Castability and Microstructural Development of Iron-based Alloys under Conditions Pertinent to Strip Casting – Specialty Fe–Cr–Al Alloys

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A strip casting simulation of the iron-based specialty alloy known as Fecral (or Kanthal) has been carried out. The alloys tested were found to be easily castable and show good surface quality. Increasing the melt superheat increased the nucleation density and heat flux, leading to a refined ferrite grain size. Although changing the gas atmosphere during casting was observed to modify the measured heat flux, this did not correlate to any change in the nucleation density. The cast strips were able to be rolled without cracking, and showed mechanical properties similar to those found in comparable alloys after conventional thermo-mechanical processing.

KEY WORDS: strip casting; specialty alloys; fecral; rapid solidification; nucleation; heat flux.

1. Background on Strip Casting of Steels

There is currently renewed interest in direct strip casting technology due to its potential to significantly reduce the energy required to convert liquid metal to sheet products.1) In addition, strip casting offers advantages of reduced capital costs and lower space requirements compared to conventional continuous casting processes. Strip casting technology has mainly been tested to produce conventional low carbon and stainless steel grades, and for this reason, most of the information available in the published literature is on strip casting of 0.06 C steels, 304 stainless steel and other comparable grades. Another class of materials that are potential candidates to be produced by strip casting is Fe-based, specialty alloys with typical alloying additions of 10 to 40 wt%. Two examples of such alloys are Fecral (Fe–Cr–Al) and Invar (Fe–Ni). These alloys are presently produced by ingot casting, and the ingots are then hot forged and/or hot rolled, prior to cold rolling and annealing. Batch ingot casting processes are highly inefficient. In addition, problems of elemental segregation, oxidation, and cracking plague alloys produced by conventional manufacturing routes and it has been reported that the overall yield from ingots to cold rolled strips could be as low as 50%.1) Strip casting followed by direct cold rolling provides a highly efficient alternative manufacturing route to overcome such problems.

Cooling rates of 10² to 10⁴ °C/s are typically encountered during strip casting and the information on castability of different alloys under rapid solidification conditions have been largely gathered either by characterization of industrial scale strip cast products5–10) or by carrying out targeted experiments with techniques such as levitation droplet melting and solidification,11–14) and/or immersion solidification testing using a specially designed dip tester.15–20) The dip tester15) is based on the principle of rapidly submerging a copper substrate into a bath of molten metal, with a liquid-solid contact time of around 0.2 s. Thermocouples embedded below the substrate surface measure the temperature response of the substrate with millisecond resolution, and these measurements are used to calculate instantaneous heat flux during solidification. Small coupons produced during immersion provide enough material for surface quality evaluations, microstructure investigations, hardness testing and cold rolling simulations. The data presented in the following three papers have all been obtained from the immersion technique.

The most important factor that determines castability of an alloy under strip casting conditions is the initial contact response of the molten metal to the substrate.15,16) The intiative of this contact is reflected in both the peak heat flux measured during solidification (usually occurs within 20 ms of the initial contact) and the number of nucleation events that occur per unit area on the contact surface; both increase with enhanced contact response of the molten metal on the substrate.15) The significance of substrate surface condition on controlling heat fluxes has been demonstrated in a few studies.14–18) For example, with molten Ni and Cu it was shown that higher heat fluxes could be achieved when substrates were smooth with roughness value (Ra) below 0.5 μm.14

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The opposite effect was found when contact was imposed through deliberately patterned substrate surfaces; for example, with 304 stainless steels heat fluxes doubled when the substrate had regularly spaced parallel ridges 150 μm apart (15) as opposed to a smooth substrate. Two other process variables that could be used to control the initial contact response of the melt on the substrate were surface tension of the melt (addition of surface acting elements such as sulphur to low carbon steels lowered surface tension and increased both heat flux and the nucleation density (11)) and melt superheat (higher melt superheat reduced both heat flux and nucleation density for 304 stainless steel (15)). The impact of such variables on strip castability of specialty alloys was not the focus of our study; however, the information presented in this paper series will provide useful reference data to be incorporated into future detailed studies on strip castability of Fe-based specialty alloys.

