CHAPTER

CONTROLLED SOLIDIFICATION TECHNIQUES

17

17.1 CONVENTIONAL SHAPED CASTINGS

Most castings will solidify from the outside and will grow grains that will finally merge and consolidate in the centre of the casting. Central regions that form hot spots not in communication with a source of feed metal will cause some local shrinkage that may take the form of internal porosity if the porosity can be initiated, meaning in practice that bifilms will be required to be present. Alternatively, in the absence of bifilms (i.e. very clean metal), local shrinkage may create external porosity in the shape of surface sinks.

Although many foundry texts recommend the provision of a feeder to counter this problem, my personal approach is to avoid the provision of a feeder if at all possible. I prefer to use some kind of chill. This may take the form of a substantial block of cold metal forming part of the mould, or might even be an internal chill around which the melt freezes. More usually, however, I opt for the provision of cooling fins.

Cooling fins work excellently for high-conductivity alloys such as Al and Mg alloys and some Cu-based alloys (although not particularly well for aluminium bronzes). Unfortunately, the concept works so poorly for steels that fins cannot be recommended. For grey cast irons, the situation is somewhat intermediate, with fins often working sufficiently well to strike back white iron locally into the casting; such an effect has been used historically for the tips of cams on camshafts for automotive engines, although there seems to have been a move away from this technique to the placing of shaped chills in the interests of greater reproducibility and control. More recently, there is interest in the production of wear faces on cams not by solidification control but by subsequent heat treatment of the casting by local application of rapid induction heating.

In the special case of conventionally poured and solidified polycrystalline turbine blades, the fundamental problem in the casting of these alloys in a conventional vacuum induction furnace is seen in Figure 16.30. The height 'h' of the fall of metal into the mould, followed by freezing from all directions, means that bifilms created by the fall have little chance to escape, but become trapped in the casting. As a result of them being pushed by advancing dendrites, they naturally finish up in grain boundaries. As a result, during creep or other tensile failure modes, the castings fail by 'cavitation' at the boundaries, the growing cavities gradually joining to lead to complete rupture. This explains the relatively poor properties of polycrystalline equiaxed turbine blades compared with other grain structures (Figures 17.1 and 17.2) as described in the following sections.

17.2 DIRECTIONAL SOLIDIFICATION

The unidirectional solidification of castings has a long history, promoted initially by the desire to obtain high soundness; the high temperature gradient shortening the pasty zone so that feeding could occur to the roots of the dendrites with greater efficiency. Directional solidification (DS) is mostly used to imply unidirectional solidification. It has been used for permanent magnet manufacture, but its main use has been for turbine blades for jet engines.

In practice, DS has nearly always been carried out vertically in an upward direction. It was not suspected that this particular mode of freezing would yield an additional important effect; a significant reduction in the bifilm population

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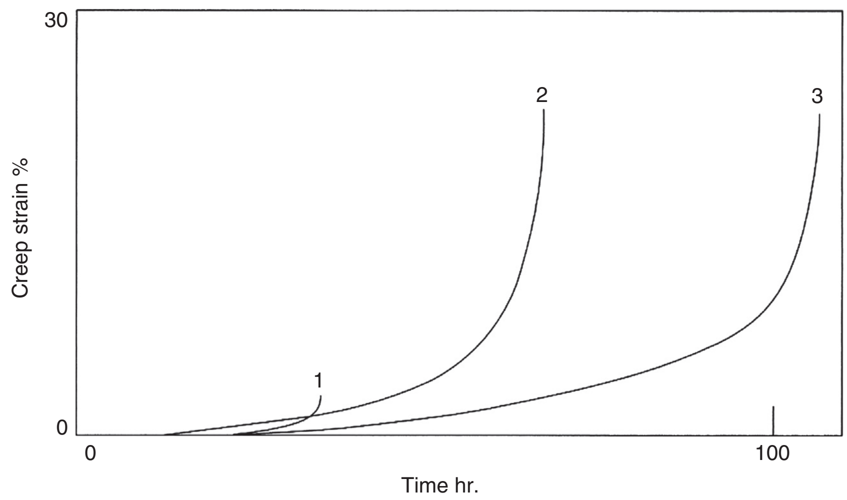

FIGURE 17.1

Creep behaviour of Ni-base alloy Mar M200 at 980°C and 207 MPa cast as (1) conventional equiaxed structure; (2) directional solidification (DS) columnar structure; (3) single crystal.

