Morphology and Properties of M_2C Eutectic Carbides in AISI M2 Steel

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In as-cast structure of AISI M2 steel, the predominant type of eutectic carbides is M_2C , the morphology of which has crucial influence on the distribution and dimension of carbides in final products. In the present work, the morphology and properties of M_2C carbides formed at different cooling conditions have been investigated by means of optical microscope (OM), scanning electron microscope (SEM), energy dispersive X-ray spectroscopy (EDS), transmission electron microscope (TEM), X-ray diffraction (XRD) and differential thermal analysis (DTA). With increasing cooling rates, the morphology of M_2C transforms from the lamellar type to the rod-like one, and the two carbides show different growing characteristics during solidification. Compared with the lamellar carbides, rod-like M_2C is less stable and decomposes faster at high temperatures, accelerating the separation and spheroidization of carbides, even after hot deformation. It is concluded that the formation of rod-like M_2C in cast ingots promotes homogeneous distribution and refinement of carbides in the final products, favoring the improvement of mechanical properties of high speed steels.

KEY WORDS: M_2C ; morphology; cooling rate; decomposition; thermal stability; high speed steel.

1. Introduction

High speed steels are widely used in making cutting tools which require high hardness and wear resistance at high temperatures. Among them, AISI M2 steel is the most popular one, owing to its excellent combination of hardness and toughness.

The mechanical properties of high speed steels are determined by the dimension and distribution of carbides, which are closely related to the as-cast structure of ingots, particularly the morphology and properties of eutectic carbides.¹⁾ Different kinds of eutectic carbides are generated by the decomposition of liquid, which can be expressed as liquid $\rightarrow$ austenite + eutectic carbides, including M_2C , M_6C and MC .^{2,3)} For M2 steel, the predominant type is M_2C .⁴⁾

Previous studies have shown that the morphology of M_2C can be classified into two types, namely the lamellar shape and the rod-like shape.^{4,5)} Lamellar M_2C is favored by low cooling rates or high vanadium whereas rod-like M_2C is promoted by high cooling rates or minor elements.^{4,6,7)} Nevertheless, the mechanism of morphology transition of M_2C has not been clearly understood. Fredriksson *et al.* have studied the relationship between the morphology of M_2C and its vanadium content, and proposed that the morphology transition is due to the change of vanadium segregation before eutectic solidification.⁶⁾ This proposal is contradicted by Boccalini who shows that nitrogen favors the formation of M_2C rather than M_6C , in which the vanadium content is lower (if the proposal is true, the opposite should occur).⁷⁾

M_2C is metastable and decomposes at high temperatures. Many researchers have studied the decomposition process of lamellar M_2C , which can be described by the following formula $M_2C + Fe(\gamma) \rightarrow M_6C + MC$.^{8,9)} In contrast, the properties and decomposition process of rod-like M_2C have been rarely reported so far. Little is known about the effect of morphology change of M_2C on the microstructure and mechanical properties of final products.

The main objective of our present work is to investigate the differences in properties of M_2C formed at different cooling conditions. Furthermore, the morphological characteristics of both the lamellar and rod-like M_2C have also been carefully examined and a possible transition mechanism is proposed.

2. Experimental Details

2.1. Material Preparation

Material used in this work was AISI M2 high speed steel, the chemical compositions of which are listed in **Table 1**. The steel was remelted by a non-oxidation process with a 15 kg medium frequency furnace. Then it was cast in the sand mould and iron mould, the dimensions of which were 60 mm $\times$ 60 mm $\times$ 150 mm and 30 mm $\times$ 30 mm $\times$ 150 mm, respectively. The specimens used in the following proce-

Table 1. Chemical compositions of investigated AISI M2 steel (wt%).

C	Si	Mn	W	Mo	Cr	V	S	P	Fe
0.89	0.35	0.24	5.93	4.82	3.95	2.08	0.009	0.029	Bal.

dures were taken from the centre of the ingots at about one third height.

