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Liquid Metal Feeding Through Dendritic Region in Ni-Hard White Iron

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ABSTRACT

Liquid permeability in the dendritic regions is one of the factors that determine porosity formation and macro segregation in castings. Permeability in the dendritic structure of Ni-Hard white iron was measured as a function of temperature. Effect of microstructural coarsening on the permeability was also investigated. Permeability increased with coarsening dendritic structure in Ni-Hard white iron.

INTRODUCTION

In formation of shrinkage porosity and macro segregation during solidification of dendritic alloys, permeability of the dendritic structure to the interdendritic liquid is one of the important determining factors. Therefore, there have been a number of investigations to date to determine permeability of dendritic structures in different alloys. Pb-Sn [Nasser-Rafi, Streat], Al-Cu [Duncan, Murakami, Piwonka], Al-Si [Apelian], and translucent borneol-paraffin organic systems [Murakami] have been studied using different measuring techniques.

The Interdentritic Flow (IDF) experiments are similar to the experiments performed by D'Arcy to characterize the permeability of different porous media to liquids. The porous medium here is the dendritic structure that has formed as the alloy approaches its eutectic temperature. The liquid, filling the spaces among the dendrites at this temperature is near the eutectic composition. By D'Arcy's equation, the area-averaged velocity of liquid flow through a porous column is proportional to the pressure drop across the column of that medium and also inversely proportional to the viscosity of the fluid [Bejan]. Given constant viscosity, this fluid velocity can be used to determine the "permeability" of the porous column.

D'Arcy's equation,

$$u = \frac{K}{\mu}\left(\frac{dP}{dx}\right) \tag{1}$$

where u is the fluid velocity, K is the permeability of the porous medium, μ is the fluid's viscosity and dP/dx is the pressure differential in direction of flow across the porous medium, can be seen as a force balance. However, this factor actually describes the "effective pore diameter" of the medium and D'Arcy's equation can be derived from a strictly geometrical starting point. It was the purpose of the experiments described here to determine the permeability of the dendritic structure of Ni-Hard white iron alloy to its eutectic liquid. This permeability might then be used to predict the likely pressure drop across a given thickness of casting to assist in the design of pours to toward prevention of porosity caused by decreasing permeability on cooling.

One important distinction of the permeability measurement method employed in this study compared to above mentioned studies is that it allows the flow of liquid through newly formed dendritic structure as in a casting (i.e., formation of dendrites and the flow of interdendritic liquid take place during the same cooling ramp).

EXPERIMENTAL PROCEDURE

A Ni-Hard white iron alloy with an overall chemical composition (in weight%) of 4.48Ni, 2.55Cr, 2.56C, 0.59Mo, 0.56Mn, 0.42Si was used as dendritic (plug) alloy. Its interdendritic composition (eutectic composition) was determined using wave-length dispersive X-ray analysis in an scanning electron microscope as (in weight %) 4.34C, 5.70Cr, 2.51Ni, 0.68Mo, 0.67Mn, 0.14Si, balance Fe. An alloy with this eutectic composition was produced in a vacuum induction furnace to be used in the permeability tests as permeating liquid.

In order to reproduce, as closely as possible the progress and cooling of metal during casting, a process was devised where the Ni-Hard could be melted and then allowed to cool as in a casting. When the Ni-Hard approached the melting temperature (1178ºC) of its eutectic component, a charge of liquid eutectic alloy was allowed to flow through the porous dendritic structure of the mostly frozen Ni-Hard. The progress of the eutectic liquid was noted and used to determine the velocity of the interdendritic flow for the experiment.

To enable this sequence of events, a two-chambered, quartz crucible, Figure 1, was designed and built. The lower chamber was charged with Ni-Hard, the upper with eutectic metal. The floor of each chamber consisted of a removable plate of quartz. The lower chamber also included quartz pegs to prevent the mostly frozen Ni-Hard from falling out during the flow experiment.