Microstructural features of strip cast products are fairly well described in the literature. (13) For those alloys that do not undergo solid-state phase transformation during cooling (e.g., 304 austenitic and 430 ferritic grades), the cast microstructures consist of columnar grains with the longest axis in the solidification direction towards the center of the strip. These grains are typically 100–200 μm wide and can have lengths up to the thickness of the solidified shell (e.g., 700–1 000 μm). (21) Although cast microstructures thus described are much finer than the several mm size cast grain structures typically produced during conventional casting of slabs or ingots, strip cast grain structures are significantly coarser than what would be obtained for strips produced by the conventional casting plus thermo-mechanical processing route. For those alloys that undergo one or more solid-state phase transformations during cooling, the cast microstructures can be significantly different and notably finer. For example, strip casting of low carbon steels (20) typically produces a mixed microstructure consisting of relatively finer polygonal ferrite grains (typically 10–50 μm size) and large colonies of Widmanstätten and acicular ferrites.

Strip cast products are usually cold rolled and annealed to produce thin sheets and it is imperative that the coarse cast microstructure is refined and the mechanical properties improved during these steps. It should be noted however that the processing window for the thin (~2 mm) strip cast material is significantly more limited than that is available for a conventionally cast slab or ingot (tens of mm thick). There is relatively little information available on the microstructure development of strip cast material resulting from thermo-mechanical processing. In the case of low carbon steels, it has been reported that recrystallisation after cold reduction was sluggish (20) when compared to conventional hot strip mill products, an observation usually attributed to coarse cast grain structures and the presence of nano-scale second phase particles. (10)

Strip castability of several Fe-based specialty alloys containing high levels of one or more of Cr, Ni, Al and Cu were investigated in this program. The cast microstructures were characterized for all alloys. The response of the cast products to conventional cold rolling and annealing treatments and the resulting product mechanical properties were assessed, and compared against the reported industry information. The results obtained for Fe–Cr–Al (Fecral), 200-series stainless steels and Fe–Ni (Invar) are presented in the following set of three companion papers. Results obtained for other specialty alloys tested, and the response of the selected alloys for thermo-mechanical processing will be examined in future publications.

In the present communication we describe the results of our strip casting simulations of an Fe-based specialty alloy commonly known as Fecral or Kanthal. This alloy is used in heating elements because of its excellent corrosion resistance at high service temperatures of 1 100 to 1 300°C, combined with its relatively high electrical resistance. (22) Fecral is typically produced by conventional metallurgical processing of a cast ingot, consisting of solution treating, hot rolling and then cold rolling. In this paper, we present results from casting simulations of this alloy directly into thin strip. We have chosen to study this particular alloy because current processing is complex with many steps and significant yield losses, which could potentially be reduced through strip casting. Also, a review of the literature suggests that there has not yet been a study that investigates the strip casting behaviour of Fecral alloys.

2. Experimental Methodology

Casting experiments under rapid solidification conditions pertinent to strip casting have been carried out using a casting simulator. A full description of this apparatus has been published previously (15) and will not be fully described here. Briefly, the apparatus is based on the rapid immersion of substrates into a molten metal bath. There were two copper substrates mounted in an immersion paddle; each had dimensions of 35 × 35 mm and a smooth surface finish of 0.4 Rz. One of the substrates was chrome coated, a practice used in commercial strip casting to reduce wear of the rolls. The second substrate was equipped with a thermo-couple at a suitable position to measure the temperature changes in the substrate from which heat flux during solidification could be calculated. The heat flux calculation methodology has been described in reference. (23)

Five conditions were tested, and these are detailed in Table 1. The tests included two different compositions of Fecral: Fe–15Cr–4Al (Fecral 125) and Fe–20Cr–5Al (Fecral 135), and their compositions are detailed in Table 2. For each alloy composition a series of dips was carried out under either a nitrogen or argon atmosphere. All experiments were carried out at a melt temperature of 1 620°C (±3°C), with the exception of the first five experiments which were carried out at a melt temperature of 1 580°C (±2°C). This gave a total of five test conditions. Typically 6–8 samples were produced under each condition, resulting in a total of 30 tests for the entire experiment. During initial melting, and in between tests, the melt was kept under a...
flowing argon atmosphere to minimise oxidation and slag formation. Chemical composition of the melt was periodically analysed to ensure that the melt composition did not drift from the target. The substrates were mechanically cleaned with a wire brush before each dip, and all dips were carried out at 1 m/s casting speed.

After casting, the samples were evaluated for nucleation density by optical microscopy of the contact surface between the molten metal and the substrate. To examine the microstructure of the samples through the thickness of the casting, selected specimens were sectioned and mounted in the transverse direction. These samples were then metallographically prepared using standard polishing techniques. Polished sections were etched using acid ferric chloride (5 g ferric chloride, 50 mL HCl and 100 mL water, and immersion time of ~10 s) to reveal as-cast microstructures.