2.2. Morphology and Structure Characterization of M_2C

The morphology of carbides was observed by optical microscope using Murakami etchant ($3\text{ g K}_3\text{Fe(CN)}_6 + 10\text{ g NaOH} + 100\text{ mL H}_2\text{O}$), in which M_2C carbides were selectively etched rather than the matrix.¹⁰⁾ Further observation on the three-dimensional morphology of carbides using FEI Sirion-400 SEM was performed on the specimens which were deeply etched in an etchant of $5\text{ mL HF} + 100\text{ mL H}_2\text{O}_2$. The chemical compositions of both the carbides and matrix were measured quantitatively using Genesis 60S EDS. They were measured at ten different positions and then averaging. In order to get the accurate content of alloying element, the condenser current of SEM was increased to the maximum, minimizing the diameter of electron beam.

In order to study the phase transition of eutectic carbides with cooling rates, XRD was carried out in the range of 30 to 90° glancing angles at a rate of $0.2^\circ/\text{min}$, using XD-3A diffractometer with $\text{Cu K}\alpha$ radiation, operating at 40 kV and 30 mA . The samples for XRD include the blocks sliced from the ingots and the carbide powders extracted from the ingots. The extraction of carbides was performed using electrolysis, operating at 40 V , 0°C . The electrolyte contained 7 g citric acid, 20 mL hydrochloric acid and 250 mL methanol.

2.3. Property Characterization of M_2C

DTA was used to study phase transformations of the ingots during heating process ranging from room temperature to 1200°C at a rate of $3^\circ\text{C}/\text{min}$, using Diamond TG/DTA. The whole heating process was protected in an argon atmosphere. The specimens for DTA were about 30 mg in weight and loaded in an alumina tube.

Samples from the ingots were heated at 1150°C for 1 and 3 h in a furnace, respectively. The morphology change and phase transition of carbides after heating were observed using SEM and JEM 2000EX TEM. Carbide powders were extracted from the heated samples and examined by XRD.

Further study on the evolution of M_2C during the subsequent forging process was also performed. The ingots for forging were prepared as mentioned above. Then, the ingot solidifying in the sand mould was machined to dimensions of $30\text{ mm} \times 30\text{ mm} \times 150\text{ mm}$, identical to those of the ingot solidifying in the iron mould. They were heated at 1100°C for 1 h and then forged to square billets with sectional dimensions of $20\text{ mm} \times 20\text{ mm}$ and $13\text{ mm} \times 13\text{ mm}$. The corresponding forging reduction ratio was about 1.75 and 4 , respectively. Specimens were taken from the center of the billets. The microstructure was investigated using SEM. The carbides in the billets with a reduction ratio of 1.75 were extracted and examined using XRD. The samples from billets with a reduction ratio of 4 were heated at 1200°C for 5 min , followed by being quenched in the oil and tempered at 560°C for 3 h . The hardness after quenching and tempering was measured.

3. Results and Discussion

3.1. Morphology of M_2C Formed at Different Cooling Conditions

Figure 1 shows the typical microstructure of M_2 cast ingots under different cooling conditions. It consists of the matrix and networks of M_2C eutectic carbides distributed in the interdendritic regions. The secondary dendrite arm spacings at the test positions have been measured, and the cooling rates are calculated by the equation $\text{DAS} = A(\text{dT}/\text{dt})^{-b}$.⁵⁾ It is estimated that the cooling rates are about 0.9 K/s and 20 K/s at the test positions in the ingot solidifying in the sand mould and iron mould, respectively. At low cooling rates, M_2C carbides present a needle-like or lamellar shape, and the interface between the eutectic colony and primary grain is ragged. With increasing cooling rates, the carbides bend and develop into a rod-like shape. Meanwhile, the carbides are refined and the spacing between them also decreases, outlining the interface clearly. It is also found that the content of alloying elements in rod-like M_2C decreases remarkably while the opposite occurs in the matrix between adjacent M_2C , as shown in Table 2. This is in agreement with previous work.³⁾

The three-dimensional morphology of M_2C is illustrated

Fig. 1. The typical microstructure of M_2 cast ingots under different cooling conditions: (a) lamellar M_2C in the ingot solidifying in the sand mould where the cooling rate is low and (b) rod-like M_2C in the ingot solidifying in the iron mould where the cooling rate is high.

Table 2. The content of alloying elements in M_2C and matrix between adjacent M_2C (wt%).

