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Effect of solidification conditions and surface pores on the microstructure and columnar-to-equiaxed transition in solidification under microgravity

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A B S T R A C T

Microgravity solidification experiments were carried out in the Material Science Laboratory on board the International Space Station. The influence of grain refinement, rotating magnetic field (RMF) and surface pores on the microstructure and columnar-to-equiaxed transition (CET) were investigated in two selected Al-based samples solidified under microgravity conditions. The increase of the furnace pulling velocity leads to a finer dendrite structure, a smaller eutectic percentage and a more uniform eutectic distribution in the interdendritic regions. On the one hand, grain refinement ensures the occurrence of CET, which is progressive in the studied experiment because of the high temperature gradient. On the other hand, in the non-refined alloy a RMF applied during solidification fails to trigger the CET, because the forced liquid flow is too weak compared to the solidification front velocity to transport fragments from the mushy zone above the solidification front. The presence of the pores at the sample surface leads to a peculiarity in the eutectic percentage and weakens the decrease of the dendrite arm spacing for both samples. These effects are ascribed to a forced extra liquid flow into the mushy zone due to the pore that promotes the growth of the dendrites along the liquid flow direction, resulting in elongated grains and postponing the CET in the refined alloy.

1. Introduction

Morphological development during solidification remains of tremendous interest for both academic research and industrial applications, due to the tight relationship between the microstructures and the final properties [1]. Indeed, growth in a high temperature gradient may result in elongated columnar structures, whereas a more uniform temperature favors isotropic equiaxed grains. Accordingly, a change from columnar to equiaxed grain structure is often observed during industrial processes such as ingot casting or welding by a simultaneous decrease of the temperature gradient and an increase of the growth velocity. CET is an important morphological transition in metallic alloys, which is mainly controlled by the liquid undercooling ahead of the dendritic front [2]. CET occurs when the number of equiaxed grains and the volume they occupy become sufficient to block the columnar front [3–5].

Equiaxed grains have two main origins: purposely added nucleating particles and dendrite fragmentation. Industrially, the main approach to favor the equiaxed microstructure is to add grain refiners, which act as preferential sites for nucleation with a low nucleation undercooling [6,7]. For non-refined alloys, the most potent mechanism for the origin of the equiaxed grains ahead of the columnar front is the dendrite fragmentation [8,9], which is believed to be at the origin of the central equiaxed core region in casting processes. Fragmentation occurs when dendrite branches are detached from the main primary trunks, as well as from the secondary or higher order arms. Fragmentation is favoured by the dendrite coarsening process but also by the initiation of local remelting due to the pile-up of solute within the partially solid-liquid sample region (mushy zone) [10,11]. The additional effect of gravity force (buoyancy force or natural convection) [12] or the impact of thermo-electro-magnetic force [13] cannot be excluded in fragmentation phenomena. If these fragments are transported ahead of the columnar front by buoyancy forces or convection and if they can continue to grow, an equiaxed grains microstructure is

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formed that can stop the advancing columnar front. The competition of equiaxed grains with the advancing solidification front is a complex topic, which involves mechanical blocking as well as thermal and solutal interaction. J.D. Hunt proposed a geometrical criterion to explain this transition in terms of mechanical blocking [3], but it is now recognized that the dominant mechanism for the CET is the solutal blocking, due to solute rejection from growing equiaxed and columnar dendrites [5].

CET as well as the final microstructure features are strongly affected by gravity induced phenomena namely, sedimentation effect and coupling between flow and the solid-liquid interface. On Earth (1 g), the melt flow plays a critical role in mass and heat transfer, and consequently affects the final microstructure, as well as the CET and the eutectic formation [14–17]. Microgravity experimentation is a perfect tool to deepen the study of CET, as it provides unique benchmark data by suppressing most of the gravity-driven phenomena during solidification, such as natural convection, as well as sedimentation or buoyancy. In microgravity environment, we can expect to achieve nearly diffusive conditions for transport [18–20]. Nevertheless, even in gravity-free conditions, fluid flow, generated by the sample shrinkage induced by the difference of solid and liquid densities, still exists [21,22]. Another cause of fluid flow in gravity-free conditions is Marangoni convection [23,24], due to the presence of free interfaces like liquid-gas surfaces. This contribution to convection is often negligible in directional solidification for which the only gas-liquid surface is at the top of the sample.