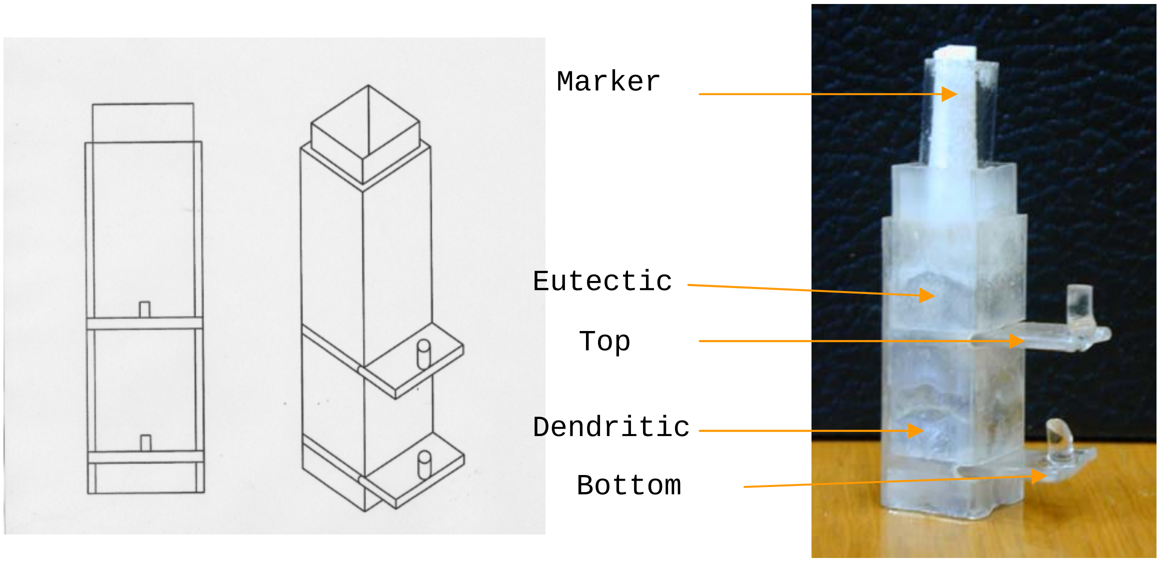

Figure 1: Two chambered crucible for IDF experiments

Preparation for the interdendritic flow experiments began with the measuring of the dimensions of the two-chambered crucible. The dimensions of the cross-section of the crucible were recorded as were the positions of the Ni-Hard retaining pegs. Next, the bottom plate ("gate") was inserted and held in place by a tungsten spring. Following this, charges of Ni-Hard and the eutectic alloy were weighed and Ni-Hard was placed in the lower chamber. The upper gate was then installed, supported by another spring, and the upper chamber was charged with the eutectic.

At the temperatures at which the flow experiments occurred, the metal in the crucible and the ceramic holder used to keep the crucible vertical in the furnace became incandescent, preventing the viewer from distinguishing and tracking the progress of the position of the liquid in the tube during flow. A single-ended quartz tube, tall enough to be discernible above the crucible and holder, was floated on top of the eutectic charge. The charged crucible was then placed in a ceramic holder..

These experiments were carried out in a tube furnace. A flow of argon (99.999 % purity) was maintained in the tube to prevent oxidation. To the crucible gates was attached a rod which extended, through a seal, to the outside of the furnace, allowing the experimenter to manipulate the gates. This assembly was placed in contact with three thermocouples, which extended to the approximate midpoint of the furnace tube. This final assembly, comprising an end-cap of the tube-furnace, the thermocouple sheaths and the manipulating-rod, or "poker", as well as the crucible in its ceramic holder, was slid into the furnace tube until it pressed against retaining rods reaching from the furnace's other end-cap. The furnace tube was then sealed and filled with Ar at low positive pressure. This gas flowed, from front to back, through the tube during all phases of the experimental run. Figure 2 shows a schematic of the experimental setup.

