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Observation of the initiation and propagation of solidification cracks by means of \textit{in situ} synchrotron X-ray radiography

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Abstract. We report on the \textit{in situ}, time resolved, observation of solidification cracking in a thin sample of an Al-15wt\%Sn alloy at ESRF BM05. During the experiment, solidification cracking was seen to occur during natural cooling of the sample at a solid fraction of $\sim$95\%, between directionally agglomerated dendritic networks. Through detailed analysis, three stages of crack growth were observed: crack initiation, which typically occurs asymmetrically with the liquid film separating from one side of the dendritic network prior to full detachment; crack propagation, where the liquid film generally detaches from both sides simultaneously; and crack coalescence. We correlate our observations using scanning electron microscopy, which shows that voids between grains, and also spikes, can appear asymmetrically on either side of the crack surface.

1. Introduction

Solidification cracking, or hot tearing, is a significant problem in melt processing, for instance, in direct chill castings [1], single crystal and directionally solidified superalloy castings [2], and welding [3]. It occurs at intergranular and interdendritic boundaries during the final stage of solidification. At this stage, the remaining liquid is increasing in solute concentration and decreasing in volume fraction. In general, it is accepted that hot tearing occurs due to poor liquid feeding to compensate for the solidification shrinkage and thermal deformation. Just as with pores, hot tears can form sub-surface defects in cast products, and so are of extreme concern since they act as stress concentrators and hence are potential initiation sites for fatigue cracking.

In recent years, great advances have been made in the modelling and simulation of solidification cracking, mainly through analyses based upon the principles of granular physics. The Gourlay-Dahle experiment showed dilatancy in a mechanically deformed equiaxed mush, exemplifying the existence and importance of granular behaviour in solidification [4]. This was
subsequently reinforced through in situ radiography experiments [5]. Vernede, Rappaz and co-workers went on to use the methodologies of granular simulations in discrete element simulations of hot tearing [6–9]. These studies show that hot tearing is a highly complex phenomenon, which involves heat flow, fluid flow under capillary action and various other factors including alloy composition and process parameters.

The potential driving forces for hot tearing are well-established: solid contraction in a thermal gradient; solidification shrinkage; and differential contraction between the mould and the solid. Many authors have examined the hot tear susceptibility in terms of the stress and strain rate sensitivity, allowing for coarse-graining into finite element models. As a result, there are many hot tear criteria and these have been comprehensively reviewed by Eskin et al. [1, 10]. Guven and Hunt pointed out a distinction between interdendritic and intergranular hot tearing: interdendritic hot tearing can be induced when shrinkage driven flow is impeded; furthermore, the irregular structure and increased solute content at grain boundaries leads to increased propensity to intergranular hot tearing [11]. Thus, it is pertinent to perform in situ investigations of the microstructural effects on crack initiation and propagation at the dendritic length scale in order to complement the viewpoint already obtained by several authors.

Many of the recent advances in the understanding of hot tearing have been achieved by the application of synchrotron X-ray imaging. Firstly, Phillion et al., performed three dimensional imaging experiments on the aluminium alloy AA5182 in order to characterise the morphology of hot tear damage [12]. More recently in situ ultrafast X-ray tomography and radiography experiments have shown the highly localised nature of hot tearing and cracking in isothermal tensile deformation of semi-solid Al-Cu alloys [13,14].

Using in situ radiography, we have been able to observe cracking during natural cooling following the directional solidification of an Al-Sn alloy. We identify three stages in crack growth which we have termed crack initiation, crack propagation along liquid channels separating dendritic grains, and crack coalescence. In this paper, we present these stages with both evidence from radiography and supporting scanning electron microscopy.

2. Experimental methods
The Al-Sn binary system is well-suited to the study of hot tearing owing to the limited solubilities of the end members of the phase diagram and a broad freezing range, increasing the time that the alloy spends in the brittle temperature range (BTR) during which the solid fraction $f_s$, is between 0.85 and 0.95 [16]. An alloy of composition Al - 15 wt% Sn was prepared by melting in a sealed silica tube before being homogenised at 180°C for 72 hours. Specimens with dimension $37 \times 6 \times 0.2$ mm$^3$ were produced and polished in order to have a uniform thickness and to reduce spurious nucleation on scratches and other defects. Laboratory and synchrotron radiographs of the as-prepared specimens showed no evidence of porosity, hot tearing or macro segregation.