After Versnyder and Shank (1970).

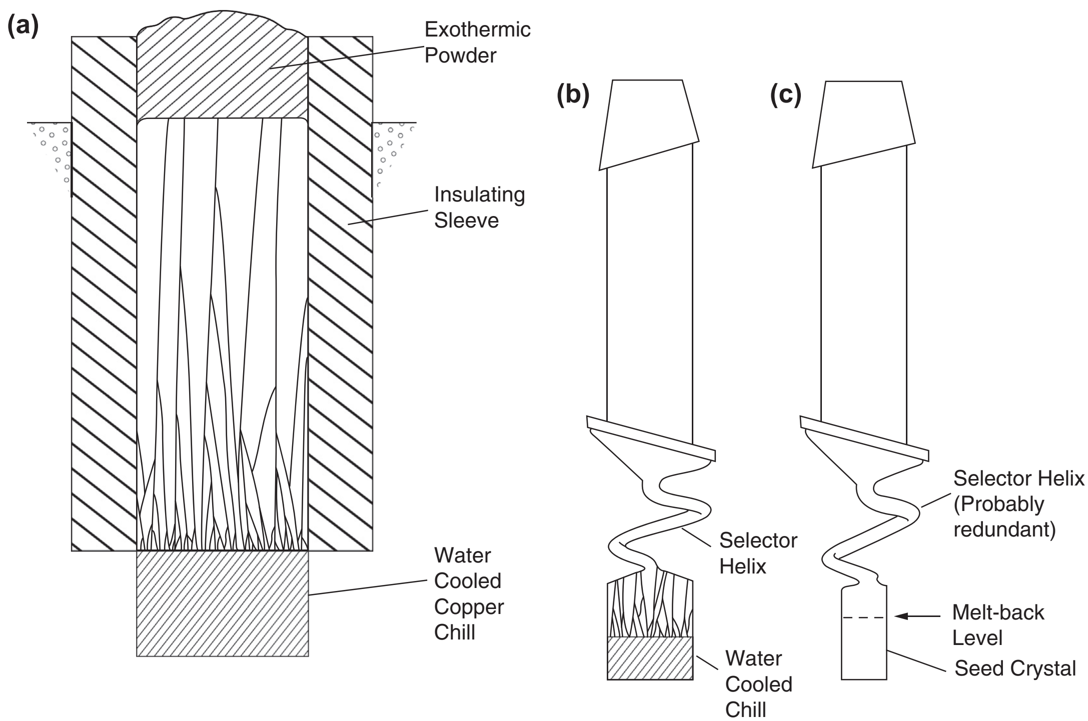

FIGURE 17.2

(a) Directional vertical solidification of a steel casting *(after Polich and Flemings, 1965)*; (b) single crystal by selection from a chilled DS base; (c) single crystal grown from a seed and therefore accurately oriented.

This occurs partly (1) by the vertically directed freezing allowing bifilms to float upward, away from the freezing front; and (2) because of the unusually long time available for this flotation because of the relatively slow rate of advance of the front during DS; and (3) any residual bifilms will have the best chance to be pushed ahead of the advancing solidification front, thereby keeping the solid relatively free from serious defects.

Work at MIT first published in 1965 for steel castings, by various combinations of the authors Nereo, Polich and Flemings, quickly showed benefits to the strength, toughness and fatigue resistance revealed by directional solidification. Later work (Flemings and Mehrabian, 1970; Hurtuk and Tzavaras, 1975) has further confirmed the beneficial effects of DS for steel castings. The melting and pouring in this work was all carried out in air into relatively simple moulds (Figure 17.2(a)) approximately 100 mm diameter and 230 mm tall so that significant oxidation and bifilm contamination of the melt would be expected, particularly during the final pour into the moulds.