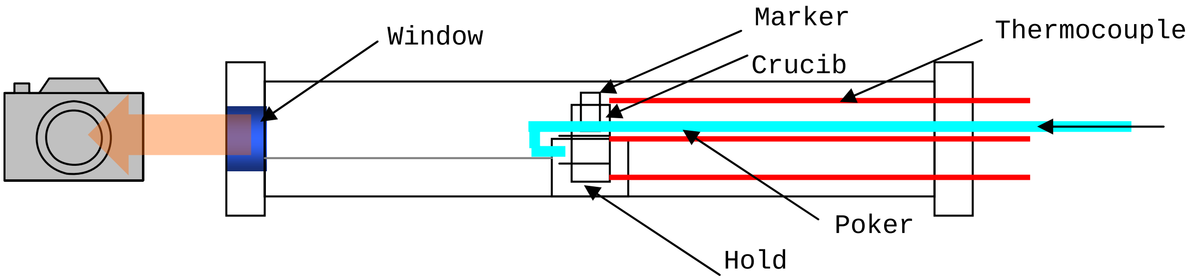

Figure 2: IDF station

The furnace heated the crucible and its charges first to 1360° C at which Ni-Hard was molten, and then free-cooled to the flow experiment temperature. At this temperature (approaching the melting point of the eutectic fluid) the experimenter used the quartz "poker" to pull the gates from their position in the IDF crucible. The top gate opened first, allowing the eutectic charge to fall and settle atop the Ni-Hard plug. The bottom gate was then opened to allow flow through the porous dendritic plug. The progress of the marker as the eutectic fluid flowed through the dendrites, was observed and recorded using a digital camera, fitted with a filter, which peered through a port in the other end-cap into the furnace. The change in position of the marker telegraphed the progress of the liquid flowing through the porous dendritic plug. The flow continued until the marker came to rest on the surface of the porous plug. In order to take into account the dynamic structure of the porous dendritic plug, this flow experiment was conducted either immediately on reaching the desired temperature, or after a pause of as long as 30 minutes, during which the experiment's temperature remained constant.

Recorded images showing the marker's progress were measured to determine the change in position over time. This gave the velocity of the marker, and so, of the eutectic fluid passing through the porous dendritic plug. Pictured below, in Figures 3 is a sample of the recorded images measured to determine flow velocity.

The position measurements were plotted and a line fit to them to determine the magnitude and character of the velocity, u, (see equation 1) of the marker, and so of the eutectic fluid through the dendrites. The slope of the line gave the velocity value for the experiment. The pressure drop through the porous dendritic plug, dP/dx, was taken as the average drop across the plug ($\Delta P/L$). The value for this pressure drop was taken after analyzing the forces present at each end of the plug throughout the experiment. The forces taken into account were the weight-density and volume of eutectic fluid both above and below the dendritic plug, the weight of the marker, and the force exerted by surface tension both above and below the plug. The permeability of the porous dendritic plug was then calculated algebraically through D'Arcy's equation (1).

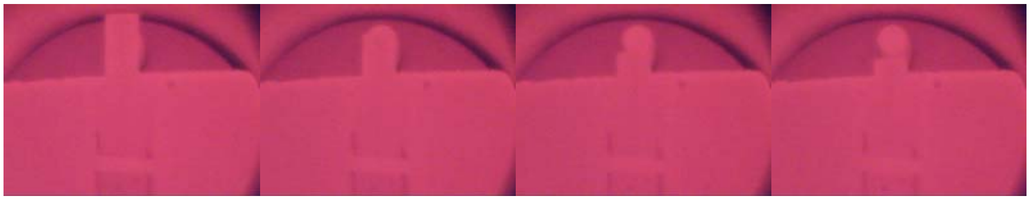

Figure 3: Sample of images measured to track marker position over time.

RESULTS AND DISCUSSION

The eutectic flow velocity and the velocity-derived permeability of the dendritic plug are shown in Figures 4 and 5. The velocity of the eutectic fluid through the dendritic plug was seen to increase with temperature of the experimental system. This increase in velocity for higher temperatures was even more marked in the experiments which soaked the system at the flow temperature before allowing flow. These results follow the accepted relationship between dendrite density and

