JOURNAL OF MATERIALS SCIENCE 31 (1996) 1865–1871

Modification of hypoeutectic low alloy white cast irons

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In order to modify the continuous network of eutectic cementite normally found in low alloy white cast irons into a dispersive distribution, strategies of controlling the morphology of eutectic cementite by additives are discussed. Qualitative arguments are presented and applied to the development of a complex modifier REAlNTi. With the addition of this modifier to low carbon white cast irons nearly one fifth of the original eutectic cementite network can be modified into blocky particles in the plane of polish, and one third into isolated irregular ones. The experimental data shows that Ti(CN) may act as an effective nucleant or a grain refiner for eutectic cementite. The modification effects of rare earth elements are attributed to their abilities to purify the melt, promote divorced eutectic growth and refine the microstructure. Aluminium contributes to the modification by its purification effect.

1. Introduction

Three different metallographic morphologies of eutectic cementite can be observed in hypoeutectic low alloy white cast irons, these are (a) ledeburitic (b) network-like and (c) plate-like [1]. The plate-like modification usually forms under large undercooling conditions [1–3] or high contents of P [4], Si [2], Te, Sb [1] and rare earths (RE) [5–10]. Thus the ledeburitic and network-like modifications are the ones most generally observed in morphologies of low alloy white cast irons. They are generally produced in the form of a massive and continuous network (playing the role of matrix [11]) which ideally we would wish to break up since dispersed particles would help in the toughening of these alloys. A high temperature holding treatment can be effective in this regard [12], but it is a time and energy consuming process. The application of hot working techniques creates nearly ideal distributions of the eutectic cementites and has the secondary effect of deformation strengthening [13, 14]. However, the commercial application of it, has been limited due to inherent shape forming restrictions in addition to economic considerations. The modification technique in which a microstructure is altered by the addition of small amounts of additives has been investigated in the Al–Si and Fe–C systems. Since it is a convenient and economic approach, it would be an ideal approach for the Fe–Fe_3C eutectic we are investigating. It is reported that a number of additives have been attempted [1–10] but to date the modification effects achieved on the cementite are still far from the desired dispersive distribution and in addition the understanding of the process is poor. In this paper, strategies for controlling the morphology of eutectic cementites by additives are discussed, and a complex modifier is developed. It is shown that the network of eutectic cementite can be partially modified into isolated particles by the addition of additives.

2. General considerations of the modification treatment

Under usual cooling conditions hypoeutectic white iron solidifies with primary austenitic dendrites. As the temperature falls, the dendrites grow and develop until the eutectic reaction begins. Depending upon the liquid composition, ternary impurities and the cooling rate, either a divorced eutectic or a ledeburitic reaction will dominate. In either case, the cementite forms as a tiny plate, and will branch [15] or grow preferentially in some crystallographic direction as the reaction proceeds. These preferred directions are characteristics of the cementite structure. When the growth is characterized by extension of the plates that usually lie in the heat–flow direction together with limited rod-like growth perpendicular to the plate [16], it solidifies as quasi-regular ledeburitic. Otherwise it solidifies as anomalous structures such as the network-like, plate-like or other irregular types. Among them the network-like may be regarded as being occasioned by the bridging of the quickly developing branches from different cementites, and the plate-like can be considered as a modified form of the former in that the network is composed of a lot of tightly connected cementite plates as is revealed in [1].