Significantly, the benefits to properties were a strong function of dendrite arm spacing (DAS) in non-directionally solidified castings but *not* for DS castings. This result is difficult to explain unless one assumes that bifilms are trapped amongst grains during equiaxed freezing but not in DS material. Thus for the relatively clean DS material, the properties are not affected by DAS as seen in Figure 9.26 confirming that mechanical properties are a function of bifilms; the *apparent* strong effect of DAS in most castings and many alloys is the result of presence of unfurling bifilms.

This work on steels coincided with a technically analogous, but, as it turned out, far more important and exciting breakthrough in Ni-base superalloys. Versnyder and Shank (1970) review the early development of DS and single crystals for blades and vanes for use in aircraft turbine engines. Their world-famous summary of their creep behaviour is seen in Figure 17.1. These early studies also indicated that DS polycrystal castings possessed up to 10 or 100 times greater fatigue life compared with equiaxed polycrystal castings (Leverant and Gell, 1969). Despite this spectacular advance, Zhou and Volek (2007) draw attention to the fact that performance, particularly in creep, remained limited by crack formation along both the longitudinal and transverse grain boundaries of the DS castings, although the failure of transverse boundaries is most usually cited as the driving force for the development of single crystals, in which, of course, all grain boundaries were eliminated. Because, of course, grain boundaries are enormously strong, it seems most likely that these grain boundary failures were not the result of failures of the grain boundaries themselves but the result of bifilms segregated to the boundaries by dendrite pushing. This seems especially likely in view of the extreme turbulence associated with the manufacture of these vacuum cast blades, so that the presence of bifilms is guaranteed. They are such serious defects that it is reasonable to suppose they will have a major effect on properties.

If, as seems reasonable and logical, it is accepted that grain boundaries are strong, the Versnyder and Shank result shown in Figure 17.1 cannot be explained by conventional metallurgy. The DS and single crystal technology for turbine blades in jet engines is, perhaps, the best evidence yet for the profound effect of bifilms in castings.

It is sobering to realise that the poor properties shown by the equiaxed casting in Figure 17.1 are typical of the bifilm-crammed structures of conventional castings which we produced in our aluminium, steel and titanium foundries every day; in fact, of course, in practically all our foundries of all types. This is where metallurgy is today; we are currently producing poor metallurgical products which have enormous potential for improved properties as we achieve progressively better ways to bring bifilms under control for the first time.

17.3 SINGLE CRYSTAL SOLIDIFICATION

The benefits of DS are brought to complete fruition in single crystal growth.

By the provision of a crystal selector such as a helical channel (known as the ‘pig tail’), a single grain orientation can be selected close to the <001> favoured dendrite growth direction. Grains growing in other directions impinge on the walls of the helix and are eliminated (Figure 17.2(b)). Alternatively, the provision of a seed crystal allows any direction of growth to be selected, effectively making the pig tail redundant (even though to this date the pig tail seems to have survived as in Figure 17.2(c) for no good reason that anyone can think of).

After the melting and pouring of the metal into the mould, the mould is withdrawn slowly from a hot zone at a temperature above the melting point of the alloy into a cold zone. The two zones are separated by a baffle designed to fit around the mould as closely as possible to maintain the temperature gradient as high as possible. Withdrawal for an average turbine blade might take up to 2 h.

By the production of a single crystal, grain boundaries (thought to be the weak features of the structure) could be eliminated. In addition, the alloy could be developed for maximum properties such as strength and oxidation resistance because the absence of grain boundaries meant that complications of the control of grain boundary precipitates were avoided. Thus turbine blades and vanes for aircraft engines took a further leap forward, beyond the attainments of the DS castings, and are now used for the most demanding locations in turbines, withstanding extremes of stress and temperature, justifying their name ‘superalloys’.