M_2C	Elements in M_2C					Elements in the matrix between M_2C				
	V	Mo	W	Cr	Fe	V	Mo	W	Cr	Fe
Lamellar type	11.4	27.4	38.7	5.6	16.9	1.0	2.7	4.1	3.2	89.0
Rod-like type	5.7	14.3	24.1	5.1	50.8	1.9	4.6	5.3	3.4	84.8

Fig. 2. The three-dimensional morphology of M_2C carbides in M2 cast ingots after the matrix is removed by an etchant of 5 mL HF + 100 mL H_2O_2 : (a) lamellar M_2C in the ingot solidifying in the sand mould and (b) rod-like M_2C in the ingot solidifying in the iron mould.

in **Fig. 2**. It can be seen that M_2C exhibits a lamellar structure with flat plates at low cooling rates while it has a rod-like structure with round outlines at higher cooling rates, suggesting that the two carbides may have different growing characteristics. It seems that lamellar M_2C grows anisotropically and much faster in the directions parallel to the crystal surfaces of plates. In contrast, the growth of rod-like M_2C is somewhat isotropic and no preferred growing directions are observed.

It is well known that the crystal morphology during solidification is determined by the solid/liquid interface structure which can be classified as the faceted phase and non-faceted phase, depending on the fusion entropy and interface undercooling.¹¹⁾ Due to high fusion entropy, the carbides are generally considered to be a faceted phase, the growth of which is anisotropic. The crystal planes with higher index grow more rapidly, leaving the close-packed crystallographic planes as the facets on the crystal surface.¹²⁾ For M_2C with the hexagonal close-packed structure,¹³⁾ the most close-packed planes are $\{0001\}$. It is inferred that lamellar M_2C is a faceted phase and the crystal surfaces of plates should be parallel to $\{0001\}$ planes.

However, the characteristics of the faceted phase disappear in rod-like M_2C , including anisotropic growth and having special crystallographic planes on the surfaces. The arrow in **Fig. 2(b)** shows this tendency. It can be seen that the plate is not strictly flat and tends to get curved. More generally, M_2C grows into a dendritic and rod-like structure with round outlines, presenting the characteristics of the non-faceted phase.

It is widely accepted that the faceted phase can transit into a non-faceted phase with increasing undercooling, which has been proved by the transformation of Si phase

Fig. 3. XRD profiles of M2 cast ingots solidifying in (a) the sand mould and (b) the iron mould. (c) and (d) are the corresponding XRD profiles of carbide powders extracted from the ingots, respectively.

from faceted plates to non-faceted dendrites in Al–Si alloys.^{14,15)} Thus, it is inferred that rod-like M_2C formed at high cooling rates is a non-faceted phase. The decreasing amount of strong carbide forming elements in rod-like M_2C may also help to reduce the fusion entropy of carbides, favoring the above transition.

The preceding conjecture is supported by the following XRD results, as shown in **Fig. 3**. Figures 3(a) and 3(b) illustrate the XRD profiles of ingots solidifying in the sand mould and iron mould, respectively. It can be seen that the carbides in both ingots consist of M_2C and a small amount of MC, and there is little difference between them. It indicates that the types of carbides in M2 cast ingots are not influenced by the cooling rates.

Surprisingly, the XRD profiles of carbide powders extracted from the corresponding ingots differ greatly, as shown in **Figs. 3(c) and 3(d)**. It is noted that the intensity of (0002) diffraction peak of lamellar M_2C increases significantly and becomes the strongest. Further observation on the lamellar carbide powders using SEM reveals that the original plates of M_2C are broken into smaller pieces during preparation of XRD specimens. Most of the small plates are nearly parallel to the surface of XRD specimens, which is expected to result in the remarkable increment in the intensity of (0002) peak. It suggests that the crystal surfaces of plates are parallel to (0002). In contrast, the intensity of (10 $\bar{1}1$) peak of rod-like M_2C is the strongest and a little higher than that of (0002) peak (**Fig. 3d**). The relative intensity among all peaks of M_2C is similar to that in the corresponding ingot. It indicates that rod-like M_2C has less special crystallographic planes on the crystal surfaces, confirming the previous assumption.

3.2. Thermal Stability of M_2C During Heating

M_2C is metastable and decomposes into M_6C and MC at high temperatures.^{8,9)} Compared with M_6C , MC is rich in vanadium and can also be signified as VC. The differences of thermal stability between the lamellar and rod-like M_2C are investigated in this section.