With the foregoing understanding as a starting point we are led to the following strategies for controlling the morphology of eutectic cementites. Firstly, the solidification of hypoeutectic white irons proceeds with the development of austenitic dendrites. Hence, if more austenitic dendrites could be intentionally promoted and bridged with each other in local areas of the melt prior to the eutectic reaction, the formation of isolated eutectic cementites should become more favourable there, because the cementite has to nucleate and grow within the isolated interdendritic space in such a case. Two important factors for this are a low carbon equivalence (CE) to provide large amount of primary austenites and also the creation of sufficient nuclei to allow heterogeneous nucleation to occur throughout the melt. Moreover, if the divorced eutectic reaction occurs instead of the ledeburitic reaction the eutectic austenite will grow directly from the primary austenites. This would not only contribute to the bridging but also narrow the free space in which the eutectic cementite can develop, thus favouring the modification. Secondly, like all solidification processes the nucleation temperature and the rate of nucleation of eutectic cementite can influence the eutectic grain size and the solidification conditions under which initial growth occurs. Therefore, attempts should also be made, if possible, to create sufficient nuclei for eutectic cementites. It is probable that apart from providing nucleating sites these nucleants could also indirectly suppress the initial preferential growth of the eutectic cementites due to the decreased undercooling. In Fe–C–V [8], Fe–C–Cr–V [17, 18] and Fe–C–Cr–Nb [19] white irons it has been confirmed that an effective nucleant for VC or NbC could significantly lead to the spheroidization of both primary and eutectic VC and the formation of highly compact eutectic VC. Thirdly, the morphology of growing crystals depends upon the growth conditions. The cooling condition is always one of the most significant factors. In this work all the specimens will be prepared by green sand casting processes with a view to practical applications. A cooling rate slower than that but not obviously affecting the number of nucleants should not be detrimental to the modification effects, because lower undercooling is generally unfavourable to the growth of perturbations on the solid–liquid interface. Besides, in order to produce blocky or spheroidal growing crystals a higher surface tension of the melt is always desirable in stabilizing the spherical surface by suppressing the growth of perturbations on it. Sulfur and oxygen, the former in particular, strongly influence the surface tension of the Fe–C melt. Evidence has been presented that spheroidal graphite and VC can be produced respectively in high purity Fe–C–Si or Ni–C [20] and Fe–C–V alloys [8] owing to the extremely low levels of sulfur and oxygen. So, strong desulfurizers and deoxidizers will be treated as an integral part of the modifier. In addition to its above mentioned effects a divorced eutectic growth condition is also needed in order to avoid a ledeburitic structure. This can be realized by adding RE elements to the low CE white iron melt [5–10].

Potential modifiers will be developed in the next section using these arguments.

3. Determination of the potential modifiers

3.1 Nucleating agents for primary austenite

The selection of suitable nucleants remains to a large extent an empirical exercise [16]. For austenite, numerous investigations have indicated that titanium carbide/nitride can act as an effective nucleant (or at least as a grain refiner) in either steels or cast irons. This is reviewed by Wallace and Ruff [21, 22]. In this paper, we will follow this conclusion and choose TiC or Ti(CN) as the nucleant for proeutectic austenite in hypoeutectic white irons.

3.2 Nucleating agents for cementite

The following empirical conditions are used to define a potential nucleant [16]; (i) The nucleant shows metallic bonding. (ii) There is a high melting ratio between the nucleant and the solid. (iii) A low nucleant–solid interfacial energy σ_{n-s} exists. Since quantitative evaluation of σ_{n-s} is still impossible in most cases, we will work in terms of lattice mismatch, a factor that would strongly influence σ_{n-s}. As pointed out by Flemings [23], if the effectiveness of a nucleant is to be measured, not in how much it reduces undercooling for nucleation, but in how effectively it refines the grain, the effectiveness of various commercial grain refiners can all be rationalized on the basis of lattice mismatch ideas. Bramfitt's planar disregistry model [24], a modified form of the Turnbull–Vonnegut model [25], will be utilized here to help to select the nucleants for cementite. This model has been successfully used to explain the heterogeneous nucleation phenomena in several systems [24, 26–31]. It can also be used to understand the use of TiC as a nucleant for austenite.