It is a pity that at this time the casting of these excellent materials has to suffer casting conditions that risk the introduction of defects. The risk is significant because of the huge fall in properties that is clearly possible as illustrated in Figure 17.1 (interpreting the figure as illustrating the effects of bifilm rather than the effects of grain boundaries). The latest vacuum furnace designs have reduced the height h (Figure 16.30) of the melting crucible above the mould, but it remains inescapable that the fall will spell problems for good control of both structure and properties.

The target to achieve a single crystal is often undermined by the growth of various kinds of secondary or stray crystals. Carney and Beech (1997) found oxides at the root of most such misaligned crystals, as might be expected if bifilms were present, simply because crystals would not, in general, be expected to grow through bifilms. Although the directional growth of single crystals would be expected to push many bifilms ahead, thus clearing the casting of many if not most defects, some bifilms that stretch into or across the casting section will be expected to be attached to the mould walls, and thus be immovable. Such barriers to growth will lead to the undercooling of the melt on the far side of the barrier as the mould continues to be withdrawn into the cool zone of the furnace. Eventually, the undercooling will become sufficient to nucleate a new grain, having no necessary orientation relation to the original growth direction. This stray crystal would probably cause the casting to be rejected.

It is important to note that the bifilm barriers may not only be oxides. Ford and Wallbank (1998) found that all castings with additions of nitrogen to the melt formed stray crystals, suggesting the existence of nitride-based bifilms.

In passing, we should note that the seed crystal is covered with an oxide film which appears to be no problem to the orientation of the seeded growth. This is probably as a result of the oxide on the seed being an old, thick oxide and/or nitride that grew in the solid state. It would be expected to be highly fractured and porous, unlike most bifilms which would be only micrometres or nanometres thick, and probably, like most thin films in their early phase of growth, rather perfect.

Other factors emerge to erode the perfection of growth of the single crystal.

1. Although the advance of the front drives the solidification of the dendrites in a vertical direction, these long stalks of solid grow relatively true to their <001> direction. However, when they arrive at a point in the casting where they have to grow sideways, the cantilevered weight of the dendrite (denser than the liquid by a few percent of course) can be supported for a limited distance. Beyond this, the bending moment at the root of the dendrite causes the sideways arm to hinge at the root, until it falls, its fall arrested by the mould or other dendrites. In this way, low-angle boundaries are created (Newell et al., 2009). The situation is analogous to the formation of the branched columnar zone in steel ingots, despite the wall-like secondary arms creating box-girder type morphology of low-carbon steel dendrites which helps to resist bending. This fact suggests that different Ni-base alloys may have dendrite morphologies which will resist this problem to different degrees.
2. The microsegregation between dendrite arms accumulates near the roots of the advancing array. If this is denser than the matrix alloy, this liquid tends to flow to the edges of the mould, creating a convex front. If the segregated liquid is lighter than the bulk alloy, the situation is unstable, and plumes of segregated liquid can ascend through the mesh, dissolving the mesh as it goes, forming a channel segregate. The dendrite arms that are melted off tumble into new orientations in the channel, giving the name ‘freckle defect’ when studied in the etched condition. Ever since McDonald and Hunt uncovered the mechanism for the formation of the channel segregate in 1969, there has been a huge amount of work on this subject, particularly in relation to superalloys (for instance Purvis et al., 1994) and more generally reviewed by Beckermann (2002). A key parameter now included so far as possible in new alloy

designs is the balancing of elements in an attempt to make the interdendritic liquid as near neutral buoyancy as possible. A second powerfully controlling factor is the spacing of primary dendrite stems; fine spacing provides useful viscous drag to help dampen interdendritic flow.

Although the Ni-base superalloys have excellent resistance to oxidation at high temperature, their resistance must be enhanced further for the highest temperature applications. This is achieved by various kinds of coatings, often by a kind of aluminising process, in which aluminium is diffused into the surface of the casting and then subjected to oxidising conditions to generate a strong protective layer of alumina. The beauty of such a coating is that it is mainly metallic, having some ductility and thus resistant to damage such as spalling experienced by brittle or poorly adherent coatings. Also, because of its reserve of aluminium in depth, it is self-healing if damaged.

