LETTER

Supercooling of metal in fine filters

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A commercially available technique for assessing the quality of liquid aluminium alloys is the Prefil-Footprinter test in which a quantity of liquid metal is forced to pass through a fine filter. Normally the filtration rate is measured and compared with recorded information. A clean melt retains the open pore structure of the filter keeping its resistance to a minimum and achieving a high rate of filtration. In contrast, a low flow rate would indicate the build up of inclusions leading to partial choking of the filter [\[1](#page-2-0)]. The curve of filtrate weight versus time provides a measure of the relative cleanliness of an aluminium melt by comparison with a wide variety of benchmark results [\[2](#page-2-0)]. The filter and its deposit can be subsequently sectioned and the inclusions identified by metallographic techniques.

Some of the most important and common types of inclusions detectable by the test are those entrained from the surface as films, such as oxides. The entrainment mechanism is a folding action, ensuring that all such entrained films are double, and have been called bifilms [\[3](#page-2-0)a].

Shortly after a period of bulk turbulence such as immediately after pouring, the bifilms are in a convoluted and compact shape, and so a high proportion is expected to pass through most filters [\[3](#page-2-0)a]. However, the filter used in the Prefil test is particularly fine, with a pore size in the region of only $60 \mu m$ so that a higher proportion of the coarser bifilms is expected to be retained. Due to their

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double nature, the thin layer of residual air trapped between the dry faces of the films ensures that bifilms are natural barriers to the growth front during solidification. Conversely however, the wetted outer faces of the double film are seen to constitute excellent favourable substrates for the formation and growth of many intermetallic and second phases [\[3](#page-2-0)a].

During the course of work with the Prefil technique, and examination of a metallographic section of a filter revealed a curious feature: regions of very fine dendrite arm spacing (DAS) were observed immediately adjacent to regions of coarse DAS (Fig. [1](#page-1-0)a–f). Since DAS is typically taken as an indication of the freezing time of the alloy $[3c]$ $[3c]$ $[3c]$, the implication of such extremes of cooling rate in such a conductive metal at such close spacing appeared impossibly improbable.

Optical examination and scanning electron microscope (SEM) microanalyses show that these regions possessed the same average composition as the original alloy (and thus were not any kind of curious inclusion or second phase).

On closer examination (Fig. $1a-j$ $1a-j$), it was noted that the regions of contrasting DAS were separated by thin films, many decorated with silicon or other intermetallic particles, sometimes on both sides of the films; an indication of an occasional symmetry suggesting that the films were almost certainly oxide bifilms (not all bifilms are symmetrical because sometimes one film is thicker, or of a different composition or structure than the other, so that sometimes precipitates occur on one side but not the other [\[3](#page-2-0)a]). Thus, it was concluded that the regions of differing DAS constituted pockets of liquid alloy isolated from each other by bifilms.

The phenomenon of a liquid Al alloy in the form of droplets, isolated from its neighbour by oxide films

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Fig. 1 The observation of fine DAS isolated by pockets of oxide films

(necessarily bifilms, since each droplet would have its own film, so adjacent drops would be separated by a double film, with their dry sides facing each other) is commonly observed in Al melting practice. Such material is known as 'dross', and may contain over 90% of good metal, but the good metal is not easily extracted from this dull, dry, unpromising—looking material.

Those regions exhibiting the very fine microstructure indicate regions of extremely rapid freezing. Such rapid freezing, adjacent to regions of relatively slow freezing indicated by coarse structure, can only be envisaged to be the result of very high undercooling, implying in turn a lack of nucleation sites in this particular isolated volume. The further implication is that the oxide does not form a favourable nucleation site for the nucleation of the alpha Al phase. This is in agreement with many other experimental demonstrations of the supercooling of melts by hundreds of degrees Celsius of pure metals and alloys contained either in their oxide skins, or in oxide containers [4, 5].

The DAS is of the order of 10 µm in the fine structure and [1](#page-1-0)00–200 μ m in the coarser regions (Fig. 1 a–j). From these values, we can estimate their solidification times as having been around 1 s and 1,000 s (17 min) respectively [3b].

In passing, it can be noted that supercooling of metal droplets dispersed by atomisation or by precipitation in a matrix has formed the basis of many elegant experiments on the nucleation of the solid [4]. Such experiments, of course, have provided direct measurements of the undercoolings involved, whereas only the solidification times (or rates) can be deduced from the present observations.

Such pockets might be expected in the fine filter used in the Prefil test because the filter collects the films and acts as rigid mechanical framework keeping the pockets intact. In many conventional gravity castings, such isolated pockets are less common, probably being carried away into feeder heads, or burst as a result of the forces of turbulence. Even so, instances appear to have been seen in gravity castings from time to time and in fact may be more common than we suspect. For high pressure die castings, where the sections are generally thinner, and the oxide bifilm density expected to be high, such structures are seen often, and numerous examples have been recorded [3c]. This adventious feature of castings is highlighted here for the first time.

References

- 1. Makarov S, Apelian D, Ludwig R (1999) Inclusion removal and detection in molten aluminum: mechanical, electromagnetic, and acoustic techniques. AFS Transactions, 107
- 2. Rochette P, Simard AA (2000) Cleanliness measurement using the Prefil-Footprinter instrument. Aluminium, 76(1/2): 20–26
- 3. Campbell J (2003) Castings, 2nd edn. Elsevier (a) pp 17–69; (b) p 136; (c) p 45
- 4. Walker JL (1959) In: StPierre GR (ed) The physical chemistry of process metallurgy, Part 2. Interscience, New York, p 845; summary in B. Chalmers (1964) Principles of solidification. Wiley, New York
- 5. Liu RP, Herlach DM, Vandyoussefi M, Greer AL (2004) Metall Mater Trans 35A:607–612