The structure of cementite is orthorhombic with its smallest structure unit being a triangular prism, as is shown in Fig. 1 [32, 33]. For later convenience, we define the top plane ABC in this prism as the $(0\,0\,1)^+$ plane, and the bottom plane DEF as the $(0\,0\,1)^-$ plane. These two planes were considered as the low-index planes when the planar disregistry was calculated between cementite and the selected compounds and the results of the calculations are listed in Table I. From these disregistry values it is inferred that titanium or vanadium carbide/nitride may act as a potential nucleating candidate for cementite. ZrC/ZrN, WC and

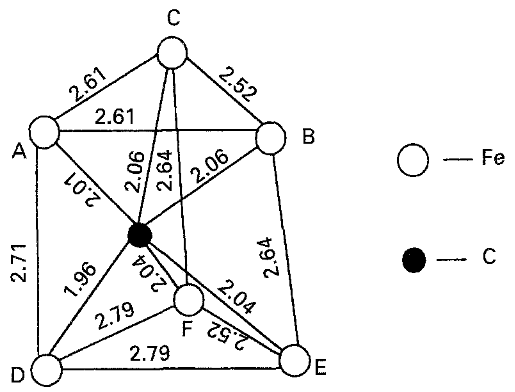

Figure 1 The structure unit of cementite.

BN are also likely, but each of them favours only one of the $(0\,0\,1)^+$ and $(0\,0\,1)^-$ planes compared with Ti or V carbide/nitride. Moreover, the density ratios of WC and BN to the alloy melt are not so attractive either. As TiC is more stable than VC and the others, it is then preferable.

3.3 Elements favouring the formation of isolated cementite particles

Rare Earth (RE) elements have a great chemical affinity with sulfur and oxygen and thus they would increase the surface tension of the melt. They also promote divorced eutectic growth, refine the microstructure including the primary austenitic dendrites and cause breakup of the eutectic cementite network [5–10]. These modification effects are believed to be occasioned by the RE elements screening the crystal growth front due to their poor solubility in both austenite and cementite [5, 7]. We also note that a higher silicon content may favour divorced eutectic ($Fe–Fe_3C$) growth. Thus a type of RE–Si–Fe commercial alloy containing 31% RE, 40% Si, and less than 6% Ti is adopted for these studies. In addition aluminium which is a strong deoxidizer is also adopted. It should be noted that small additions of aluminium are reported to refine the austenitic dendrites and increase their quantity in cast irons [38–40].

Thus the finally selected potential modifier will consist of RE, Al, N, Ti, and is named REAlNTi for easy identification.

4. Experimental procedures

4.1 Materials and modification treatment

The Mn–Cr–Cu alloyed white cast iron, having the nominal compositions (wt%) C2.20–2.80, Si1.20, Mn4.0, Cr1.80, Cu1.50, S0.053, P0.065 and Fe balance, was melted in an induction furnace with the superheating temperature of 1500 ± 20 °C. The modification treatment was conducted firstly by adding 0.3% Al to the alloy melt, and then 1.0% Fe-30Ti and 0.5% N–Mn–Fe (4% N) alloys. Following that, the melt was tapped into a ladle that contained a 2.5% RE–Si–Fe alloy. After being stirred, the melt was poured into green sand molds of round bars (20 mm diam. × 120 mm). Five batches of the alloys with the carbon content varying in the range of 2.20–2.80% were treated. In order to distinguish the modification effects of different ingredients sections of one batch were also treated with either RE (RE–Si–Fe) or N–Ti (Ti–Fe + N–Mn–Fe). Samples for examination and analyses were all cut from the middle of the small ingots, and all were etched in 4% nital unless otherwise specified.

4.2 Morphological parameter (SF_i)

To quantify the morphological changes of the eutectic cementites, a parameter defined as roundness area fraction (SF_i) is utilized. $SF_i = A_i/A_0$, where SF $(2(\pi A)^{1/2}/L_P$ [39]) denotes the roundness of particles, A_i is the area of the eutectic cementite with a certain roundness, and A_0 is the area of the total eutectic cementites. Using the classification of the morphology of particles in terms of the roundness [41], the shape of eutectic cementite can generally be distinguished into three types. Namely, the network-like and lediburitic as $SF < 0.25$, the irregular as