Selected as-cast specimens were also examined using scanning electron microscopy (SEM). SEM was used to examine the microstructure of samples using backscattered imaging, and the composition of inclusions was examined using energy dispersive spectroscopy (EDS). The SEM analysis was carried out on a Supra VP SEM at 20 kV, and the EDS system used was an Oxford Instruments analysis system.

3. Results

3.1. Castability and Surface Distortion

The solidification rate can be approximated by the use of the K factor, see Eq. (1). Rather than reporting the thickness of the solidified shell, the K factor takes into consideration small differences in the residence time between samples and gives a better approximation of the solidification rate.

\[ K = \frac{d}{\sqrt{t}} \]  (1)

where \( d \) is the thickness of the solidified strip, and \( t \) is the time in the melt. The samples produced from the dip testing were typically 0.7 to 0.9 mm thick. The solidification time at the centre of the substrate was 148 ms, which corresponds to a K-factor of between 15 and 18 mm/min\(^{0.5}\). The average K factors for the four test sequences carried out at 1620°C are shown in Fig. 1(a). There was not a particularly large difference in K factor between the two alloys, nor was there a large change in K factor with a change in gas atmosphere. However, there is some indication that the solidification rate was marginally higher on the Cr coated substrates. The effect of melt superheat is shown in Fig. 2. Again, there was little difference in the solidification rate between the two melt superheats tested.

The surface quality of the dip cast specimens can be used to provide more information on the castability of the alloy. Generally, the quality of the dip tested samples was satisfactory, being free of cracks or major warping (or distortions). Some defects caused by oxide/slag accumulation on the
melt surface were observed, Fig. 3. The cast samples occasionally had relatively large (15–20 mm) surface distortions (see Table 3) that are caused by thermal factors such as a loss of contact during solidification and a disparity between the thermal contraction of the sample and the substrate. This coarse scale warping of the strip samples is not considered to be a significant casting defect because in industrial strip casting they can be overcome by imposing uniform contact through the casting roll surface.

3.2. Heat Flux during Solidification

The heat flux measurements showed good reproducibility, Fig. 4(a). Typically, the heat flux increased rapidly to 15–25 MW/m² within the first 10 to 15 ms, which corresponds to the point of establishing good contact between the substrate and the liquid metal. For the next 30 to 40 ms, the heat transfer rates drop rapidly as the thickness of the solid shell increases. The average heat flux over the entire solidification period, calculated by measuring the area under the heat flux curve reflects the total heat removed during solidification. Both the peak heat flux and the average heat flux measurements are shown in Fig. 1(b) for the samples produced at a melt temperature of 1620°C. Although the experimental error is relatively large, it appears that the heat flux is similar for the first three casting conditions. However, for the 20Cr–5Al alloy, the peak heat flux appears to be consistently higher when cast under nitrogen compared to argon, see Figs. 1(b) and 4(b). The effect of the melt superheat on the measured heat flux is shown in Fig. 2(b). Both the average and peak heat flux increased with increasing melt superheat.

3.3. Nucleation of Solidification

The nucleation of individual grains during solidification on a smooth substrate produces a distinctive surface feature on the contact surface of the sample that is retained after casting. These characteristic nucleation points can be viewed optically, and have been counted on selected samples in order to evaluate the nucleation density during solidification. This is a particularly important parameter in strip casting because the grain size of the casting is intimately related to the nucleation density, with higher nucleation densities being beneficial for the alloy grain refinement, and therefore its amenability for down-stream processing and improved mechanical properties. Typical examples of the surface nucleation structures obtained are shown in Fig. 5.

The images show a range of nucleation patterns, from a sparse nucleation arrangement with a relatively coarse dendritic structure (Fig. 5(a)) to a dense nucleation arrangement.

Table 3. Occurrence of sample warping >15 mm in diameter.

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<thead>
<tr>
<th>Casting condition</th>
<th>Occurrence of coarse scale surface distortion</th>
<th>Percentage</th>
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<tbody>
<tr>
<td>Fe–15Cr–4Al, N₂</td>
<td>1 from 8 specimens</td>
<td>13%</td>
</tr>
<tr>
<td>Fe–15Cr–4Al, Ar</td>
<td>2 from 6 specimens</td>
<td>33%</td>
</tr>
<tr>
<td>Fe–20Cr–5Al, N₂</td>
<td>2 from 4 specimens</td>
<td>50%</td>
</tr>
<tr>
<td>Fe–20Cr–5Al, Ar</td>
<td>3 from 5 specimens</td>
<td>60%</td>
</tr>
</tbody>
</table>
with a fine dendritic structure (Fig. 5(b)). It is clear from the optical micrographs that increasing the melt superheat significantly increases the nucleation density (compare Figs. 5(a) and 5(b)). It was also apparent that the 15Cr–4Al samples cast under an argon atmosphere had a slightly higher nucleation density compared to those cast under nitrogen, Figs. 5(b) and 5(c). However, the 20Cr–5Al alloy showed little difference in the nucleation density when cast under different atmospheres.