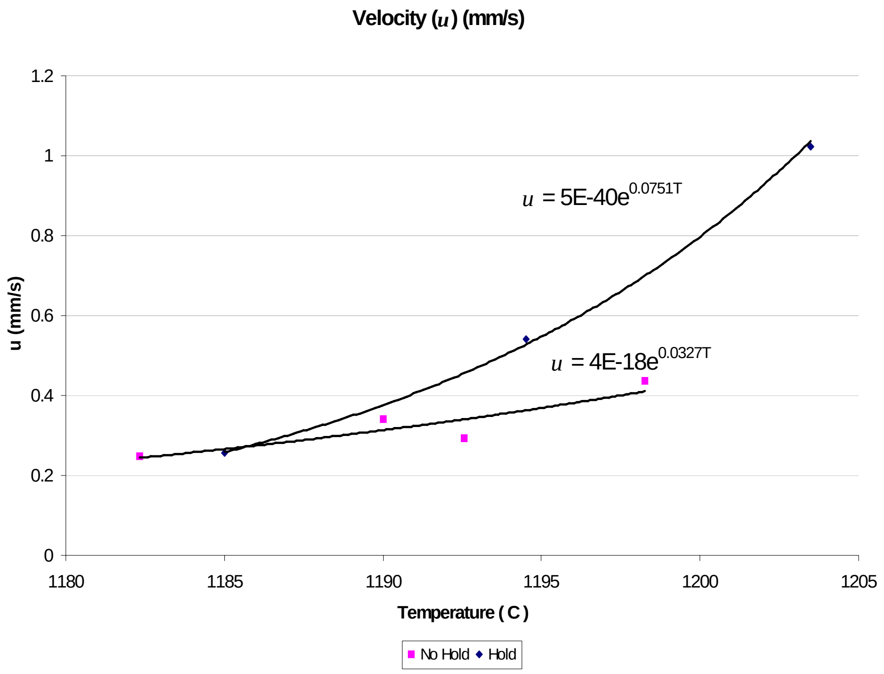

Figure 4: Velocity of interdendritic flow at various system temperatures.

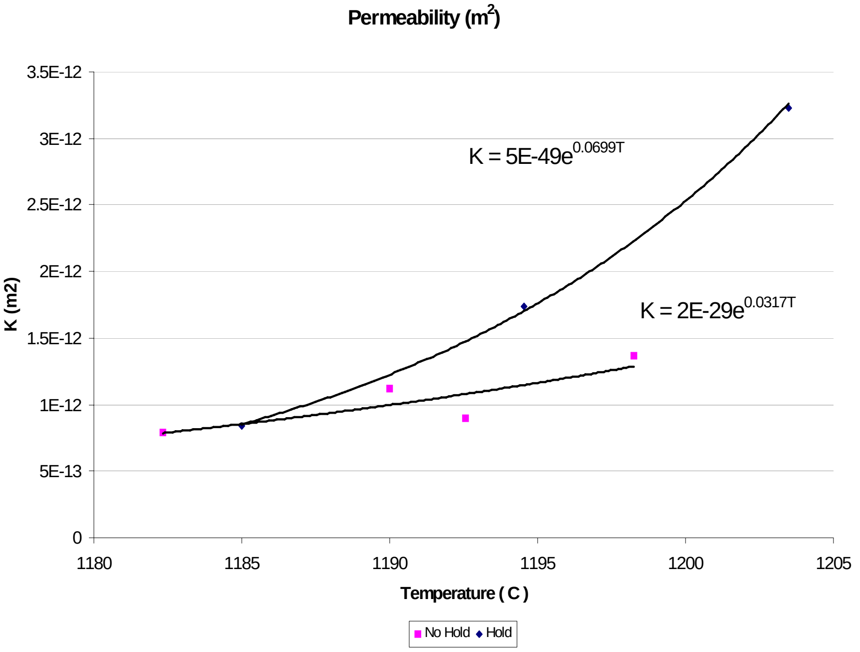

Figure 5: Permeability of the porous dendritic plug, determined through D'Arcy's equation.

coarseness. The cooling rate for the experiments is constant. During cooling, dendrites are nucleating and growing, with nucleation rate increasing and growth rate decreasing with temperature drop. When cooling is halted and the system held at a constant temperature, nucleation for that temperature has largely completed and dendrite growth and coarsening now are the primary change occurring in the system. The coarsening of dendrites increases the permeability of a given structure by decreasing the average surface to volume ratio of the dendrites as they coalesce. Temperature shows an effect here in the rapidity of isothermal coarsening. The higher temperatures show evidence of greater coarsening and so greater increases in permeability given the same amount of time.