TABLE I Crystallographic planar disregistries between different compounds and cementite*

Compound	Prototype	Lattice parameter a_0 Å		Planar disregistry (%)				Melting point (°C)	Density ($g\,cm^{-3}$)
				$\delta^{(0\,0\,1)NaCl}_{(0\,0\,1)^-Fe_3C}$	$\delta^{(0\,0\,1)NaCl}_{(0\,0\,1)^+Fe_3C}$	$\delta^{(1\,1\,1)NaCl}_{(0\,0\,1)^-Fe_3C}$	$\delta^{(1\,1\,1)NaCl}_{(0\,0\,1)^+Fe_3C}$		
TiN (TiC)	NaCl	0.4246 (0.4327)		6.91	7.16	11.28	13.53	3290 (3060)	5.43 (4.25)
VN (VC)	NaCl	0.413 (0.417)		7.62	5.47	10.08	12.34	2340 (2800)	6.10 (5.36)
ZrN (ZrC)	NaCl	0.456 (0.4696)		6.15	14.189	14.58	16.97	2960 (3540)	6.93 (6.9)
		a_0	c_0	$\delta^{(0\,0\,0\,1)hex}_{(0\,0\,1)^-Fe_3C}$		$\delta^{(0\,0\,0\,1)hex}_{(0\,0\,1)^+Fe_3C}$			
WC	WC	0.29036	0.2837	4.03		14.53		2785	15.70
BN	ZnS(w)	0.250	0.666	10.429		3.41		3000	2.34
AlN	ZnS(w)	0.3114	0.4986	11.57		20.31		2800	3.10

*The data are all taken from [34–36]. When the disregistry is calculated the expansion of the lattice parameter is considered following Bramfitt's method [24]. The melting point of cementite is 1250 °C [34]. The density of white iron melt is about 7.3 $g\,cm^{-3}$ at 1200 °C [37].

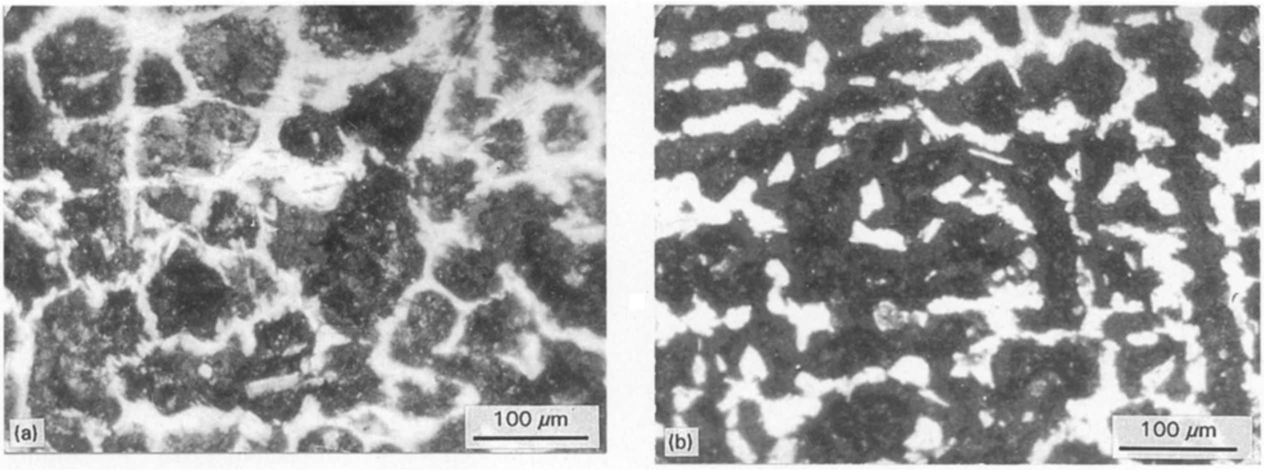

Figure 2 The morphology of eutectic cementite in hypoeutectic white cast irons; (a) base iron and (b) modified.