We need to note, however, that in their studies of the protection of Rene 80 with an aluminide coating, Rahmani and Nategh (2010) observed that cracks in the casting associated with grain boundaries and carbides (features strongly suggesting the presence of bifilms) unfortunately overwhelmed the effects of the coating because the cracks from the matrix extended through the coating. Once again, it seems that bifilms are likely to be responsible for this major potential failure mode of an otherwise excellent technology, making improved casting technology an urgent issue for superalloy castings.

If it is true that single crystal blades derive their extraordinary properties mainly by the adventitious reduction of bifilms, it follows that there are almost certainly much cheaper ways to obtain bifilm-free alloys by sensible melting and casting techniques. Thus polycrystalline blades, produced quickly and cheaply, could in principle equal the creep properties of single crystals, although they would not have the unique directional properties of single crystals which are exploited in some blade designs to improve stiffness and vibrational response of course.

Alternatively, of course, an improved melting and casting approach could bring further benefits to single crystals, eliminate the current risks to integrity, providing manufacturing economies and possibly further improving properties to give extended times between service inspections of engines.

17.4 RAPID SOLIDIFICATION CASTING

The use of the term rapid solidification has traditionally been attributed to such processes as splat cooling of perhaps a gram of material between cold anvils, or the production of ribbon off a rapidly rotating wheel. These process give cooling rates in the region of millions of degrees per second, but the product is hardly a shaped casting, but rather more a heap of bits or ribbons requiring some other process for consolidation.

Here we shall content ourselves with more limited rates of cooling, at rates in the region of perhaps a hundred degrees per second. These more modest rates are still capable of delivering excellent if not astonishing properties, but at the same time, of course, can directly deliver engineering products such as shaped castings.

Sophia and Hero processes

During freezing of a conventional shaped casting, the casting contracts away from the mould, and the mould expands, opening the so-called ‘air gap’ between the casting and the mould (possibly containing a variety of gases except air). The gap is a major barrier to the rate of escape of heat from the casting. Thus the air gap, above nearly all other resistances to cooling, controls the rate of cooling. The metallurgist will be concerned about the fineness of the microstructure, particularly the DAS, but as we have seen the mechanical properties of castings seem mainly dependent on the unfurling of bifilms. Thus, an absence of bifilms, if we could achieve this, would make rapid cooling unnecessary to achieve good properties, but it is important not to overlook that the finer dendrite spacing will be a great benefit to the reduction in heat treatment times.

Some Al alloy investment castings are subjected to a more rapid solidification by being immersed in a coolant immediately after the mould is filled with liquid metal. The Sophia Process is known to involve quenching the mould and its liquid metal contents into a soluble oil. At the same time, a pressure is applied to close porosity so far as possible. These actions significantly increase the properties of casting, and make any further enhancement by, for instance, hot isostatic pressing, to be unnecessary.

The Hero Process used by Tital in Germany is thought to be similar. However, the details of the process remain closely guarded despite the claimed original patent now likely to be nearly or actually expired.

The quenching of the complete investment mould and its liquid metal into a cooling liquid certainly raises properties, but is clearly a messy process, especially if the quenchant is an oil. Furthermore, of course, the efficiency of heat transfer is also limited by the presence of the investment shell that remains in place during freezing. These issues are largely solved by the following sand casting process, even though, of course, an aggregate mould process cannot match the surface finish enjoyed by the investment casting.

Ablation casting

I first came across the word ‘ablation’ in relation to a NASA shuttle space vehicle re-entering the Earth’s atmosphere. To avoid the vehicle heating up and being destroyed, its leading surface was covered with ceramic tiles that were designed to heat up and vaporise, the vapour carrying away and leaving behind the frictional heat generated by the atmosphere so that the space craft itself did not heat up. Thus the term ablation denotes an eroding away and carrying away process.

