

Eutectic Modification in a Low-Chromium White Cast Iron by a Mixture of Titanium, Rare Earths, and Bismuth: I. Effect on Microstructure

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The present work studies the effect of small additions of a mixture of Ti, rare earths (RE), and Bi on the eutectic solidification of a low-Cr white cast iron (WCI) commercially designed as Ni-Hard Class I Type B according to the ASTM A532. For this purpose, systematic additions of a mixture of ferrotitanium-rare earths-bismuth (Fe-Ti-RE-Bi) up to 2% were made to a low-Cr WCI. By means of these additions, a modified carbide structure was obtained. Eutectic carbides changed from a highly interconnected ledeburite structure to more isolated, finer blocky structure. This microstructural change caused variations in the mechanical behavior of the WCI. The decrease in size and reduced connectivity of the eutectic carbides increased fracture toughness as well as wear resistance under dry sliding conditions. The effect of the admixture on the microstructure and mechanical properties is discussed in terms of the segregation and refining effects of the elements that comprise the mixture.

Keywords eutectic, microstructure, modifier mixture, rare earths, white iron

1. Introduction

The main alloying elements involved in low-Cr white cast iron (WCI) are Ni, Cr, Mo, and Mn (Ref 1), and the as-cast characteristic microstructure, according to this chemical composition, is a network of eutectic M_3C carbides (where M represents the metallic species Fe, Cr, Mo, Mn, etc.) in a matrix of austenite plus martensite. The high amount of alloying elements promotes hardenability, and therefore, an austenitic matrix in the as-cast conditions. In addition, the high volume fraction of hard eutectic carbides makes these alloys very suitable for applications where a high wear resistance is required. Consequently, the mining and mineral processing industries are the main consumers of these alloys (Ref 2).

However, the high volume fraction of carbides with their high connectivity in the matrix makes these irons quite brittle. The use of these alloys is, therefore, limited to applications where high abrasion resistance, in addition to some resistance to corrosion, is required, and the castings do not undergo repetitive impact that may cause fracture (Ref 3). An increase in fracture toughness may be obtained by reducing the amount of carbon in the alloy since this reduces the carbide volume fraction. However, wear resistance will be decreased as a result. On the other hand, by increasing the amount of carbide forming elements, the eutectic carbide volume fraction is increased, improving wear resistance. On the other hand, this reduces the fracture toughness (Ref 3). On this basis, research is underway

around the world with the purpose of making iron alloys not only more wear resistant but also more fracture tolerant than currently available WCI.

It is well known that one possible strategy to improve fracture toughness in WCI, as well as their wear resistance under sliding conditions, involves a refinement of the carbide structure by reducing their size and making the shape more rounded (Ref 4-6). Rapid cooling (Ref 7, 8) or small undercoolings (Ref 9) during ironmaking has been used to increase eutectic carbide nucleation and to limit their growth, producing fine carbides. However, this procedure is limited to thin sections. The majority of castings are thick section, and the low cooling rates experienced in these castings produce coarse structures of highly interconnected carbides (Ref 10, 11). Another possible strategy for improving fracture toughness is to change the carbide to a more spherical shape by long heat treatments (Ref 12). However, this implies an increase in production costs with limited benefits. The most appropriate way to get smaller more isolated carbides in these alloys is by mean chemical modification. By adding small amounts of modifier elements, the eutectic austenite/ M_3C can be modified, producing finer carbides (Ref 13-21). In addition, carbide forming elements have been added such as Nb (Ref 22-24), or V and Ti (Ref 23, 24), to change the carbide stoichiometry which may change its shape. Small amounts of B (0.1-0.3%) (Ref 15, 19, 21) have been used, particularly in high-Cr WCI. It is believed that B diminishes C solubility in the austenite producing higher C precipitation, consequently, increasing the number of nuclei for eutectic carbide precipitation. The effect of Si has been also investigated (Ref 19). Those using Si report a refinement of the structure and posit that Si inhibits carbide growth. Other work (Ref 18, 20) has found that Si diminishes the nucleation process, does not affect carbide growth, and results in coarser structures.

The use of rare-earth (RE) additions to modify eutectic growth has been studied in low-Cr WCI. Qinxiang et al. (Ref 13) found that the continuous carbide network could be

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changed to a structure of more isolated carbides by adding RE to a Cr-Ni WCI (1.7%Cr-4.5%Ni-3%C). They explained such a phenomenon on the basis that the RE segregates in front of the austenite crystals during solidification, generating sufficient undercooling to allow polycrystal formation instead of single-crystal growth. Under these conditions, many austenite dendrites connect with others, preventing the eutectic M_3C carbides from growing mostly in the $\langle 010 \rangle$ crystallographic direction.

On the other hand, Qian et al. (Ref 14) found that cementite in low-alloyed irons could be modified into irregular blocky crystal by adding an admixture of ReAl-N-Ti. They attribute these results to the combined effects of the elements in the modifier alloy. Rare-earth elements are very reactive with O and S and clean the alloy of these impurities. They also promote the divorced eutectic growth and refine the microstructure, including primary austenite dendrites that cause a disruption of the cementite network. Aluminum also contributes to the cleaning of the alloy while titanium-carbonitrides Ti(CN) contribute to austenite nucleation. The effectiveness of the modifier alloy was higher in low-C irons.

In unidirectional solidification experiments, Li and Smith (Ref 16) observed a ledeburite structure that converted into laths during the eutectic austenite/(Fe,Cr) $_3$ C solidification when Ce was added to the liquid alloy. They argued that the high solute concentration reduced the local temperature of the solid-liquid equilibrium and retarded carbide phase growth.

According to these authors, the modification of the eutectic structure of carbides has been carried out, particularly in low-Cr irons where the eutectic is of the type austenite/ M_3C . From the present work, similar results were obtained.