The linear relationship between the marker position and time (i.e., constant velocity) as shown in Figure 8 imply the constancy of both the pressure drop across the plug and the viscosity of the eutectic fluid over the time during the flow experiment.

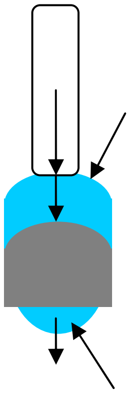

Figure 6: Force balance on interdendritic fluid.

The pressure difference was modeled mathematically, using knowledge of the density of the eutectic fluid, the surface tension results of related sessile-drop experiments with the eutectic alloy, the recorded heights of the liquid column over the dendritic plug and the weight of the marker. The force balance shown in Figure 6 was used to determine the pressure drop across the dendritic plug.

A dominant force was found to be the surface tension on the pendant of liquid that emerges from the bottom of the plug during experiments. This force grows from zero, when the pendant is just appearing to a maximum at the point where the pendant is closest to hemispherical in shape. Beyond this point, the pendant is experiencing plastic deformation and finally necking. To eliminate the regions where the pressure drop would be decreasing (as the pendant approached the hemispherical shape), the change in height of the marker was calculated as a function of the changing volume of the emerging pendant. The pendant volume was modeled by calculating the volume of spherical sections which would include the current pendant shape.   Data points associated with the change in height required to fill this hemisphere were not taken into account in pressure-drop calculations. An acceleration of the flow at the end of most of the experiments was explained by the characteristic increase in fluid flow during drop necking [Shin]. Position data from this acceleration point was also ignored to enable calculations with a constant pressure drop. Figures 7, 8 and 9 show, respectively, the comparison of forces previous to full hemisphere formation, a plot showing regimes of different force balance characteristics, and the appearance of the pendant during plastic deformation, but before necking.

The resulting permeability assumes a constant pressure drop across the plug, which holds both pendant surface tension and meniscus surface tension both at maximum.

Surface tension affects not only the forces involved in the IDF experiment, it also affects the shape of the dendritic plug and the eutectic column. The dendritic plug is thickest in the middle and thinnest at the corners. However, the change in thickness is slight until the corners of the plug are reached. Maximum difference in thickness is approximately 20%. The area of greatest thinning is, essentially, the difference in area of the crucible cross section and a circle inscribed in it (~27%). The average thickness of a corner is approximately 83%. Because the meniscus of the eutectic column is similar in curvature to that of the dendritic plug, the pressure at any point on the surface of the plug is essentially the same. The combination of equal pressure and unequal thickness leads to a probable higher velocity ($\leq$ 17%) of flow at the corners than at the middle of the plug. Since, however, the area experiencing this velocity difference and the velocity difference itself are small, it was decided that an average thickness of the plug would be representative for the determination of permeability.