$SF = 0.25$–0.50, and the blocky and nodular as $SF = 0.50$–1.00. Consequently, A_1, A_2 and A_3 refer to the area of the three types of eutectic cementites, respectively. SF_1, SF_2 and SF_3 are then correspondingly the fraction of network-like, irregular, and blocky eutectic cementites. All the measurements were conducted using a Cambridge Q900 microscope, and for each specimen 20 viewing fields, which exclude the surface cooling layer of the specimen, were analysed under a magnification of 200 times.

5. Results and discussions

In both the base irons and in the treated irons it is noted that in the surface cooling layer the strong heat flows present mean that the eutectic cementites solidify as parallel or radiative arrays of strips or lamellae. Clearly, the cooling condition has a dominant influence on the microstructure in this layer compared with the modifier. Away from this layer the eutectic cementite is typically network-like as shown in Fig. 2a, in which 80.72% of the eutectic cementite is network-like; 13.15% is irregular; and 5.53% is blocky (Batch no.1 in Table II). However, in the treated irons this network structure is greatly altered (Fig. 2b). The networked eutectic cementite reduces from 80.72% to 36.49%, whilst the blocky and irregular eutectic cementites increase from 5.33% to 23.87% and 13.95% to 39.73%, respectively (Batch no.2 in Table II). All the other batches of alloys that contain different carbon contents present very similar morphological changes when modified. The quantitative analyses of these morphological changes are also given in Table II. The optimum modification effect occurs in the alloy with the lowest carbide fraction (5.53%), in which only 22.3% of the eutectic cementite remains network-like after modification. On average, nearly half of the original networked eutectic cementite can be modified into blocky and irregular particles in all the treated alloys.

TABLE II Morphological changes of the eutectic cementites in different batches of alloy

Batch no.	$V_f^{Fe_3C}$ (%)	SF_1 (%)	SF_2 (%)	SF_3 (%)	Modifier
1	11.16	80.72	13.95	5.33	Base iron
2	11.84	36.49	39.73	23.78	REAlNTi
3	5.33	22.63	44.20	33.17	REAlNTi
4	6.83	36.27	43.11	20.62	REAlNTi
5	7.00	36.69	47.02	19.30	REAlNTi
6	9.52	40.48	41.23	18.29	REAlNTi
6	9.41	61.10	27.57	11.33	RE
6	9.26	69.81	21.80	8.39	N-Ti

In order to look into the modification mechanisms, a careful examination was made of the microstructure that showed the best modification effect. As can be seen in Fig. 3a, some modified blocky eutectic cementites do exist in the space of the austenite dendrites, which have apparently formed a solid network structure in certain areas of the alloy melt before the eutectic reaction. Although the interdendritic space could indeed restrain the eutectic cementite from growing and developing outside it, the shape of the eutectic cementite does not depend completely on it. Often in each space several blocky eutectic cementite particles are formed as is shown in Fig. 3a. This is a significant difference to the behaviour observed in the untreated irons. Fig. 3b is taken from the same specimen as Fig. 3a after slow etching in 4% nital to demonstrate the blocky eutectic cementite. It is interesting to note that in a number of the blocky eutectic carbides there exists a tiny black spot (Fig. 3a, b). By scanning electron microscopy (SEM) it is revealed that the spot is a black square compound, which, as indicated in Fig. 4, can exist either in the centre of the blocky eutectic cementite or at the edge of it. An energy dispersive X-ray analysis study detected that the titanium concentrates in this black compound up to 85.37 wt %. Also, the use of a wavelength dispersive spectrometer (WDS) showed that N and C are also concentrated in it (Fig. 5). On the basis of these observations we believe that this compound is Ti(CN), which can also be found in the transformed austenites (Fig. 6) in the treated alloys. It is tempting to speculate that the Ti(CN) retained after the proeutectic solidification has played a certain role in nucleating the eutectic cementite.