For the present work, Ti was selected since it forms a high melting point carbide, or carbonitride, which forms in the alloy before it solidifies. This could produce a high number of nucleation centers for austenite. In addition, RE elements as commercial mischmetal (i.e., 48% Ce, 25% La, 17% Nd, 5% Pr, and 5% Fe) and Bi were also selected. It is well known that these elements have a very low solubility in liquid Fe and segregate to the eutectic region during solidification. It is expected that these elements will segregate to the carbide/matrix interphase boundary and limit carbide growth from the liquid surrounding the eutectic cells. Under these conditions, the structure of the WCI would be composed of smaller and more isolated carbides.

In this study, additions from 0% to 2% of the admixture of 30% FeTi-50% RE-20% Bi (Fe-Ti-RE-Bi) were added to a low-Cr WCI. The resulting alloys were characterized by optical and electron microscopy and x-ray diffraction. The microstructural features were correlated with mechanical behavior (i.e., hardness, microhardness, fracture toughness, and wear resistance).

2. Experimental Procedure

According to the ASTM specification (Ref 1), the chemical composition of the Ni-Hard Iron class I Type B is 5-3% C, 1.3% Mn (max), 0.8% Si (max), 3.3-5.0% Ni, 1.4-4% Cr, and 1% Mo (max). The alloy used in the current study was made in a laboratory induction furnace using high-purity raw materials. A 25 kg charge of the alloy was melted and poured at 1450 °C into 5 kg capacity SiC crucibles where the modifier alloy was added. Therefore, 5 crucibles with different additions of the

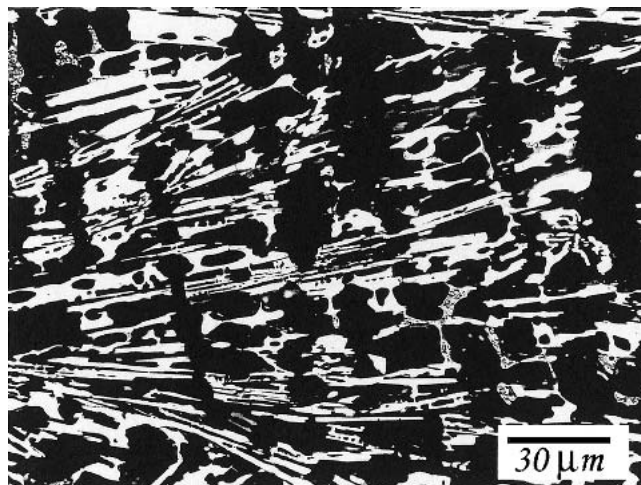


Fig. 1 Microphotograph of a deep-etched sample of the experimental WCI showing high contrast between matrix (dark phase) and the eutectic carbides (white phase); light microscope

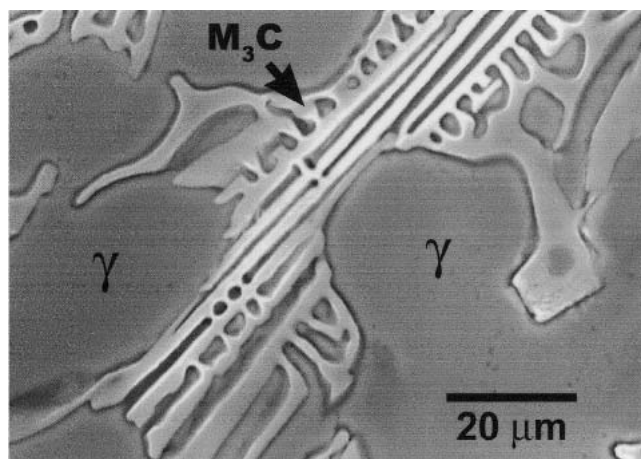


Fig. 2 As-cast microstructure of the experimental WCI showing zones of proeutectic austenite (γ) and the austenite/ M_3C eutectic; SEM; Vilellás etch for 30 s

Table 1 Chemical composition for the experimental WCI (wt.%)

C	Cr	Fe	Mn	Mo	Ni	P	S	Si
2.91	2.9	85.1	2.14	0.95	5.11	0.021	0.016	0.83

modifier alloy were obtained which were then poured into sand molds to obtain the final square (2.5×2.5 cm) cross-section ingots. The amount of the modifier alloy was zero for the first ingot, 0.5% for the second, 1% for the third, 1.5% for the fourth, and 2% for the fifth. Chemical analysis was undertaken by spectroscopy from chill samples obtained during the casting of each ingot.

Samples for metallography, hardness, wear, and fracture toughness tests were cut from the cast bars. Samples were cut using an abrasive alumina wheel at as slow a cutting speed as possible to avoid excessive overheating, which could cause cracking in the samples. Copious amounts of water were used as coolant.

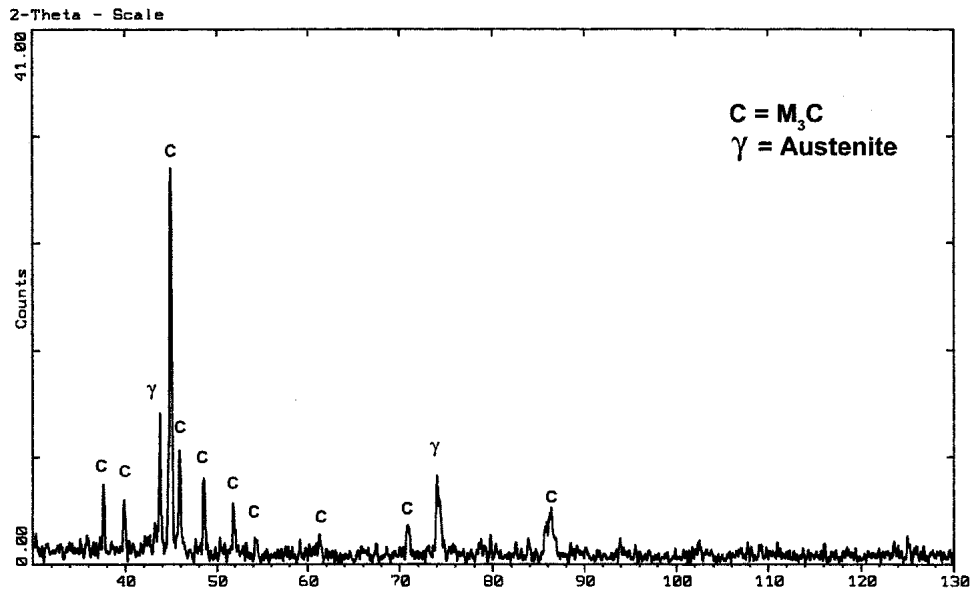


Fig. 3 XRD pattern indicating the WCI is composed by austenite and carbide of the type M_3C ; sample with 1.5% of the modifier admixture