Ablation casting is a new approach to the making of an aggregate moulded casting in which the fundamental limitation to heat flow presented by the air gap is removed by the application of coolant direct onto the surface of the solidifying casting. As a necessary prior step the mould is removed to allow direct access of coolant. This is achieved by the application of a solute directed onto the mould, but with the mould aggregate bonded with a binder soluble in the coolant. The mould is thereby dissolved away and removed in the flow of the coolant. Thus, unlike Sophia or Hero, in ablation casting, the mould does not obstruct cooling.

In practice, therefore, the liquid metal is poured into an aggregate mould bonded with a water-soluble binder. While the metal is still molten, the mould is ablated away by water jets (Figure 17.3(a)). Because the mould is progressively removed, disappearing steadily as ablation advances along its length, the water is enabled to contact the metal casting directly. A high temperature gradient is progressed through the casting, assisting to eliminate porosity especially in thick sections. The outcome is solidification of a shaped casting under (1) an unprecedented temperature gradient and (2) high solidification speeds. A unique microstructure characterised by extreme soundness and fineness of the last phases to solidify is thereby produced. Porosity can be controlled to be extremely low or effectively zero even in such features as isolated bosses. This is because freezing conditions are now no longer under the control of the mould, but under independent control from ablative cooling which is managed and directed by the casting engineer.

The solidification can be directed to a final corner of the casting where a feeder is located, possibly planted off the casting on a short extension, as an extended feeder neck. Because the freezing front arrives at this point fairly quickly, the feeder can be arranged to drain almost completely, its final form being only a metal skin like an empty paper cup. Feeding can therefore be extremely efficient, so that metal yields can be high.

At the completion of ablation, the mould has been completely removed and the casting sits on the ablation station clean and cold, ready to be picked up immediately for further processing (Figure 17.3(b)).

The mechanical properties achieved by the process are equal to or surpass the best competitive processes such as squeeze casting. This is perhaps not so surprising because the pouring conditions for most squeeze products are poor, introducing defects. In addition, cooling conditions for most squeeze castings are heterogeneous, with only parts of the casting feeling the compressive force holding the casting and mould in contact. Ablation casting can use any form of filling, with the current gravity filling processes naturally suffering defects. Nevertheless, despite the general use of gravity filling during this early stage of development of the process, rapid solidification freezes in bifilms in their compact state so that properties remain good. Also, it is worth noting that the company, Alotech Limited, generally applies filling systems that conform to the principles of this book, which has to be a further significant advantage over most competition at this time. Even so, the properties are surprisingly good, leading one to suspect some additional factor. It seems likely that the unusually powerful temperature gradient of the process may be efficient in assisting the freezing front to push bifilms ahead, effectively cleaning large areas of the casting. When the process is further developed, perhaps introducing a filling process such as counter-gravity, properties from ablation should give yet further improvement, even though, of course, competitive processes should also improve if provided with such advantageous filling systems.