Experiments were then conducted to identify the modification effects due to the RE elements and those due to the Ti(CN). The results are shown in Fig. 7 with

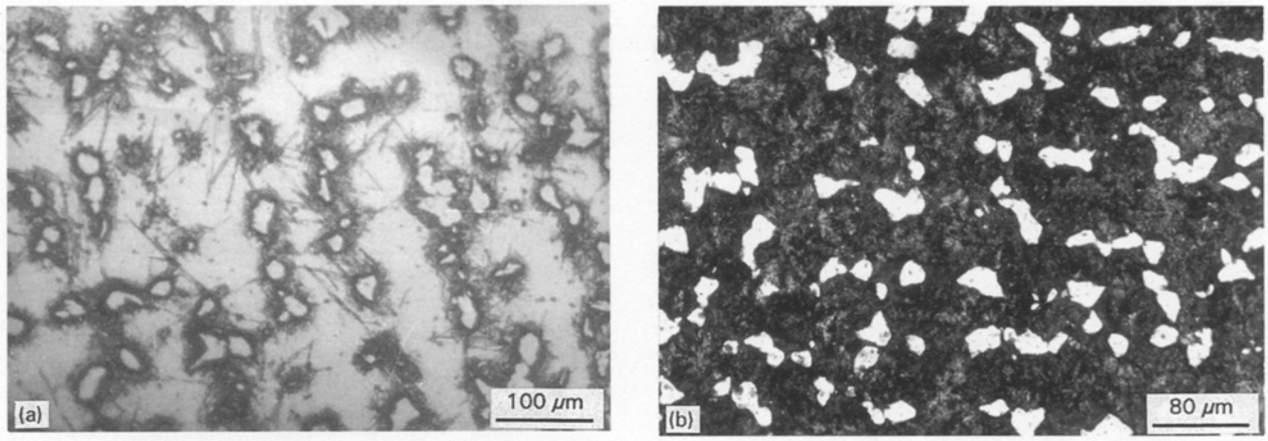

Figure 3 Distribution of the modified eutectic cementite particles; (a) etched in 1% picric plus 5% HCl in methanol, (b) etched in 4% nital.

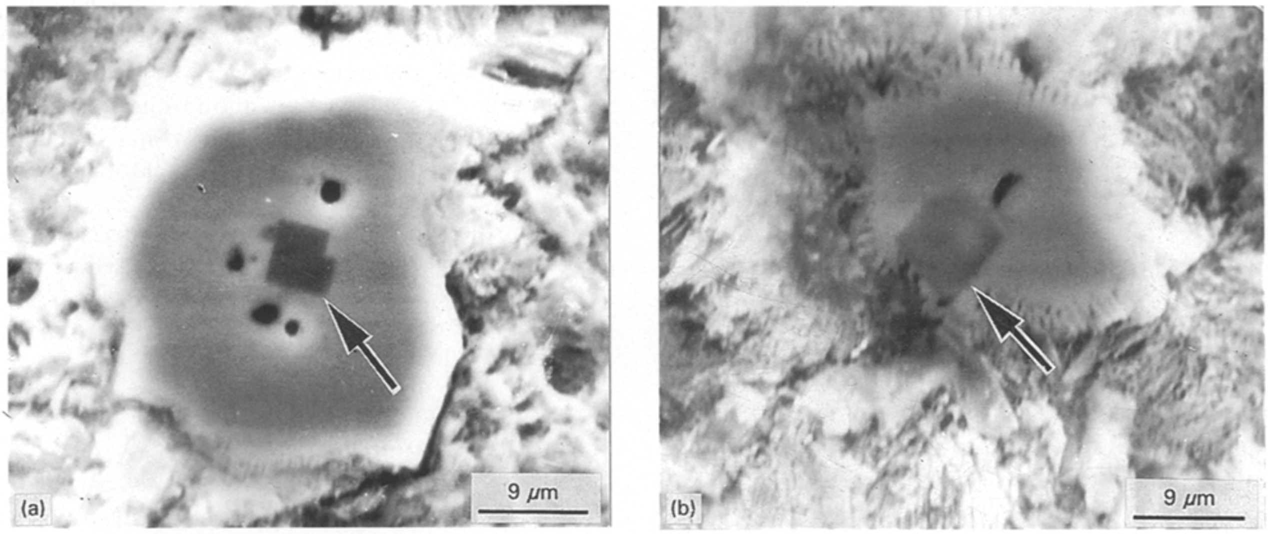

Figure 4 The black and square compound; (a) in the center of the and (b) at the edge of the eutectic cementite.