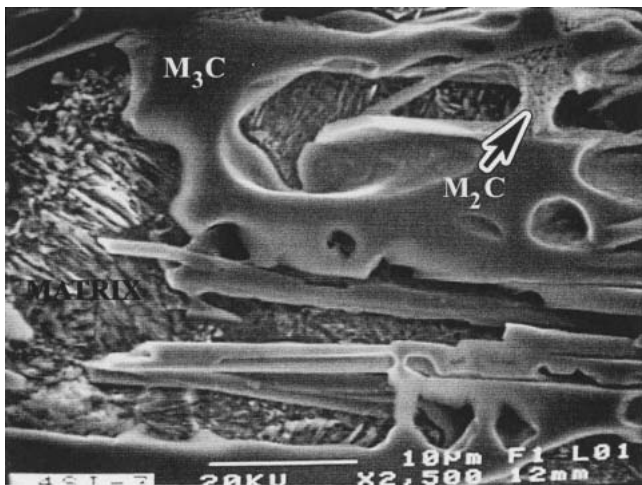


Fig. 4 Deep etched sample of the experimental WCI showing the presence of the Mo-rich M_2C carbides; SEM etched for 3 h in a solution of 50 ml $FeCl_3$ plus 20 ml HCl in 930 ml ethanol

Alloy characterization was undertaken by light optical microscopy, scanning electron microscopy (SEM), microanalysis by energy-dispersive spectrometry (EDS), x-ray diffraction (XRD), and PC-based image analysis.

Samples for metallography were prepared using conventional metallographic techniques. Once polished, the specimens were etched with one of two different reagents, Vilellás reagent (5 ml HCl, 1 g picric acid in 100 ml ethanol) for 30 s to reveal the overall microstructure, or a solution of 50 ml $FeCl_3$ plus 20 ml HCl in 930 ml ethanol for about 3 h for a deep etching. The latter reagent removed part of the matrix without affecting the carbide, providing very high contrast between matrix and carbides when the structure is observed under the light microscope.

Carbide volume fraction and carbide size were measured by image analysis using digitized pictures taken on the optical microscope at about 250 \times . For this purpose, samples were

deep-etched to get high contrast (Fig. 1). Twenty micrographs were processed to obtain carbide information. XRD was used to identify phases and SEM was used to examine the etched microstructures.

3. Results and Discussion

Table 1 shows the nominal chemical composition for the WCI. The norm specifies a maximum amount of Mn of 1.3%. However, the final content in the alloy resulted in 2.3%, 1% higher than the specified content. An excess of Mn affects the final microstructure. The typical microstructure of the matrix should consist in a mixture of austenite and martensite (Ref 9, 13, 23). However, the present alloy resulted in a highly austenitic matrix since Mn stabilizes this phase. Despite the high Mn content, the main objective of the study was still achieved, that is, the effect of alloy modifier on eutectic solidification.

3.1 As-Cast Structure

Figure 2 shows the as-cast structure of the alloy where the austenitic matrix and eutectic carbide can be seen. Since the alloy is hypoeutectic, austenite dendrites are the first to solidify followed by the austenite/ M_3C eutectic. Therefore, the structure is mainly M_3C in a matrix of austenite, as shown by the XRD pattern in Fig. 3. In addition, small amounts of the M_2C and martensite were also detected around the eutectic carbides. The presence of the Mo-rich M_2C in these irons is well documented (Ref 3, 25). Figure 4 shows a deeply etched micrograph where the M_2C can be seen interconnected with the eutectic M_3C . Furthermore, Fig. 5 shows details of the M_2C and its chemical composition by EDS.

The main goal of adding Mo to WCI is to improve hardenability. However, only part of the Mo contributes to this goal. The presence of C in the alloy and its chemical affinity for Mo promotes the formation of M_2C carbides at the end of the solidification process (Ref 3, 25-28). The volume fraction of M_2C is actually quite low. Due to the much higher quantity of

M_3C eutectic carbide, the M_2C has very little effect on the alloy's properties. However, it is present in the structure and must be mentioned.

Small amounts of martensite were also present in the alloy. This phase could only be observed on some micrographs at high magnification (Fig. 6). Martensite, which was not detected by XRD, was commonly located at the carbide/matrix interface. The typical structure of Ni-Hard is a network of the eutectic M_3C carbide in a matrix of austenite and martensite. However, due to the excess Mn, in addition to the high Ni content, these alloys exhibited a predominantly metastable austenitic structure. The presence of martensite at the carbide/matrix interface has been widely reported, and its presence is attributed to C and Cr impoverishment at these areas as a result of diffusion towards the carbides during eutectic solidification, and also during the subsequent cool down (Ref 2, 9, 29).

Element segregation was also studied by EDS in the WCI

with 1.5% of modifier. Figure 7(a) shows a SEM micrograph of that as-cast WCI. Microanalyses were undertaken at the matrix (Fig. 7b), at the carbide phase (Fig. 7c), and at the carbide/matrix interface (Fig. 7d).

Manganese, Cr, Si, C, Ci, and Mo were seen in matrix (see spectrum in Fig. 7b). All these elements contribute to hardenability and stabilize the austenite phase. The spectrum in Fig. 7(c) shows the qualitative composition of the carbide phase. Such a eutectic carbide is of the type M_3C , as characterized by XRD, and is composed of Fe, Cr, Mn, and Mo, i.e., $(Fe,Cr,Mn,Mo)_3C$. It is well known that alloying elements such as Mn and Mo partition to both matrix and carbide (Ref 9, 13, 14). Chromium is a carbide-forming element and is mainly found in the carbide phase. However, it is also present in the matrix in minor amounts. Such is the case for carbon as well (Ref 9, 11). Other alloying elements such as Ni and Si partition completely to the matrix and they are not present in the carbide.