Figure 6 shows the nucleation density, plotted as a function of the measured heat flux for the individual samples on which the nucleation density was counted. Also included in this plot is the nucleation density data for 304 stainless from a previous study. It can be seen that the alloys cast under nitrogen corresponded well with previous data. It is also evident from this plot that casting under argon reduced the heat flux, consistent with the average values shown in Fig. 1(b). However, this did not correlate with a decrease in the nucleation density. In other words, casting under argon instead of nitrogen, produced comparable nucleation density, but a lower heat flux.

3.4. As-cast Microstructure

Typical examples of the microstructures obtained from the four different casting conditions are shown in Fig. 7. These alloys solidify as ferrite at around 1490°C and remain ferritic down to room temperature. The microstructures in Fig. 7 therefore have not undergone a phase transformation and provide an excellent indication of the solidification behaviour of the alloy. The microstructures consisted of large columnar grains that nucleated near the substrate side, growing parallel to the solidification direction and coarsening progressively towards the melt side. This was commensurate with an increase in grain width. Grain width measurements made at 1/4, 1/2 and 3/4 of the sample thickness support this observation, Fig. 8.

The microstructures presented in Fig. 7 show that the grain width varies between 50 and 200 µm depending on the solidification conditions and the position within the sample. These microstructures are around an order of magnitude finer than those conventionally produced in cast ingots, but compared to alloys produced by traditional thermo-mechanical
processing, these grain sizes are relatively large. The nucleation density at the contact surface and the cross-sectional microstructures correlate well, as evidenced from the one to one correlation between the nucleation spacing on the surface and the grain width on the section during the initial stages of solidification, Figs. 5, 7 and 8. Many grains spanned the entire thickness of the sample, and in the solidification direction had size range of 500 to 1000 μm, see Fig. 7.

The optical microstructures of the 20Cr–5Al showed distinctive coarse features decorating the grain boundaries. It was not clear from the optical micrographs if these features constituted coarse intermetallic species at the grain boundaries, or if this was simply a unique etching response of this alloy. To further investigate, un-etched samples were examined using scanning electron microscopy. There were no coarse particles at the grain boundaries evident in the SEM images, Fig. 9(a). It was concluded from this analysis that the large features at the grain boundaries that could be seen in the optical images were simply heavy etching pits that
developed during the metallographic preparation.

The SEM analysis also revealed that there was very little segregation between dendrite arms in the alloy, since no atomic contrast was evident in the backscattered images, Fig. 9. There were, however, a significant population of small inclusions that were equiaxed in shape, with an average size of 1 μm. Qualitative EDS analysis showed these to be Al enriched and therefore probably aluminium nitride, Fig. 10.

3.5. Mechanical Properties

In order to compare the strength of the materials produced by strip casting, with those obtained by conventional processing methods, both hardness testing and shear punch testing were carried out, see Table 4. The shear punch test measures an equivalent yield and tensile strength of a material, and is typically employed when there is not sufficient material available for a conventional tensile test. The shear punch test is a well developed technique, and for further information on the conversion of shear stress to equivalent tensile stress the reader is referred to the references provided.25–27

The cast samples were tested in three conditions: as-cast; rolled; and annealed. The 15Cr–4Al alloy was cold rolled to 80% reduction, but the 20Cr–5Al alloy could not be rolled above 15% reduction before cracks appeared. It is known that the brittle to ductile transition temperature for this alloy is slightly above room temperature,28 so warm rolling was used to overcome this problem. The 20Cr–5Al alloy was successfully warm rolled at approximately 200°C to 70% reduction. Both alloys were annealed at 900°C for 30 minutes under conditions comparable to those used in conventional processing of these alloys. This produced fully recrystallised microstructure with an average grain size of around 20 μm (note that a full evaluation of the microstructure and textures developed during recrystallisation will be the focus of a forthcoming publication, and will not be further described here). As can be seen from Table 4, the equivalent yield

![Fig. 10. EDS analysis of the fine particles observed in the 20Cr–5Al alloy. The particles are enriched in Al, and depleted in Cr and Fe.](image)

Table 4. Evaluation of the mechanical properties of the strip cast Fecral alloys. Data for commercially produced alloys of a similar composition are also provided for comparison.29