Figures 4(a) and 4(b) illustrate the DTA profiles of sam-

ples taken from ingots solidifying in the sand mould and iron mould, respectively. It can be seen that the two profiles are similar below 850°C. The exothermic and endothermic peaks are caused by the precipitation of carbides from the

Fig. 4. DTA profiles of the samples taken from M2 cast ingots solidifying in (a) the sand mould and (b) the iron mould.

matrix and the formation of austenite, respectively. The difference of reaction temperatures between them is due to the decreasing amount of alloying elements in the primary austenite with increasing cooling rates.

However, the two profiles differ above 850°C. An endothermic peak appears at about 898°C in the DTA profile of the sample solidifying in the iron mould. It is expected to be resulted from the decomposition of rod-like M_2C , which is demonstrated by further observation on the DTA sample using SEM. In contrast, the endothermic peak is not obvious for the sample from the sand mould. It suggests that the thermal stability between the two carbides is different and rod-like M_2C is less stable than the lamellar carbides at high temperatures, which is confirmed by the following results.

Figure 5 shows the morphology of M_2C in the ingots after heating at 1 150°C for 1 h. It can be seen that both the lamellar and rod-like M_2C decomposes into M_6C and MC. However, the decomposition process seems to be different. During decomposition of the lamellar carbides, MC is formed inside M_2C and surrounded by another new phase M_6C . In contrast, it is generally generated at the interface between rod-like M_2C and the matrix, and the dimensions of it seem to be larger, suggesting that it grows more rapidly than that generated from the decomposition of lamellar M_2C .

Detailed observation reveals that the lamellar carbides decompose incompletely and there is M_2C remaining in the centre of the plates. In contrast, little M_2C is observed in the sample after heating. This is confirmed by the XRD

Fig. 5. SEM micrographs of M_2C in M2 cast ingots after heating at 1 150°C for 1 h: (a) lamellar M_2C in the ingot solidifying in the sand mould and (c) rod-like M_2C in the ingot solidifying in the iron mould. (b) and (d) are the corresponding TEM micrographs, respectively. The arrows show the presence of different phases after the decomposition of M_2C .

profiles of carbide powders extracted from the ingots (**Fig. 6**). It is noted that there is still a great amount of lamellar M_2C in the sample while only a little rod-like M_2C remains after heating. It proves that the thermal stability of rod-like M_2C is lower than that of lamellar M_2C .

It is expected that the difference of thermal stability is determined by the decomposition process of M_2C , particularly for the formation of MC.¹⁶⁾ During decomposition of the lamellar carbides, MC is formed inside M_2C which provides vanadium for the formation of MC.⁸⁾ However, it is difficult for MC to be generated inside rod-like M_2C , due to the lack of vanadium in it, as shown in Table 2. Since the content of vanadium in the matrix increases, the matrix probably participates in the decomposition of rod-like M_2C and provides vanadium for the formation of MC, which is evidenced by the decreasing amount of vanadium in the matrix during decomposition.¹⁶⁾ Thus, MC tends to nucleate at the interface between rod-like M_2C and the matrix. Compared to the diffusion of elements inside carbides, the diffusion rate of alloying elements at the interface is much more rapid, thereby accelerating the formation of MC and decomposition of rod-like M_2C . The refinement of eutectic carbide and the decreasing amount of strong carbide forming elements in it are also expected to favor the decomposition of M_2C .³⁾

Figure 7 shows the morphology of M_2C in the ingots after heating at 1 150°C for 3 h. Both the lamellar and rod-like M_2C decompose completely. However, the morphology differs greatly after heating. It is noted that the lamellar carbides get a little thinned due to the dissolution of carbides at high temperatures, but remain coarse and almost keep their original shapes on the whole. In contrast, the rod-like carbides separate from each other and spheroidize after heating, reducing the dimensions of carbides remarkably. Lower thermal stability and refined structure of rod-like M_2C are expected to result in the difference of microstructure after heating compared to the case of lamellar carbides.

3.3. Morphology Evolution of M_2C During Forging

The results above indicate that the formation of rod-like M_2C in cast ingots helps to refine the carbides after heating.

Fig. 6. XRD profiles of carbide powders extracted from M2 cast ingots after heating at 1 150°C for 1 h: (a) the ingot solidifying in the sand mould and (b) the iron mould.