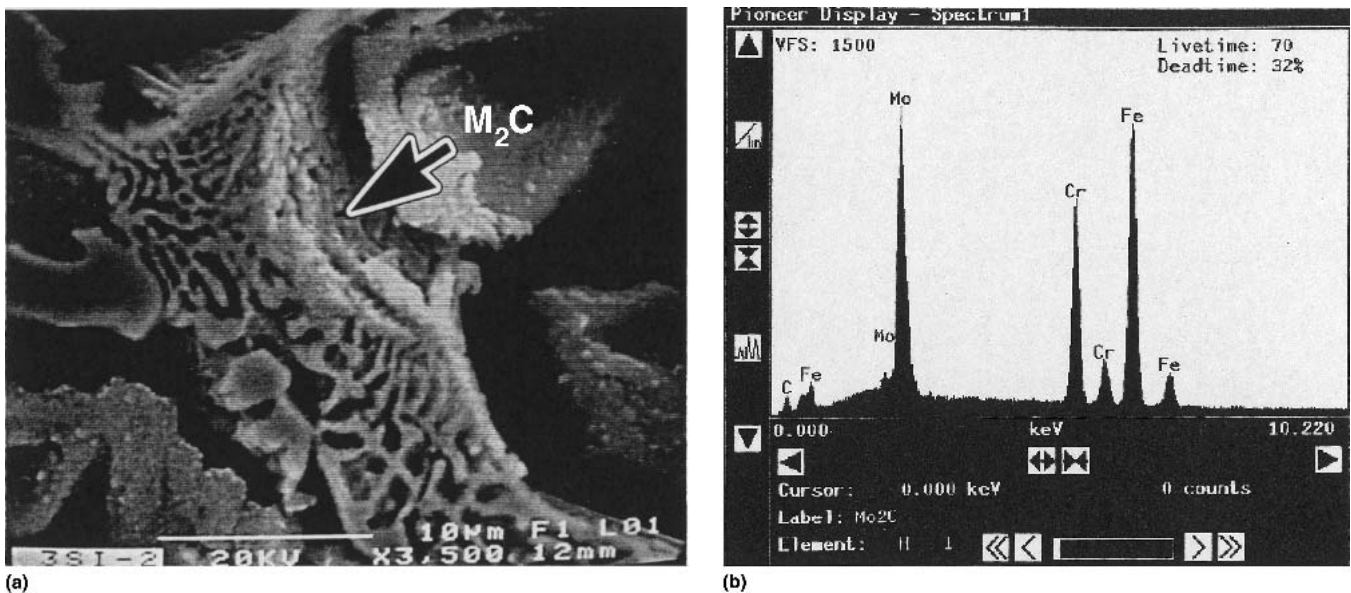


Fig. 5 (a) Detail of the M_2C carbide found in the WCI with chemical analysis by EDS; (b) Note the presence of Mo and Fe as main components of the carbide. SEM; deep etched sample

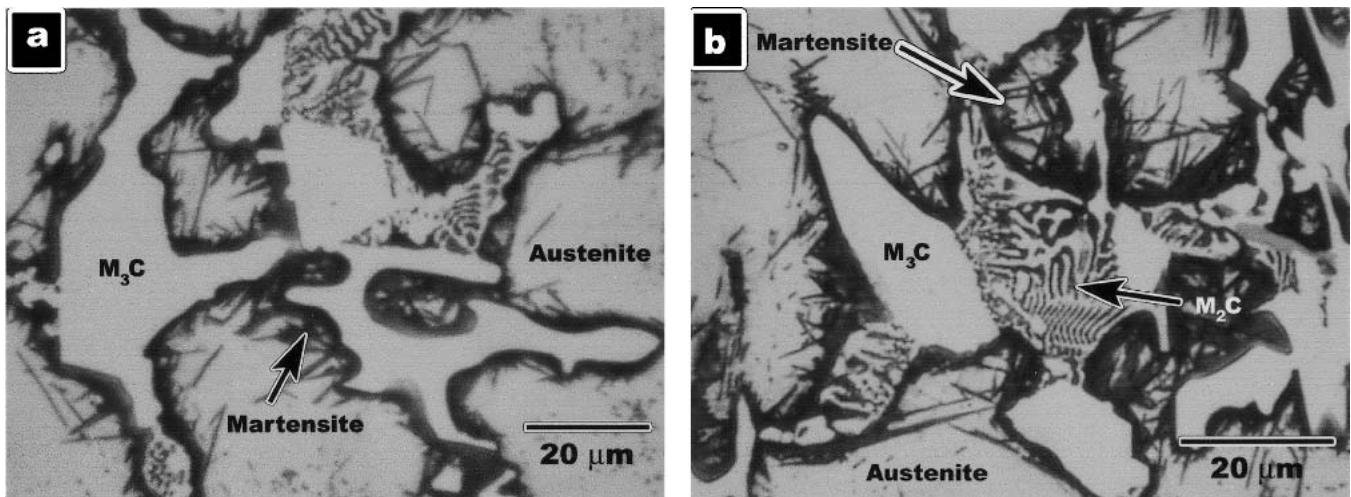


Fig. 6 Photomicrographs showing the presence of martensite needles at the carbide/matrix interface and also the presence of the M_2C -type carbides in some WCI samples; light microscopy; Vilellás etch for 45 s

Figure 7(d) shows the composition at the matrix/carbide interface for the point arrowed in Fig. 7(a). From this spectrum, the presence of Ce and Bi can be observed. Due to their low melting point and also their low solubility in Fe, these elements segregate in the last part to solidification process, i.e., during eutectic reaction at the carbide/matrix interface (Ref 11, 13, 14, 17). Since these elements were not detected in matrix or the carbide phases, it is believed that they segregated to the boundary between the eutectic components.

3.2 Structure of the Alloys for the Different Additions of Fe-Ti-RE-Bi

The systematic addition of the modifier admixture had a significant effect on the microstructure of the as-cast alloys.

Figure 8 shows a set of optical micrographs where the effect of the modifier can be observed. From these figures, a ledeburite structure can be seen in the WCI without modifier. On the other hand, as the amount of Fe-Ti-RE-Bi is increased, the carbide structure becomes less continuous and is blocky or platelike (Ref 13). This structure is not completely present in all the alloys, but it is indeed present to some degree in all and can be considered representative for each sample. Such a microstructural change improved the mechanical properties of the alloys (Ref 30).

3.3 Modification Treatment

Under usual cooling conditions, hypoeutectic WCI solidifies first as dendrites of austenite. As the temperature de-

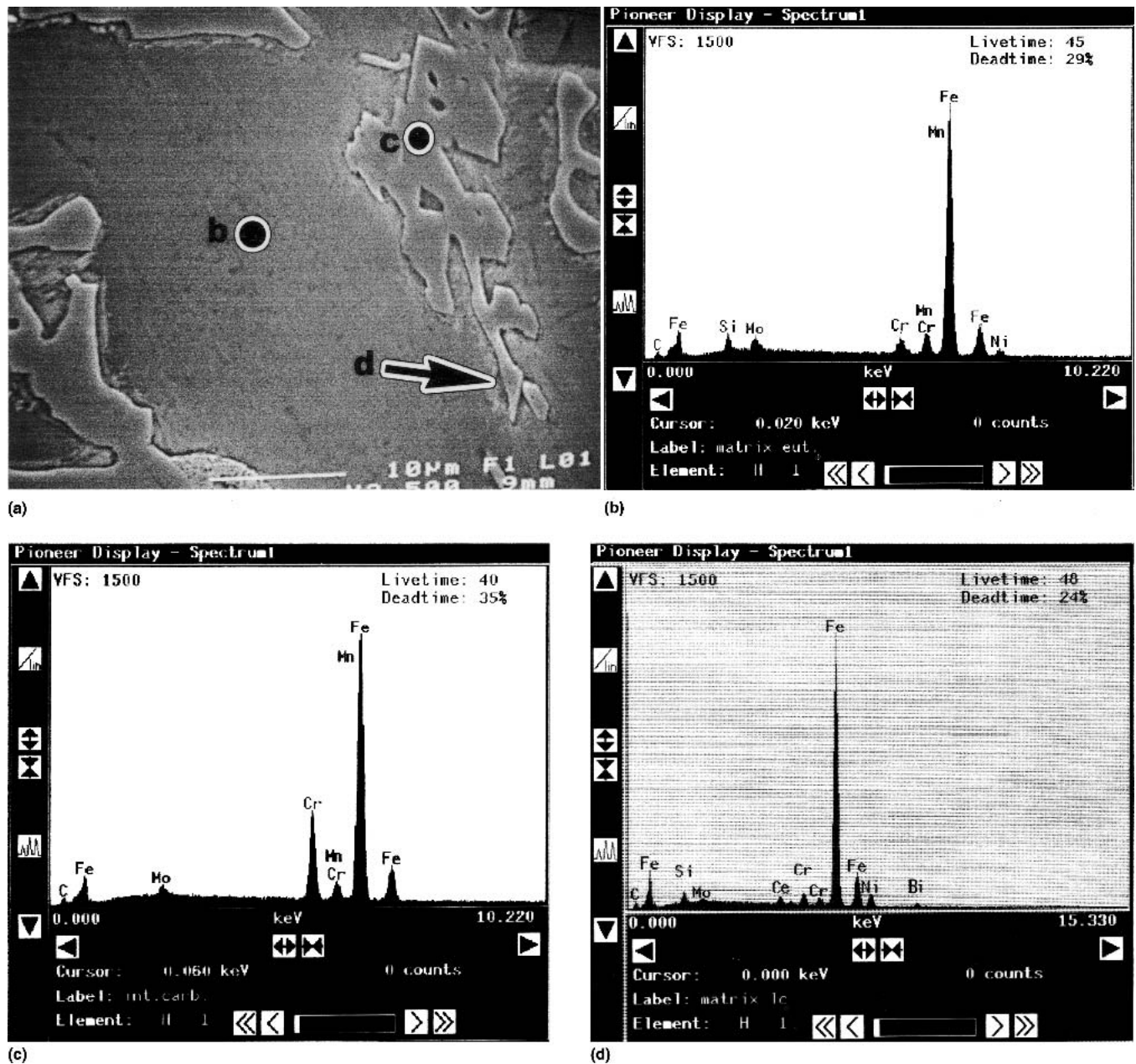


Fig. 7 Microanalyses of one WCI sample with 2% of the modifier admixture; the circles and the arrow indicate the areas where microanalyses were conducted. The spectra in (b), (c), and (d) correspond to the areas shown in (a).