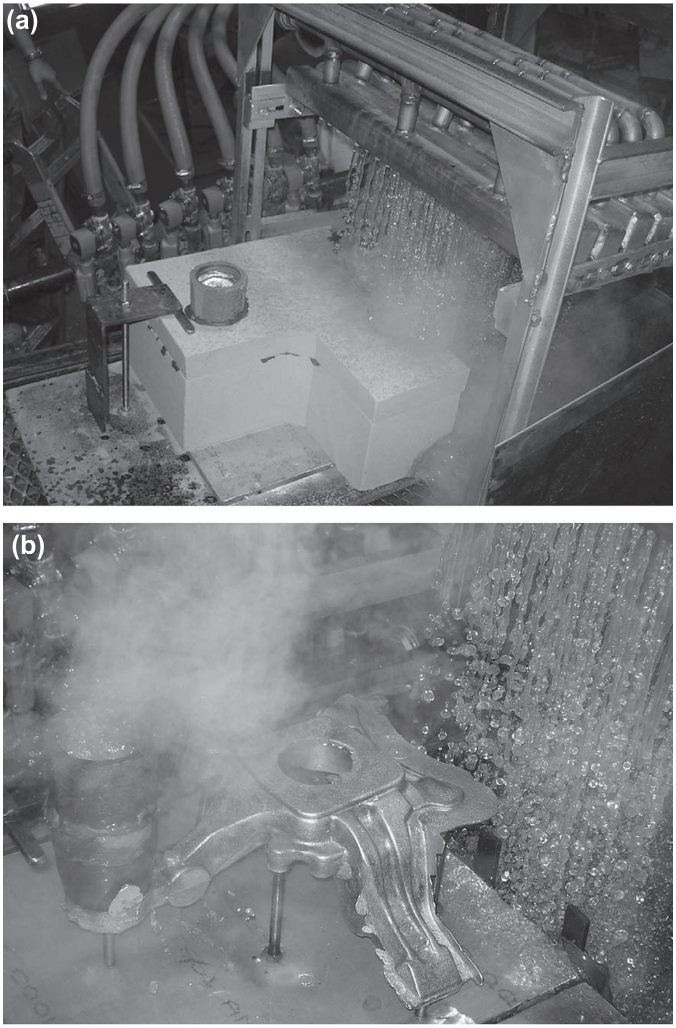

FIGURE 17.3

Ablation casting of an Al alloy; (a) eroding the mould away and cooling the casting; (b) the final casting, cold and clean.

Additional advantages for ablated castings appear to be their highly competitive cost. This arises mainly because the silicate-based binder is much less costly than competitive resin-based binders, but also benefits from the relatively low cost of capital equipment and modest cost of tooling. Further benefits arise as a result of the water-based technology. The process produces no smell, no fume and no dust (the recycled aggregate is mostly either wet or damp), saving the installation of costly fume and dust extraction equipment.

The optical microstructure of an Al-7Si-0.4Mg test casting that was allowed to solidify without ablation is shown in Figure 6.9(a), and is typical of a non-modified Al-Si eutectic. Typical ablation-cooled structures are shown in Figures 17.4 and 17.5 illustrating the extreme fineness of the eutectic spacing that is difficult to resolve even at 1000 magnification in the optical microscope (but only limited regions of the dendrite spacing have been refined).

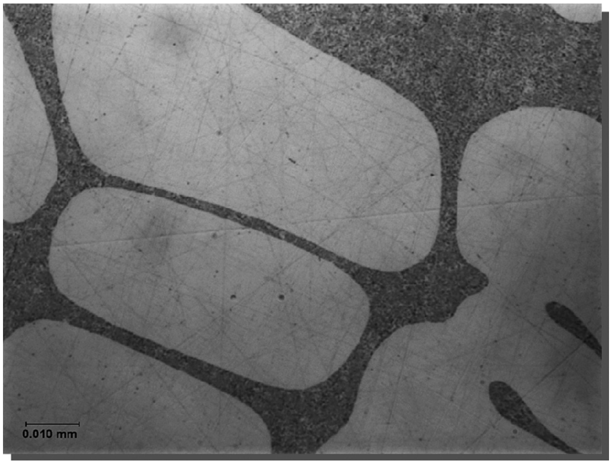

FIGURE 17.4

Structure of an Al-7Si-0.4Mg alloy achieved by ablation, without added modifiers such as Sr or Na. In the optical microscope, the dendrites are seen to be coarse, but the Al-Si eutectic is hardly resolvable even at 1000×.

The Si inter-particle spacing is under 1 μm in this structure. Note that no Na or Sr has been added to achieve this apparently well-modified structure.

Figure 17.5 illustrates the extent of freezing that has taken place before the ablation cooling has had time to reach these regions. Thus the secondary DAS is in general rather coarse, and typical of a sand casting of that section thickness. In some instances, particularly in the centres of sections, it is at first sight curiously perverse that the DAS is fine because fine structures as a result of rapid cooling are normally limited to the outer skin of the casting. The fine central regions are of course the result of ablation arriving in time to limit dendrite arm coarsening in these regions. Furthermore, such regions would often contain porosity, thus weakening the structure, whereas ablation tends to yield sound and fine grained interiors that would be expected to have enhanced properties.