Whether such an effect remains or not during the subsequent forging process is unknown. Therefore, a further investigation of it is carried out.

Figure 8 shows the microstructure of billets with a forging reduction ratio of 1.75 and 4, respectively. At a reduction ratio of 1.75, the lamellar carbides are bended and broken into pieces, owing to mechanical crushing. However, they remain coarse on the whole. Further deformation reduces the dimensions of carbides effectively. They are broken into smaller pieces with irregular shapes and distribute more homogeneously at a reduction ratio of 4.

It is also observed from Fig. 8(a) that only a little M_2C decomposes during the preheating and forging process. This is confirmed by the XRD profiles of the corresponding carbide powders, in which a great amount of M_2C remains after forging (**Fig. 9(a)**). Thus, it is expected that the morphology evolution of lamellar carbides during the forging process is mainly caused by deformation.

In contrast, the morphology of rod-like carbides changes greatly after forging. At a reduction ratio of 1.7, the carbide colony is elongated along the direction of deformation. Meanwhile, separation and spheroidization occur in the colony, reducing the dimensions of carbides. It is interesting to note that the separation of rod-like carbides after hot forging is more obvious than that after heating only (Fig. 5(c)), even though the heating temperature prior to the forging procedure is lower. With increasing reduction ratios, the carbides are set apart and distribute more homogeneously. It should be noted that the carbides are significantly refined at a reduction ratio of 4. The average dimensions are less than 2 μm , almost at the same level as those of carbides precipitated from the matrix.

A further examination of carbide powders extracted from

Fig. 7. SEM micrographs of M_2C in M2 cast ingots after heating at 1 150°C for 3 h: (a) lamellar M_2C in the ingot solidifying in the sand mould and (b) rod-like M_2C in the ingot solidifying in the iron mould.

Fig. 8. SEM micrographs of M_2C in the ingots with different forging reduction ratios: (a) lamellar M_2C at a reduction ratio of 1.75 and (b) 4; (c) rod-like M_2C at a reduction ratio of 1.75 and (d) 4.

Fig. 9. XRD profiles of carbide powders extracted from the ingots with a forging reduction ratio of 1.75: (a) the ingot solidifying in the sand mould and (b) the iron mould.

the corresponding billet with a reduction ratio of 1.7 reveals that only a small amount of M_2C remains (Fig. 9(b)). It is expected that the morphology evolution of rod-like carbides during the forging process is caused by both heating and deformation. Owing to lower stability and refined structure, rod-like M_2C is much easier to separate and spheroidize, favoring the refinement of carbides, particularly at low reduction ratios.

Table 3 illustrates the hardness of different samples after quenching and tempering. It shows that the sample from the billet with rod-like M_2C obtains higher hardness compared to the case of lamellar M_2C . This is because the refined and spherical carbides evolving from rod-like M_2C are much easier to dissolve at high temperatures, thereby increasing the supersaturation of alloying elements in the matrix after

Table 3. Hardness of the samples after quenching at 1200°C and then tempering at 560°C.

Hardness, HRC	Samples	
	Billet with lamellar M_2C	Billet with rod-like M_2C
	62.4	63.6

quenching and promoting the precipitation of fine secondary carbides from it after tempering. Furthermore, refined and spherical carbides in the final products can also improve the toughness, which is very important for tools.¹⁾ Thus, it is concluded that the formation of rod-like M_2C in the cast ingots favors the improvement of mechanical properties of high speed steels.

4. Conclusions

The present work has investigated the morphology and properties of M_2C eutectic carbides in AISI M2 cast ingots under different cooling conditions. The results obtained are summarized as follows.

With increasing cooling rates, M_2C transforms from the lamellar type to the rod-like one and the two carbides are found to exhibit different growing characteristics. It is expected that lamellar M_2C grows as a faceted phase while rod-like M_2C formed at high cooling rates is probably a non-faceted phase.

The lamellar and rod-like M_2C differ greatly in the decomposition process and thermal stability. Compared with the lamellar carbides, rod-like M_2C is less stable and easier to separate and spheroidize after heating, reducing the dimensions of carbides remarkably. The formation of rod-like M_2C in cast ingots promotes homogeneous distribution and refinement of carbides in the final products, favoring the improvement of mechanical properties.

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