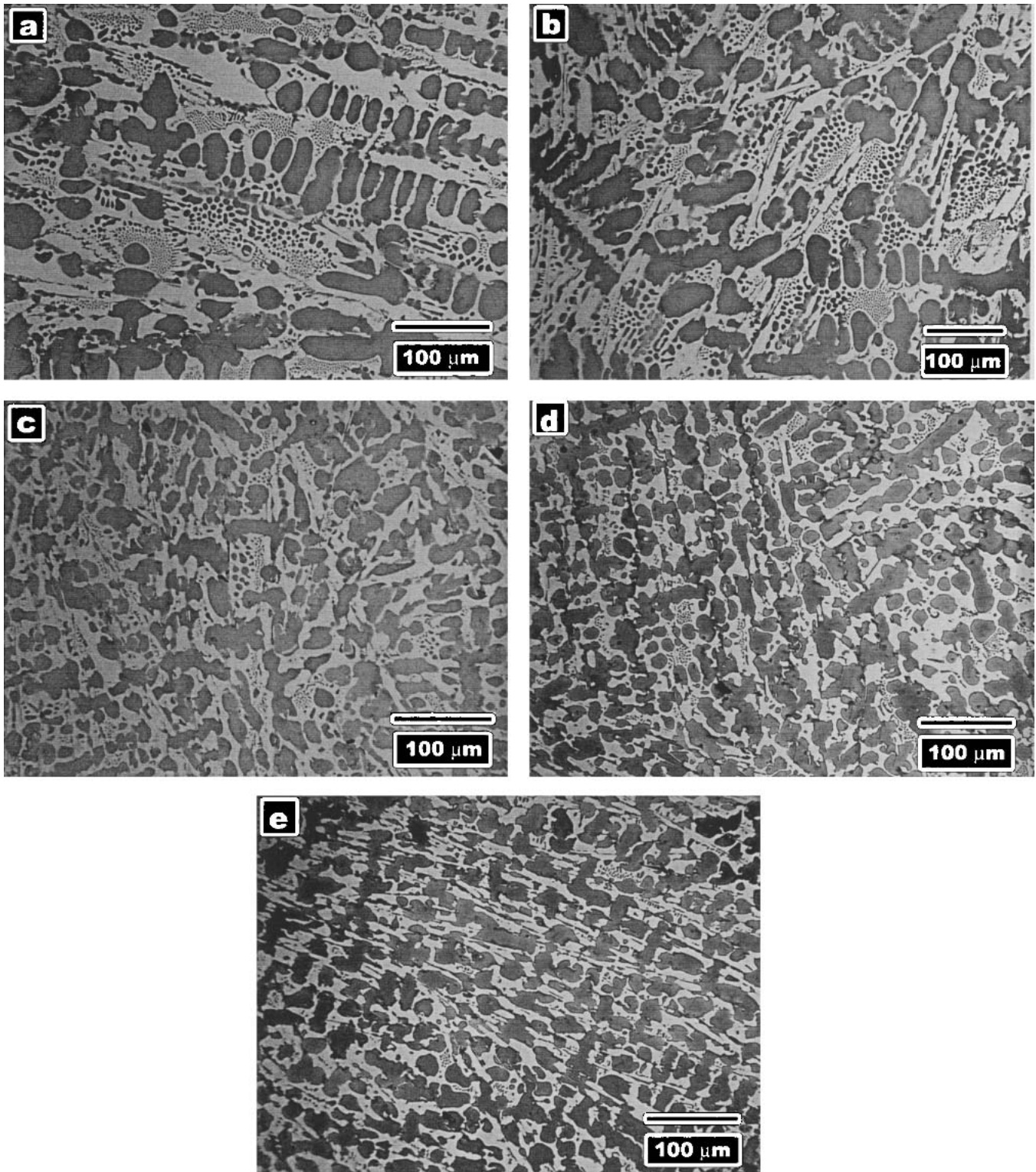


Fig. 8 Sequence of microstructures from the WCI with different admixtures of Fe-Ti-RE-Bi: (a) 0%, (b) 0.5%, (c) 1%, (d) 1.5%, and (e) 2%; light microscopy; Vilella's etch for 45 s

creases, the austenite dendrites grow until the eutectic reaction takes place. A ledeburite reaction, or a divorced eutectic, may occur depending on the liquid composition, ternary impurities, and cooling rate. In both cases, the first phase to develop during the eutectic reaction is cementite, which initially forms as small plates, and then branches and grows (Ref 31) in a preferred

crystallographic direction as eutectic solidification progresses. The eutectic carbides solidify into the ledeburite morphology when growth is dominated by enlargement of plates orientated in the flow of heat. The growth of plates oriented in other directions is limited (Ref 32). Otherwise, it solidifies as an anomalous eutectic such as a network, or a colony of

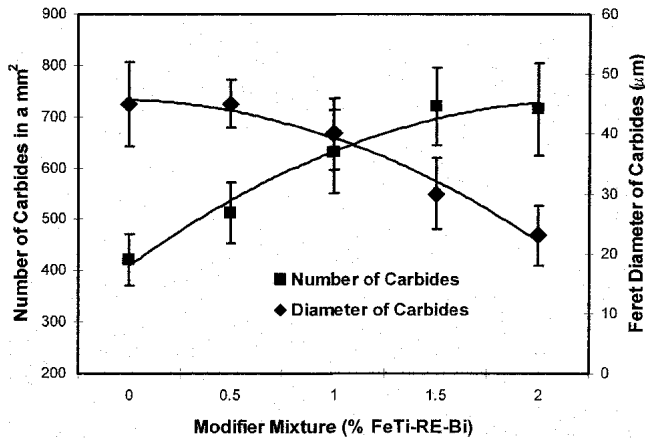


Fig. 9 Effect of the different additions of the modifier on the size and number of carbides per unit area

large plates largely interconnected like a skeleton inside the matrix.

If a larger fraction of austenite dendrites could branch into local areas within the liquid metal prior to the eutectic reaction, then eutectic cementite formation would be more isolated in these areas. This arises because cementite would have to nucleate and grow only within these interdendritic regions. There are two important factors to be considered to favor this process: A decrease in the amount of carbon equivalent (CE) to enable the formation of a high number of primary dendrites of austenite, or an introduction of sufficient nuclei to promote heterogeneous nucleation of austenite dendrites in liquid iron. Furthermore, if a divorced eutectic takes place instead of ledeburite, eutectic austenite will grow directly from the primary dendrites resulting in higher continuity with the matrix. This phenomenon limits cementite branching and also reduces the areas where cementite can develop. Other factors such as temperature and the nucleation rate of eutectic cementite may influence eutectic grain size, and also, the initial solidification and growth conditions. Therefore, it is always convenient, if possible, to create nucleation centers for eutectic cementite.

