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Boron Heterogeneities in As-Cast FeAl Intermetallics

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Abstract: Boron analysis of B doped FeAl alloys, in the as-cast condition, can give surprising results: large enrichments of B at the surface of ingots are systematically observed. The variations of B in a section of a 30 mm diameter ingot (Fe-25wt%Al-150ppmB) are typically: 180ppm B at the surface and 140 ppm B in the centre of the ingot. However, when single crystals are grown at low cooling rates (solid/liquid interface velocity about 3mm/h), the segregation of boron is regular: enrichment in the final part of the crystal is found. In Fe-B alloys, no boron segregation was found. Boron enrichment at the surface of as-cast FeAl ingots might be due to segregation of B in the liquid state by interaction with alumina superficial layers.

1 - INTRODUCTION

The aim of the present short note is to show how boron analysis in FeAl alloys can be erratic when sampling precautions are not sufficient. In a former study (ref. 1), systematic errors were observed when analysing FeAl ingots and, quite systematically, the boron analysis result was higher than the amount introduced. The first idea was to suspect the analysis method but several were compared and the true reason was found in the boron heterogeneities in sections of as-cast ingots. Different ingot sizes and shapes and boron levels were selected with different cooling rates in order to confirm the existence of heterogeneities and clarify the segregation process.

2 - EXPERIMENTS

2.1 - Materials (table I):

The selected Fe-25wt%Al is near stoichiometric composition.
- 1st type: these FeAl alloys were prepared by induction melting in alumina crucibles and cast in metallic moulds. The diameters were 80 mm and 30 mm and the mean boron levels about 30 and 150 ppm respectively.
- 2nd type: These alloys were induction melted in a water-cooled silver crucible described elsewhere (ref. 2). Solidification took place in the cold crucible in a few seconds. The mean boron content was 50 ppm.
- 3rd type: FeAl single crystals were grown in a horizontal electric furnace (Chalmers method) in alumina crucibles. The solidification rate was 3 mm/hour. The mean boron level was 15 ppm.
- 4th type: a Fe-30ppm B alloy was melted in a cooled crucible in the same way as the 2nd type FeAl alloy.
Table I - Mean analysis and preparation processes of the FeAl alloys

<table>
<thead>
<tr>
<th>Type</th>
<th>Al (wt%)</th>
<th>B (ppm)</th>
<th>Section (ppm)</th>
<th>Solidification mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>1st type</td>
<td>25</td>
<td>30</td>
<td>Φ 80</td>
<td>cast in metallic mould</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>150</td>
<td>Φ 30</td>
<td>id</td>
</tr>
<tr>
<td>2nd type</td>
<td>25</td>
<td>50</td>
<td>50x30</td>
<td>cold crucible</td>
</tr>
<tr>
<td>3rd type</td>
<td>25</td>
<td>15</td>
<td>20x15</td>
<td>3 mm/h</td>
</tr>
<tr>
<td>4th type</td>
<td>0</td>
<td>30</td>
<td>50x30</td>
<td>cold crucible</td>
</tr>
</tbody>
</table>

The various types of alloy preparation and solidification enable different levels of B content, specimen size and solidification rate to be compared and the influence of Al content to be tested.

2.2 - Boron analysis method

Two methods were used for comparison: "methylene blue" with iron extraction and "curcumin". After numerous measurements, the curcumin method was selected as easier and better reproducible.

2.3 - Sampling:

Analyses were made of specimens taken from the surface and inside the ingots by hammer breaking after cutting with a disk, for the FeAl-B alloys which were too hard to be machined, or by machining chips for Fe-B alloys. Precautions were taken in order to repeat the analysis many times.

3 - RESULTS

1st type: The boron variations in a section of an ingot 80 mm in diameter (Fe-25wt%Al-30ppmB) or 30 mm in diameter (Fe-25wt%Al-150 ppmB) are given in figure 1. The surface of the ingot section is always enriched in boron, for example: 180 ppm B at the surface and 120 ppm B in the centre of the ingot. The thickness of the enriched surface cannot be given because sampling was only possible by breaking the metal or cutting with a disk. Variations of boron content from the head to the foot of the ingot are negligible as compared with the radial variations, e.g. for a 300 mm height ingot: 175 to 185 ppm at the surface and 140 to 160 ppm in the centre.