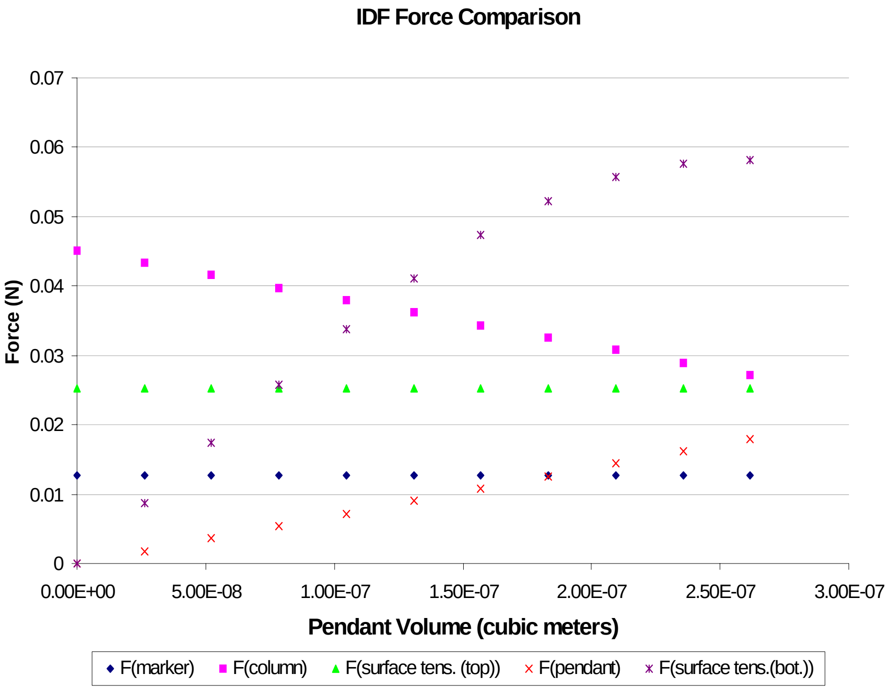

Figure 7: Force comparison as hemispherical pendant is formed.

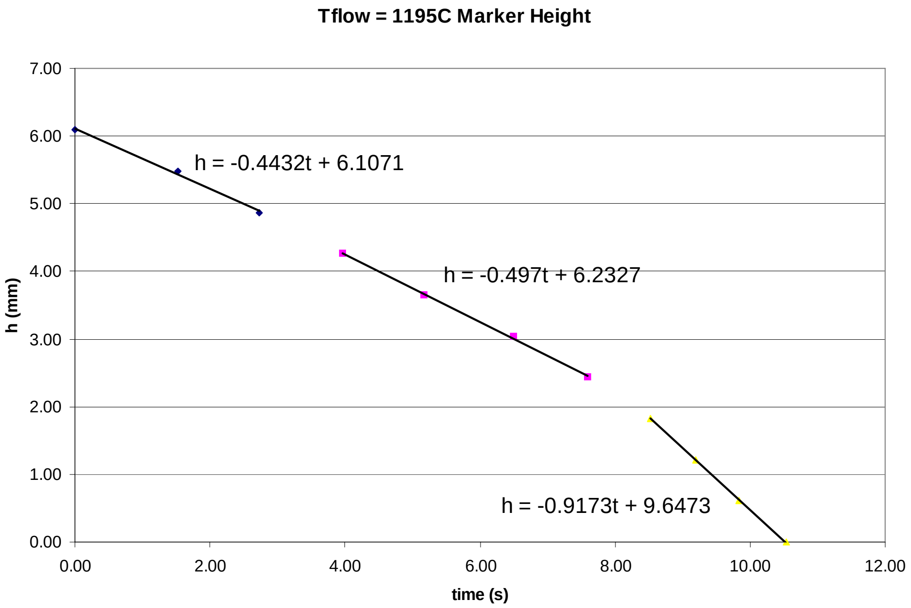

Figure 8: Velocity regimes corresponding to force balance regimes.

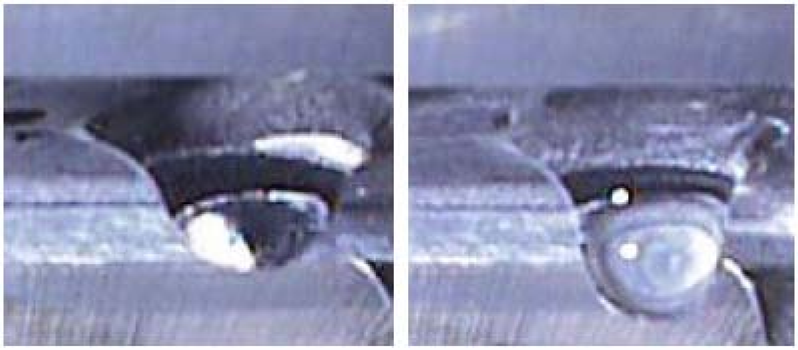

Figure 9: Pendant in plastic deformation, allowing constant flow rate and pressure drop [3].