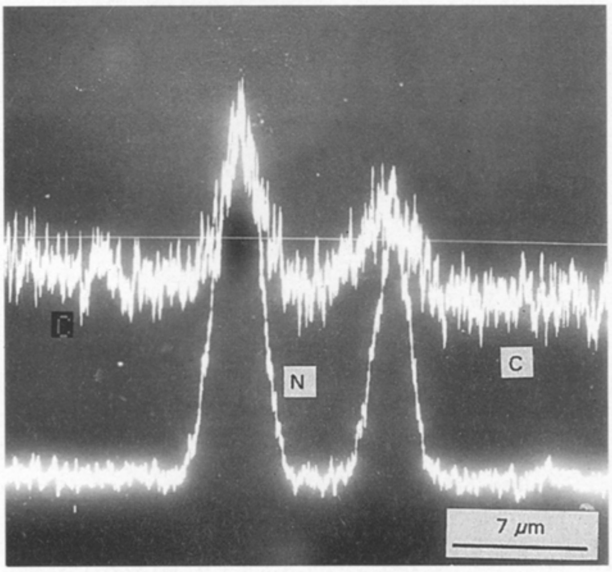

Figure 5 Distribution of N and C in the black compound by WDS.

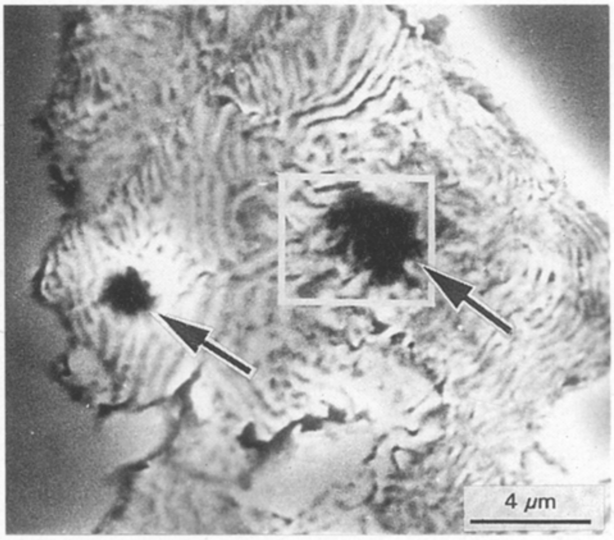

Figure 6 Ti(CN) detected in transformed austenite.

the quantitative analyses listed in the bottom two lines in Table II. The observed modification effects due to the RE elements are generally in agreement with other reported studies [5–10], and are better than those of Ti(CN). This can be attributed to the comprehensive effects that RE elements exert on the solidification process of white irons [5–10]. It is seen that both RE elements and Ti(CN) can promote a certain amount of isolated eutectic cementites, but in both cases it is far