It is extremely significant that the improved properties are clearly not directly related to DAS, which in general is not improved. Improved properties appear to be a result only from improved control of the solidification of the eutectic. The current preoccupation focussing on the achievement of a fine DAS is therefore seen in general to be a misunderstanding (although of course it is correct that DAS indicates the freezing time of the dendrites).

Alloys that are traditionally difficult to cast because of hot tearing problems such as the Al-4.5Cu series alloys prove to be relatively straightforward with ablation, although the rapid and directional solidification must be carefully controlled to avoid dendritic segregation problems (inverse segregation). Furthermore, the wrought alloys including the 6000 and 7000 series have been demonstrated to be capable of being made into shaped castings. Much of this success with otherwise difficult casting alloys arises because the casting does not build up long-range stresses during casting: the casting ahead of the freezing front remains molten and can therefore effortlessly accommodate strain without causing stress.

Perhaps surprisingly, ablation has produced excellent Mg alloy castings which have displayed properties well in excess of those of any other competing Mg casting process at this time.

At the time of writing, the process is protected by several patents, and is, in common with many new processes, probably somewhat more difficult than it looks, even though its difficulties are clearly worth overcoming. It is still undergoing rapid development, and at this time is just starting its first series production castings.

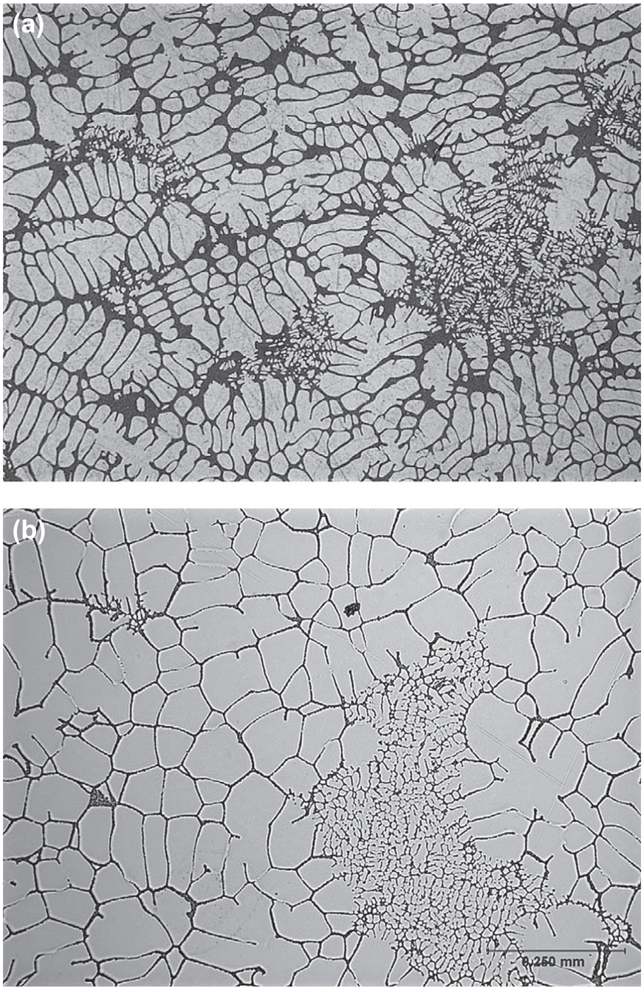

FIGURE 17.5

The dual dendrite structure often observed in ablation-cooled castings of Al alloys as a result of natural cooling finally caught up by ablation cooling; (a) in an Al-7Si-0.4Mg alloy containing significant eutectic phase; (b) in an Al-4.5Cu solid solution alloy exhibiting no significant eutectic phase.

For the future of ablation, a combination of the moulding and solidification technology with good filling, possibly via a counter-gravity process, promises to be a powerful process for both Mg- and Al-based castings. It is not known at this time whether the process can be extended to encompass higher melting point copper and ferrous alloys. As we continue to note in this book, there remains no shortage of future challenges.