CONFIRMATION OF LIQUID FLOW PATH

Finally, in order to show that the flow of eutectic liquid was occurring through the dendritic structure, and not around the dendritic plug between the plug and the wall of the quartz cell, an experiment was performed using 304 stainless steel as plug. It was shown that level of eutectic liquid did not change when the stainless steel plug was used. There was no flow through the plug due to lack of interdendritic space in 304 stainless steel, and neither was there any flow at the interface of the stainless steel and the quartz wall.

In another experiment to show the uniformity of the flow, a liquid composition which differed markedly from the eutectic composition and therefore was not near an equilibrium with the dendrites was allowed to flow through the dendritic structure of the Ni-Hard alloy plug. Because of the nonequilibrium case, there was a reaction between the liquid and the surface of dendrites. This reaction zone was delineated using picric etching and observed under an optical microscope as shown in Figure 10. Uniform thickness of the reaction zone suggests that the flow was uniform throughout the dendritic structure with no preferred path.

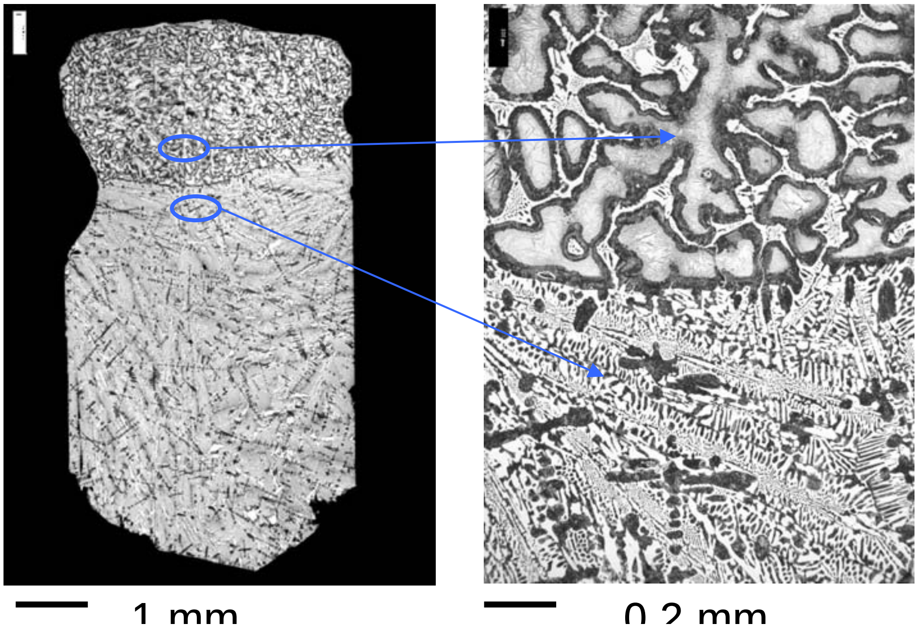

Figure 10: Micrographs showing the interdendritic Ni-Hard plug and the alloy with off eutectic composition solidified after permeating through the interdendritic structure. Note the reaction (darker layer at the periphery of the dendrites) took place between the dendrites and the liquid during the flow.

CONCLUSIONS

The permeability of NiHard's dendritic structure to its eutectic fluid increases slightly on an exponential curve over a 15º – 20º C temperature range near the eutectic reaction temperature. Difference between the permeabilities with and without isothermal holding increases with increasing temperature. As the system approached the eutectic melting temperature, the effect of holding time decreased and seemed to disappear by 1185º C. Therefore, across the temperature range studied, the pressure necessary to maintain a constant liquid velocity would, for flow without coarsening over time, need to be approximately 43% greater at the lowest temperature than at the highest. Coarsening during filling will accomplish the same maintenance of velocity as pressure increase through the upper half of the temperature range, if flow is expected to continue for approximately 30 minutes.

This is the first experimentation with in-situ full-system melting, cooling and flow in metallic alloys to our knowledge. The experiment would benefit from being able to use a larger system, as surface tension effects would become less dominant and steady flow measurements could be taken over a longer period of time.

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