![Fig. 1 - Boron heterogeneities in sections of two cast FeAl alloys (No 1 type)](image-url)
2nd type: In a small ingot (Fig. 2) melted and solidified in a cold crucible (a water-cooled, boat-shaped Ag crucible) in which the solidification rate is very high, three sections (a, b, c) were analysed. Boron enrichment at the surface of the ingot is also noticeable here: 40 ppm at the surface and 30 ppm in the center (Fig. 2), except in the c-section where the boron content does not vary from place to place. This result may be due to different solidification conditions in the a, b and c sections. Here, it was not possible to clarify this question because solidification in the cold crucible is not precisely controlled and is difficult to describe. Such results, showing enrichment of the center of FeAl-B ingots in comparable as-solidified states, were verified in several heats with different boron contents.

![Ingot length (150 mm) Ingot section (50x30 mm²)](image)

Fig. 2 - Boron heterogeneities in three sections of a FeAl ingot solidified in a boat-shaped cold crucible. The grey levels from light to dark correspond to increasing B levels.

3rd type: When growing single crystals with low cooling rates (solid/liquid interface velocity about 3 mm/h), the solid/liquid segregation of boron was found to be "regular", i.e.: enrichment in the final part of the crystal was observed (Fig. 3). It can be seen that the Al content also increases in the last solidified part of the crystal.

![Solidification rate: 3 mm/h](image)

![Boron (wt ppm) vs Distance (mm)](image)

Fig. 3 - Solid/liquid Boron and Aluminium segregation during FeAl single-crystal growth.

The preceding result shows that the solid/liquid normal segregation of boron cannot explain the surface enrichment of as-cast or as-solidified FeAl ingots. Something else must occur during melting and solidification.
Clarification was brought by the next Fe-B alloy (without Al).

4th type: In a Fe - 30ppmB ingot (without Al), prepared and solidified in the same cold crucible as the 2nd type FeAl alloy, no variation of B content from the surface to the center could be measured (Fig. 4), except in the first mm of the superficial layer, in which B is slightly lower. This result was obtained on chips machined from the ingot, a technique which permitted analysis as a function of the distance from the surface. Such a superficial boron loss can be considered as ordinary: similar observations can be made on specimens heat-treated in the solid state (ref 3).

Small boron variations can be noticed between the a, b and c sections of the Fe-B ingot (Fig.4).

4 - DISCUSSION

The surprising boron surface enrichment of FeAl ingots in the as-cast (in a mould) or as-solidified (in a cold crucible) state cannot be explained by normal liquid/solid segregation because (i) at slow solidification rates (in growing single crystals), boron enrichment is normally observed in the last solidified part (in parallel with Al enrichment) and (ii) the fast cooling of a 30 mm diameter ingot cannot be responsible for boron migration during solidification.

1st hypothesis: B in FeAl alloys may segregate in the liquid state, giving B enrichment at the surface after solidification.

However it is difficult to imagine what happens to the liquid metal during casting, because agitation of the melt during pouring might modify the B gradients established in the liquid state, but in the ingots solidified in a cold crucible, even if stirring occurs in the liquid state due to magnetic forces, there is less agitation during solidification than during ingot casting. Furthermore, some image of the boron gradient in the liquid state may be retained in these fast solidified ingots.

Comparison with Fe-B alloys in which no internal boron depletion was observed, leads to the second hypothesis.

2nd hypothesis: boron enrichment at the surface of FeAl ingots might be due to an interaction with Al, i.e. probably with the alumina layer.
5 - CONCLUSION

A tentative conclusion can be proposed:

=> Boron enrichment at the surface of as-cast FeAl ingots might be due to boron segregation in the liquid state by interaction with alumina superficial layers.

This boron analysis problem can be important when controlling the composition of FeAl alloys during melt processing. It will not be so important after hot rolling at high temperature (1100°C), which reduces the boron heterogeneities of the cast metal.

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References: