon of the melt to produce NbC or TiC at the beginning of solidification, so that the remained carbon of the melt will decrease. In such a case the interdendritic melt, which can transform to eutectic phase, will also decrease. It must be noted that V hasn’t this effect, so that the replacement of V by Ti or Nb can also reduce the eutectic carbides. It can be understood in Fig. 12 that the effect of Ti on the decreasing of eutectic carbide is much higher than that of Nb. The lower atomic weight of Ti than that of Nb can cause this effect. Also it seems that the solubility of Ti in M_{2}C carbides is less than that of Nb (Ti7, Nb3 and Nb7 steels in Table 3), hence the modifying effect of Ti in reduction of eutectic carbides can be higher than that of Nb.

The dominant eutectic carbide is M_{2}C in all cast steels, however there are some M_{6}C eutectic carbides in Nb-steels at higher than 1 wt% Nb (as mentioned in Sec. 3.1.3), which increase with increasing Nb percentages. As it is written in some references, the higher solubility of Nb in M_{6}C than that in M_{2}C (Table 3) probably causes the formation of M_{6}C carbide.

The same results of the effects of Ti and Nb on the eutectic carbides are drawn versus the ratios of Ti/V or Nb/V in Fig. 13 and [(Ti or Nb)/(W+Mo)] in Fig. 14. The reasons of eutectic carbide reduction in these figures and the higher effect of Ti than that of Nb in this reduction are the same as those have been mentioned for Fig. 12.

Figures 15–17 show that the amounts of total carbides...
decrease with increasing the amounts of Nb or Ti (Fig. 15), the ratio of Nb/V or Ti/V (Fig. 16) and the (Nb or Ti)/(W + Mo) ratio in two series of cast steels. In fact decreasing the amounts of V and W with increasing the amounts of Ti and Nb can reduce the volumes of eutectic carbides (Figs. 12–14) and increase the amount of MC (Figs. 9–11). But it must be noted that at the constant percentage of C, substitution of M6C or M2C by MC leads to lower volume of total carbides. In fact MC in which only one metallic atom bonds with one atom of carbon has lower volume than that of M6C in which six metallic atoms are present for one carbon atom, so that in such a case the volume of total carbides must decrease. But at high amount of Ti and Nb, in Figs. 15–17, the volumes of total carbides show increasing, because of the high increase of the volumes of TiC and NbC. In Nb-alloyed experimental steels, this increasing is depend on the accuracy of measurement, mentioned in Sec. 3.3.1.

3.4. Mechanical Properties

It is reported that\textsuperscript{15,25,33} the modified AISI57 which is heat treated under the optimum heat treatment cycle\textsuperscript{24} has the excellent mechanical properties.\textsuperscript{34} Figure 18 shows the effects of Ti or Nb contents on the hardness of two fully heat treated cast steels series. It can be observed that the maximum hardness can be achieved at 0.7 wt% of Ti and 1.8 wt% of Nb in Ti-alloyed and Nb-alloyed steels. The modification effects of Ti and Nb in the microstructure, i.e., less eutectic carbides, finer eutectic cells, more homogeneity in the microstructure, causing an increasing in the hardness. However, when the amounts of Ti and Nb become more than these percentages, these elements attract large amounts of carbon to make TiC and NbC. These carbides have no solubility in austenite and cannot participate in the formation of secondary carbides at tempering.\textsuperscript{27} Hence, there is insufficient carbon for other elements to make secondary carbides,\textsuperscript{1} so the hardness will decrease. The max. hardness of Ti-alloyed steels is higher than that of Nb-alloyed steels. It is due to the better distribution of TiC than that of NbC, which has mentioned in Sec. 3.2.

Figures 19 and 20 show the effect of Ti and Nb contents on the bend strength and max. bend strain of fully heat treated Ti-alloyed and Nb-alloyed steels. Increased hardness leads to increased bend strength, for the same reasons as discussed above for Fig. 18, so the general shapes of the curves in Fig. 19 are the same as those in Fig. 18, with the exception of that the maxima of bend strength are located at lower amounts of Ti and Nb than those of hardness. It must be due to that the steel will be more brittle at the max. hardness. The more homogeneous distribution of eutectic carbides in modified steels, not only increase the hardness but also increase significantly the toughness of the experimental steels, so the bend strain increase with increasing Ti.
and Nb contents up to 0.7 Ti or 1.8 Nb in Figs. 19 and 20. The bend strains show slightly decrease after the max., in Figs. 19 and 20, which is due to the formation of high amounts of TiC and NbC carbides at high amounts of Ti and Nb (Fig. 8). The bend strain increases at high amounts of Ti (1.4) and Nb (2.7), because of the high decrease in the hardness of matrix, which is mentioned above for Fig. 18.

4. Conclusion

Replacement of a part of V and W by equivalent Nb or Ti in AISI M7 high-speed steel has these results:

1) The main eutectic carbide is lamellar M₂C in Ti-alloyed and Nb-alloyed steels, however there are also some skeleton M₆C eutectic and rod-like M₂C eutectic in Nb-alloyed steels.

2) The shape of VC is single blocky particle in all cast steels, and the shape of TiC is single faceted particle in Ti-alloyed steels, but NbC has string-shape in Nb-alloyed steels.

3) Increasing Nb up to 2.7 wt% in Nb-alloyed and Ti up to 1.4 wt% in Ti-alloyed steels increase NbC volumes up to 8.3% and TiC volumes up to 5.7%.

4) Increasing the ratio of Nb/V or [Nb/(W+Mo)] and Ti/V or [Ti/(W+Mo)] increases the MC volumes up to 9.1% and 6.3% in Nb-alloyed or Ti-alloyed steels respectively.

5) Increasing Nb up to 2.7% increases the volumes of M₆C up to 5.4%.

6) Increasing the ratio of Nb/V or [Nb/(W+Mo)] and Ti/V or [Ti/(W+Mo)] decreases the volumes of eutectic carbides up to 6.9% and 5.1% in Nb-alloyed or Ti-alloyed steels respectively.

7) The optimum amounts of substitution Ti and Nb for the best hardness, bend strength and bend strain are about 0.7 wt% and 1.8 wt% respectively.

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