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<tr>
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<th>Present study</th>
<th>Conventional processing</th>
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<tr>
<td></td>
<td>Equivalent tensile strength</td>
<td>Hardness (VHN)</td>
</tr>
<tr>
<td>Fe–15Cr–4Al</td>
<td>As cast</td>
<td>482</td>
</tr>
<tr>
<td></td>
<td>Cold rolled</td>
<td>566</td>
</tr>
<tr>
<td></td>
<td>Annealed</td>
<td>382</td>
</tr>
<tr>
<td>Fe–20Cr–5Al</td>
<td>As cast</td>
<td>617</td>
</tr>
<tr>
<td></td>
<td>Warm rolled</td>
<td>786</td>
</tr>
<tr>
<td></td>
<td>Annealed</td>
<td>513</td>
</tr>
</tbody>
</table>
strength and ultimate tensile strengths for the annealed condition are close to those values that would be obtained through conventional processing of a similar composition.

4. Discussion

4.1. Effect of Melt Superheat

A number of tests were carried out on the 15Cr–4Al alloy in order to evaluate the effect of melt superheat on the solidification behaviour. It was evident that the higher melt superheat showed a higher heat flux and commensurate increase in nucleation density. However, this is the opposite trend to that observed in a previous study on 304 stainless which showed that higher superheats decrease the nucleation density. The data for that study are plotted in Fig. 11, and this graph demonstrates that the nucleation density decreases exponentially, and becomes virtually unchanged above a superheat of ~75°C. This behaviour results from the increased driving force for nucleation of solidification as the solidification temperature is approached. It is apparent that the superheats used in the present study were relatively high, 80 and 130°C. These superheats are probably beyond the region in which the driving force for nucleation is significantly affected by changes in the melt temperature. Instead, at these high superheats, improvements in the nucleation density (and vis-à-vis the heat flux) are likely to be the result of improved wetting of the liquid metal on the substrate at higher melt temperatures.

4.2. Effect of Gas Atmosphere

In this study two casting atmospheres were tested, nitrogen and argon. For the case of the 15Cr–4Al alloy, changing the casting atmosphere from nitrogen to argon had a minor impact on both the nucleation density and the heat flux. However, in the 20Cr–5Al alloy, casting in Ar substantially decreased the heat flux but the nucleation density remained largely unchanged. This is consistent with a previous study that showed that changing the casting atmosphere from argon to helium slightly increased the heat flux, but did not significantly affect the nucleation rate. It has previously been proposed that the increase, or in the case found here, the decrease in heat flux during casting under a different atmosphere, is a direct result of the increased thermal conductivity of the gas. Changing the thermal conductivity of the gas phase trapped inside the melt-substrate interfacial gap affects the conductive heat transfer and heat flux across the interface. The melt surface tension and viscosity that typically control the wetting at the interface remained the same under each atmosphere, as evident by the largely unaffected nucleation density. Despite the unchanged nucleation density, the increase in heat transfer under a nitrogen atmosphere did correspond to an increase in the solidification rate, Fig. 12.

5. Conclusions

An experimental simulation of the strip casting of the Fe-based specialty alloy known as Fecral (or Kanthal) has been conducted. Two alloy variations were examined: Fe–15Cr–4Al and Fe–20Cr–5Al. It has been concluded that:

• The two Fecral alloys considered in this study, 15Cr–4Al and 20Cr–5Al, were found to be castable under strip casting conditions. Surface quality was generally satisfactory, except for some defects observed due to oxides/slag accumulation under the experimental conditions used in this study. The more concentrated alloy was subject to more common sample warping, but this defect is not considered to be detrimental in industrial strip casting.

• Increasing the melt superheat from 80 to 120°C increased the nucleation density and heat flux during casting. It is proposed that this was caused by the improved melt-substrate wetting with increasing melt temperatures.

• It was found that casting under argon instead of nitrogen reduced the interfacial heat flux, possibly due to a decrease in the thermal conductivity of the gas trapped at the melt-substrate interface. This decrease in heat flux corresponded to a decrease in the solidification rate. However, changing the casting atmosphere, as expected had little effect on the nucleation density.

• The two alloy compositions studied here solidified as ferrite, and the as-cast microstructures consisted of coarse columnar grain structures that were typically 60 to 150 μm wide and 500 to 1 000 μm in length. Small inclusions with
an average size of 1 μm uniformly spreads throughout the as-cast structure, and these were identified to be aluminium nitrides.

- The mechanical properties of the rolled and annealed sheets were comparable to those produced by conventional thermo-mechanical processing.

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