The in situ radiography experiment was performed at the BM05 station of the European Synchrotron Radiation Facility. The apparatus, beamline configuration and experimental protocol are well established and detailed in various publications [17, 18]. A bespoke two-zone Bridgman furnace was used for the experiments with thermal control offered by regulating the power to the heaters and an initial temperature gradient of approximately 40 K cm$^{-1}$. During the first stage of the experiment, cooling was achieved by reducing the temperature at the top of the specimen at a rate of 0.75 K min$^{-1}$ whilst maintaining a constant temperature at the base of the specimen.

After the solidification was essentially complete, the power to both heaters was switched off and the sample allowed to cool naturally. During this second phase, the temperature gradient along the sample rapidly decreased towards zero as seen in Figure 2.

During solidification, radiographic images were recorded with a Fast Readout Low Noise (FReLoN) camera at a frequency of 1Hz and an exposure time of 0.3s. The incoming synchrotron...
Figure 1. General overview of the solidification experiment. A mixed columnar-equiaxed microstructure is observed to emerge with hot tearing occurring at the later stages of solidification. The times shown are those elapsed from the moment at which cooling was initiated. Two cracks are encircled, and the crack under consideration is identified in the white circle at the bottom right of the last frame ($t = 4053$ s).

Radiation was tuned to a photon energy of 29.2 keV using a Si (111) two crystal monochromator. By selecting an energy just above the absorption edge in the Sn spectrum, stronger contrast between solid and liquid can be obtained. The as-recorded images were processed using ImageJ [19]. All images were divided by a reference image, which showed a homogeneous liquid with a solid seed at the bottom of the field of view at the start of the experiment. This image processing eliminated spurious features not associated with the solidification [18]. In addition, image subtraction was employed with respect to a reference image to highlight microstructural changes between acquisitions. In each figure shown in the later sections, the first recorded image has been used as a subtraction reference. After the ESRF experiment, examination of
Figure 2. Thermometry data from the experiment is provided for reference with Figure 1: growth occurred predominately during controlled cooling and the hot cracking initiated midway through natural cooling.

the cracked specimen was performed using a JEOL 5500 LV SEM at 10kV.

3. Results
Figures 1 and 2 shows the whole field of view and the temperatures recorded by the two thermocouples during the experiment. In the first four images, a thermal gradient exists across the specimen with the temperature at the bottom of the field of view lower than that at the top. At $t = 616$ s, cellular and dendritic features were observed to grow out into the liquid on an otherwise smooth solid liquid interface. Two thirds of the way down the image, equiaxed grains can be seen to have nucleated and grown out to form the directionally agglomerated structure displayed at $t = 3400$ s. The top of the image appears lighter in the last image owing to shrinkage driven flow down into the specimen. This leads to the formation of a meniscus, effectively reducing the thickness which must be traversed by the beam and increasing the transmitted intensity.

Intergranular cracks were observed to form either at the boundaries between different columnar grains (bottom circle in Figure 1) or at the junction of columnar and equiaxed regions (upper circle in Figure 1). Cracks can be identified by a transition from black (liquid channel) to lightest grey (crack), encircled in the image at $t = 4053$ s. The equilibrium phase diagram suggests that cracking initiates in this case at a solid fraction of $f_s = 0.955$, which is comparable with the results of Guven and Hunt [11]. The crack observed at the lower right hand corner of the field of view is examined in more detail in the later sections. Figures 3 - 5 show sets of radiographs in which we identify three crack stages: crack initiation, crack propagation and crack coalescence.
3.1. Crack initiation