The above discussion is based upon theoretical hypotheses that agree with solidification experiments, and the results are well documented. It seems that these conditions occur when the modifier mixture is added to the iron. From Fig. 8, a fine structure is observed for iron with 2% admixture. One way for recording the level of refinement of the structure in cast alloys is by measuring the secondary dendrite arm spacing and/or the size of the particles comprising secondary phases. Due to the difficulty in identifying secondary arms, particularly for the irons with less than 1% of the admixture modifier, such a measurement was not undertaken in this study. A measurement of the carbide phase was done, however, to determine the size of the particles isolated in an area of the polished and etched surface. The number of these particles per unit area (i.e., 1 mm²) was also recorded. Figure 9 shows the results of the image analysis where the Feret diameter of eutectic carbides and the number of carbides in a square millimeter is plotted against the amount of Fe-Ti-RE-Bi added. From this figure, a decrease in the carbide size occurs as the modifier fraction increases. For the iron without modifier additions, the mean size of carbides is about 45 µm, while for the iron with a 2% admixture of modifier, the size of the carbide is between 20 and 25 µm. Regarding the amount of carbides observed, for iron

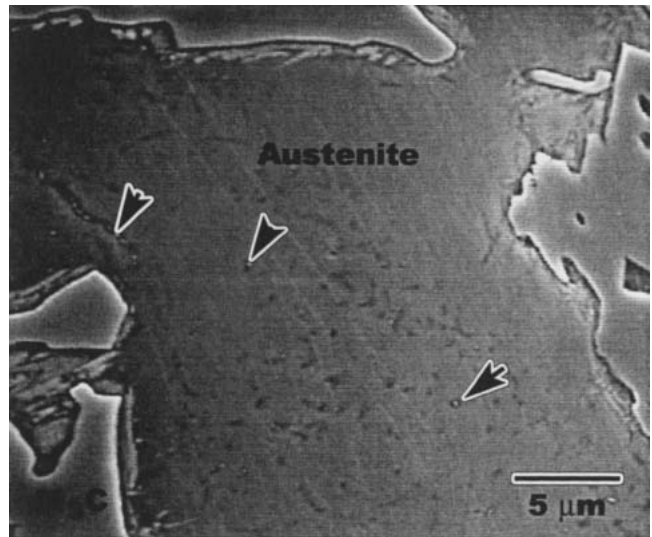


Fig. 10 SEM micrograph of the WCI with 2% of the modifier; the arrows indicate the presence of submicron particles within the proeutectic austenitic matrix.

without modifier additions, the number of carbides was about 430/mm² and increased up to 700 particles/mm² with 2% admixture modifier. The diminution of carbide size as well as the increase in number of particles in a specific area, as shown in Fig. 9, can be observed in the micrographs in Fig. 8.

3.4 Effect of the Elements Comprising the Modifier Mixture

3.4.1 Titanium. The observed characteristics in the microstructure of the modified iron revealed the effect of the single elements. Titanium additions, added as ferrotitanium (Fe-Ti), were used to create nucleation sites for austenite. These additions to the liquid iron promote the formation of Ti carbides and/or carbonitrides [i.e., TiC or Ti(CN)] during solidification. Such particles form as solid compounds while the alloy is still liquid. Therefore, it is likely they serve as nuclei for primary austenite dendrites. Some research has concluded that TiC [or Ti(CN)] can effectively act as nuclei (or at least as grain refiners) not only in steels but also in cast irons (Ref 33). From the current study, no evidence of dendrite refinement was observed but it is believed this could still have occurred. First, SEM studies on samples with 2% of admixture modifier revealed the presence of small particles in the austenitic matrix. Figure 10 shows the presence of these particles, with a size less than 1 µm (marked by an arrow in the micrograph). Microanalysis of the particle (Fig. 11) shows it is Ti-rich and may be a carbide or carbonitride. The presence of other elements in the spectrum may be due to the interaction of the electron beam with matrix since the particles are so small. Second, if the presence of these small particles promotes a higher austenite nucleation rate, a higher austenite volume fraction would be expected. This was observed when measuring carbide volume fractions for the different samples. Figure 12 shows the carbide volume fraction as a function of admixture modifier in the irons. A decrease in the carbide volume fraction from 42% to 35% was observed for the range of modifiers used. A decrease of this nature may be attributed to the TiC particles promoting high austenite precipitation, or perhaps it is the effect of the other elements from the mixture (RE and Bi), which may inhibit cementite growth.