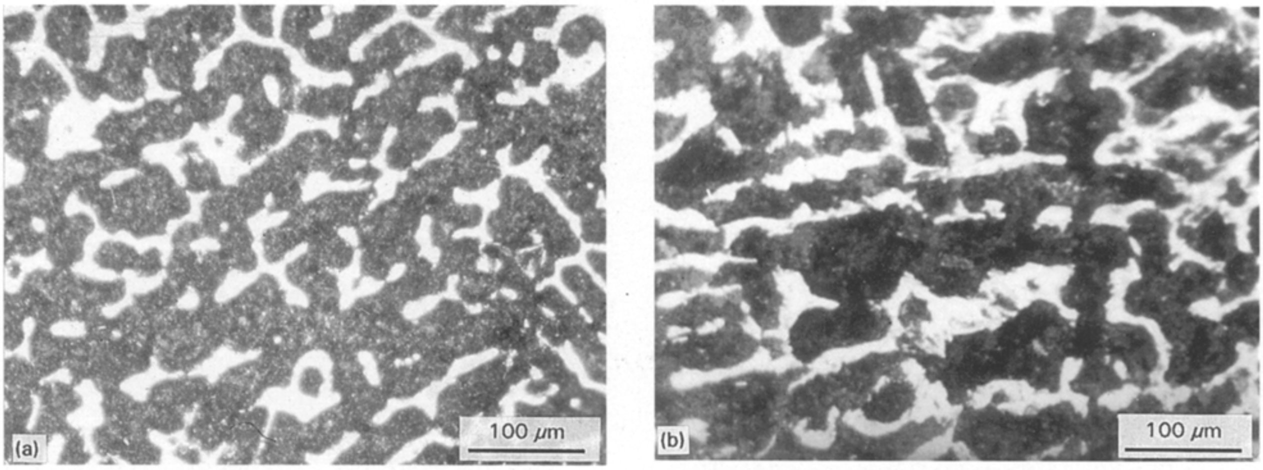

Figure 7 Morphology of the eutectic cementite after modification; (a) by RE and (b) by N–Ti (Ti(CN)).

from the amount created by the complex modifier REAlNTi. The addition of Ti–Fe and N–Mn–Fe to the alloy melt could result in the refinement of the microstructure and produce some modification effects, but Ti(CN) in itself cannot significantly change the morphology of the eutectic cementite as much as it changes that of VC [8, 17, 18] or NbC [33]. The morphology of eutectic cementite depends more upon the growing process. We conclude that the modification mechanism of the complex modifier, is a combination of (a) the physical restriction of the austenitic dendrites modified by Ti(CN) and RE elements, (b) the nucleation effects of Ti(CN), (c) the divorced eutectic growth condition created by low CE and RE, and (d) the purification effects of RE and Al.

Although the proposed modification model works within the range of low CE white irons, it appears that not all the networked eutectic cementite can be modified into isolated particles even when its fraction is as low as 5.33%. Normally, we would expect the modification treatment to become more difficult as the fraction of the eutectic cementite increases, since the network structure will consequently grow more massive. Thus more sophisticated models or theories need to be developed for better modification effects of the Fe–Fe_3C eutectic. As a guide we expect that a complex modifier would be more effective than a single modifying element.

A desirable property of a eutectic cementite nucleant is that it should not nucleate austenite thus only providing nucleating sites for eutectic cementite during the eutectic reaction. However, we have not yet been able to find a material with such a property (ZrC/ZrN and VC/VN are also known to act as nucleants for austenite [21, 22]). For this reason, no attempt has been made in the present paper to distinguish between the modification effects caused by Ti(CN) nucleating eutectic cementite from that by Ti(CN) nucleating austenite.

Finally, both the morphology and the distribution of the eutectic cementites are treated as two dimensional phenomena in this paper. Thus it is possible that the eutectic cementites which appear discontinuous in the 2D polish plane might actually be continuous within the bulk specimen [3].

6. Conclusions

(i) The normal continuous network of the eutectic cementite in hypoeutectic low alloy white cast irons can be partially modified into blocky and irregular particles by additives under green sand casting conditions.

(ii) From the experimental results it is inferred that Ti(CN) may act as a nucleant or a grain refiner for eutectic cementite.

(iii) The proposed modification model applies more favourably to low CE white cast irons.

Acknowledgement

Helpful discussions with Dr. Zhu Jun, Department Materials Science Engineering, Kyushu Institute of Technology, Japan, are appreciated.

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Received 4 April 1995
and Accepted 8 September 1995