Figure 3 exemplifies the asymmetric detachment mechanism observed in the initiation of the intergranular crack. The first direct image at $t = 3552$ s shows the dendritic microstructure just prior to any observed cracking. In the bottom centre of the image, a set of dendrites is observed. Whilst it is visible in the direct images, the subtracted image highlights a bright white feature. This can be readily identified as the thinning and detachment of the liquid film at the dendrite tips. This detachment is due to the faster downward motion of the lower grain that results in the stretching of the liquid channel. The subsequent images clearly show the crack extending along the lower solid-liquid interface, along the dendrite tips. Careful comparison of the direct and subtracted images shows that the detachment has first occurred at the dendrite tips on the lower side of the liquid channel and extends along the channel lengthways, before widening at $t = 3559$ s. Once the crack has established on one side, capillary forces extrude the remaining liquid, resulting in the full detachment of the liquid film from the opposing solid liquid interface and the oxide layer acting as a mould.

3.2. Crack propagation

Figure 4 shows the symmetric propagation of a crack towards a triple junction. It is clear from the subtracted images that there is no asymmetry in the propagation of the crack as it approaches the triple junction. Instead, the void approaches the triple junction with both solid-liquid interfaces detaching almost simultaneously. However, when it reaches the large dendrite tip at the bottom right hand corner of the image, the crack bifurcates into the liquid channel on the right with liquid first detaching asymmetrically from the large dendrite opposing its straight propagation.
Figure 4. Symmetric crack propagation along a liquid channel down to a triple junction, where the crack propagation bifurcates asymmetrically into the channel on the right when it encounters the lower dendrite network.

Figure 5. Initiation of a new crack and its coalescence with a pre-existing crack.
3.3. Crack coalescence

Figure 5 shows the coalescence of two cracks. In the direct image at \(t = 3597\) s, a pre-existing crack is seen at the bottom right hand corner of the image. In the upper middle of the images, a second thinning event has occurred ahead of the main crack. Then, the main crack extends towards the newly nucleated detachment in a symmetric manner. The two cracks subsequently join in a time interval that is not clearly resolvable at the present image acquisition rate.

4. Discussion

In the previous section, we presented the three observed stages of hot tearing, namely crack initiation, crack propagation and crack coalescence. During the experiment, crack initiation and propagation was found to be mostly asymmetric in nature, suggesting this is the kinetically favoured process for hot tearing to proceed in this situation. This is plausible, as beyond relaxing mechanical stress induced by the moving apart of the dendritic grains on the two sides of the liquid channel, which may itself be asymmetric, asymmetric detachment also provides a reduced barrier to crack initiation. Indeed, the detachment turns a solid-liquid interface into a pair of solid surfaces which comes with an energetic penalty. Thus, since only one detachment event is required for asymmetric propagation, it provides an energetically favourable route for initial detachment. The increased capillary forces owing to the increased liquid-void surface area of the remaining liquid then provide a driving force for liquid extraction.

Previous scanning electron microscopy on metallic alloys have shown that ‘spikes’ form on hot tear surfaces (see e.g. Figures 7-9 in by Farup et al. [20]). Figure 6 shows similar features. Unlike the previous study, the features appear on one side only of the crack, corresponding to the side on which detachment occurred first.
5. Conclusions

*In situ*, time resolved radiography has been used to identify features in the initiation and propagation of inter granular solidification cracks, namely the occurrence of three distinct stages in crack growth: initiation, propagation into liquid channels separating dendritic grains and crack coalescence. This leads to interesting microstructural features, with similarities and contrasts to be drawn with prior studies, especially regarding asymmetric features such as bridging spikes.

Further efforts will be necessary to examine the applicability of these results. In particular, the reduced thickness of the system may have an effect on its hot tearing behaviour. Furthermore, the issue of the dynamical interaction between melt and the soft oxide layer that encases the solidifying sample should deserve specific attention. This is, of course, a recurrent, open question in aluminium alloy casting [21, 22].

The asymmetric mode of propagation described deserves further investigation, both through physical modelling and further experiment. Indeed, this may have important implications on crack initiation and propagation. Fuller quantitative analysis of this data is already underway to facilitate further interpretation.

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