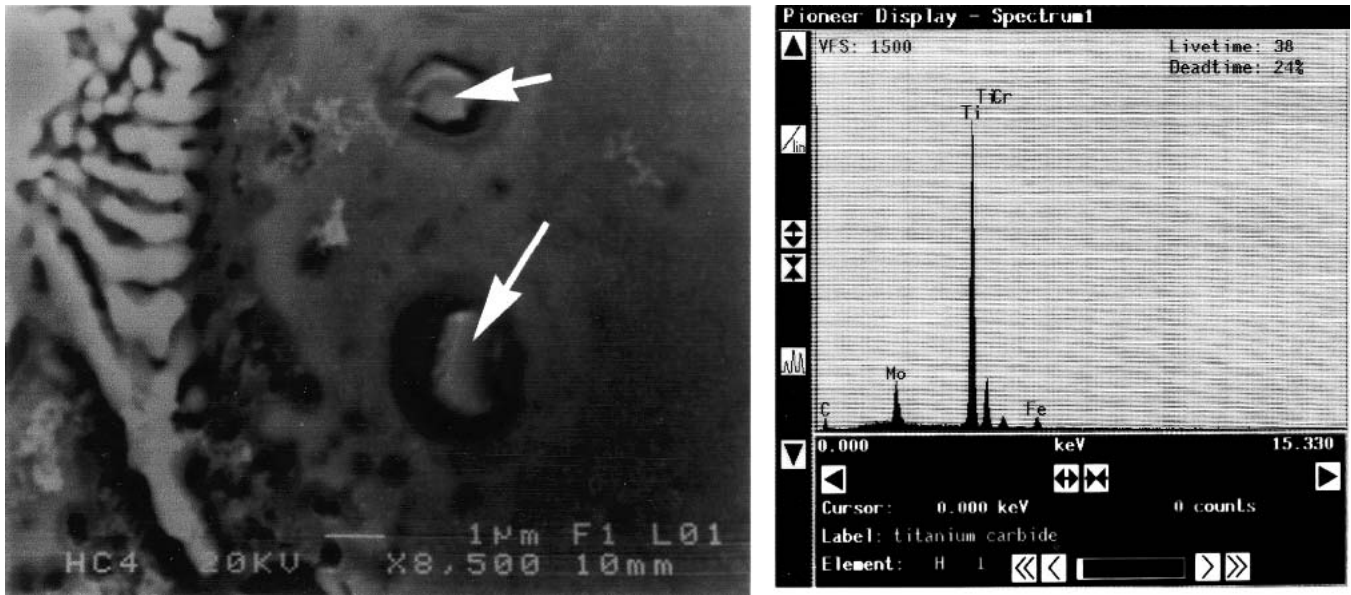


Fig. 11 Detail of particles (arrowed) found within the matrix and the EDS showing the qualitative composition of these particles; the high Ti content indicates the particles to be TiC or Ti(CN).

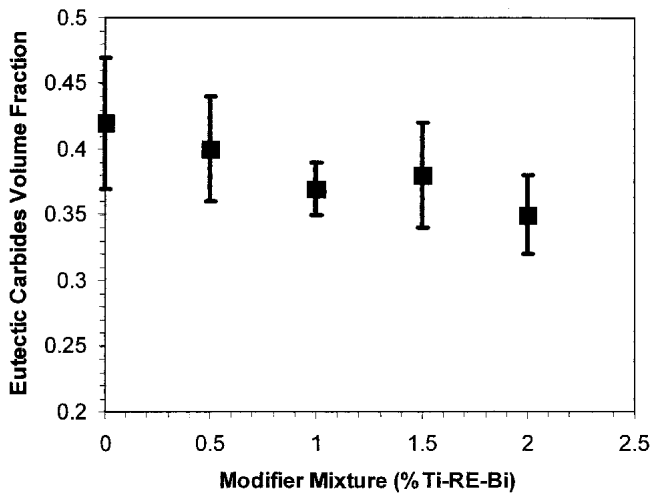


Fig. 12 Effect of the different additions of the modifier mixture (Fe-Ti-RE-Bi) on the eutectic carbide volume fraction in the structure of the WCI

It is also possible that the high number of isolated carbides in the modified irons could be attributed to higher cementite nucleation during eutectic solidification, thereby avoiding the ledeburite structure. Under these circumstances, TiC would act as nuclei for cementite. No evidence of this was found.

3.4.2 Rare Earths and Bismuth. The modified structure of the irons observed in Fig. 8 can be supported by segregation theories, i.e., segregating elements should have low solubility in both eutectic phases. From Fig. 7, it can be observed that RE elements and Bi segregate to the carbide/matrix interface. Consequently, some researchers (Ref 13-21) have proposed mechanisms to explain the change in carbide morphology in WCI.

A general assumption attributed to Li and Smith is that since the partition coefficient of the RE elements and Bi is very low in both phases, an accumulation of these elements ahead the solid-liquid interface is expected (Ref 16). At the local level,

the interface is microscopically planar as in pseudo-lamellar growth. As a result, the concentration of modifier elements will vary from place to place, being greatest at the center of each growing carbide plate. A high concentration of low melting point elements reduces the local solid/liquid equilibrium temperature that retards the carbide growth. The high concentration of solute will also promote division of the carbide into plates.

The modifying effect of RE in WCI has been well documented (Ref 13, 14, 17). A change of carbide networks into plates, or even bars, has been observed and the mechanism to explain that it is based on the divorced eutectic. In this case, the solute concentration ahead of the carbide promotes an undercooling, which favors austenite nucleation. As the austenite grows, it envelops the carbide. This means that once the carbides form, their growth is limited by an austenite shell, and subsequent growth occurs by diffusion through the austenite envelope. The change in the leading phase during eutectic solidification is another mechanism. Generally, in low-alloyed WCI, the leading phase is cementite. During eutectic solidification, where both phases grow almost at the same time, there is always a leading phase, which grows slightly ahead of the other. Therefore, this leading phase, which grows more freely, determines the growth conditions for the other phase. Liang and Su reported a change of the leading phase from cementite to austenite when adding 0.5% mischmetal to a WCI (Ref 17). They also found colonies of “blocky-like” carbides instead of the ledeburite structure. Their findings agree well with the results from the current study. It is inferred that carbide growth in the preferred crystallographic directions is restricted by the RE elements. Such elements can suppress carbide growth in certain directions as a result of segregation but they can not suppress precipitation. For example, Qingxiang et al. (Ref 13) reported that RE suppressed the M_3C growth in a Ni-Cr iron in the [010] direction, which is the preferred growth direction for this carbide. Furthermore, they observed preferred growth in the [100] and [001] directions, allowing the carbides to grow as isolated blocks.

Under these conditions, it is likely that the change in the carbide structure for the irons in the present work is due to the presence of RE and Bi at the carbide/matrix interface. Coupled with this is the fact that limiting carbide growth in some directions may have an effect on the carbide volume fraction (Fig. 12). Furthermore, the presence of RE and Bi at the solid/liquid interface during the first stage of solidification generates a high undercooling that promotes a refinement of the austenite dendrites, which in turn reduces interdendritic spacing where the eutectic reaction takes place.

Conclusions

Small admixtures of Fe-Ti-RE-Bi led to the ledeburite structure of cementite in low-Cr WCI, modifying the carbides into isolated blocks.

Structure refinement, measured by the secondary dendrite arm spacing, was observed as the amount of the modifier admixture was increased in the alloy. Such refinement was attributed to TiC or Ti(CN) which acts as nuclei for primary austenite dendrites. This was enhanced by the segregating effect of the RE and Bi, which generates constitutional undercooling and reduces the secondary dendrite arm spacing.

The effect of RE and Bi also influenced the morphology of eutectic carbides. Since RE and Bi segregated to the carbide/matrix interface and suppressed cementite growth in some directions, the carbide network was not as continuous.

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