Porosities are considered as serious and common defects formed during solidification [25], which result in a significant degradation of the mechanical properties and act as the origin of crack initiation because of stress concentration [26–28]. Up to now, the formation of porosity has been considered by numerous investigations, but, as a matter of fact, mostly focusing solely on the pore formation mechanism [29–35]. It is generally accepted that the residual gases, which are mainly hydrogen, dissolve and accumulate at the solidification front, since gas solubility is often much less in the solid than in the liquid. As a result, bubbles readily nucleate to form porosities, not only at the crystal or dendrite interface, but also on the sample surface [31,32,36]. Also reactions between the melt and the crucible walls at elevated temperatures may play a role.

Except their impact on crack initiation, the porosities also affect the solidified microstructure by triggering the deformation of the dendritic network [37,38] and creating Marangoni liquid flow around the pore region during the solidification [39–42]. Indeed, it has been reported that the mushy alloy could deform under external loads [43–45]. The deformation mechanism changes with the increase of the solid fraction [43]. The spongy deformation of the mushy zone during the solidification is also reported by Lesoult et al. together with the effect on segregation [46]. Additionally, primary crystal deformation and flow of solute-enriched liquid towards the shear deformation plane have also been observed [47,48]. Therefore, combined with the Marangoni effect, the liquid flow triggered by porosity formation should also be considered for the microstructure transformation. However, these porosity impacts on the microstructure features and CET have rarely been mentioned in experimental or numerical investigations before, to the best of our knowledge.

Our investigations are conducted by means of directional solidification in microgravity conditions. The quantitative analyses of the eutectic fraction and the dendritic structure, as well as the CET are provided and correlated with the solidification parameters. In addition, the influence of a pore at the surface of a sample on the microstructure formation has been analyzed in detail.

2. Experimental

2.1. Experiments

The Material Science Laboratory (MSL) was made available by the European Space Agency (ESA) on board the International Space Station (ISS), providing a platform for microgravity solidification investigations. In the framework of the CETSOL (Columnar to Equiaxed Transition in SOLidification processes) ESA MAP (Microgravity Applications Promotion programme) project [49–51], thirteen microgravity experiments have been conducted in two batches named Batch 1 and Batch 2a. The six samples in Batch 1 were carried out using the Low Gradient Furnace (LGF), which have presented and discussed in detail in a previous study [19]. The samples in Batch 2a, which are the objects in this paper, were carried out using the Solidification and Quenching Furnace (SQF), which is also a directional solidification furnace but allows higher temperature gradient and cooling rate compared to the LGF.

The full SQF and the sample cartridge assembly (SCA) are schematized in Fig. 1. It consists of a hot and a cold zone separated by a so-called adiabatic zone. The cold zone is realized by a Liquid Metal Ring (LMR) to achieve a high temperature gradient (up to 8 K/mm in this SCA) and to allow for quenching. The hot zone is equipped with four heaters H1-H4, which can be adjusted independently to achieve the required temperature gradient along the sample axis. Solidification of the alloy is performed by the controlled displacement of the SQF along the fixed SCA at a chosen velocity, which can be varied during the experiment. The metallic alloy sample (8 mm in diameter and 245 mm in length) is mounted inside a protective Al_2O_3 tube crucible together with Shapal plugs at both bottom and top ends. Twelve N-type (Nicrosil-Nisil) thermocouples (TC1-TC12), spaced by 20 mm, are located in four machined external axial grooves at the outer surface of the crucible to measure the temperature profile (purple dots in Fig. 1).

In the present work, a comparative study of two samples, labelled B2F1 and B2F2, from Batch 2a is conducted. The solidification conditions of the two selected experiments are the same in

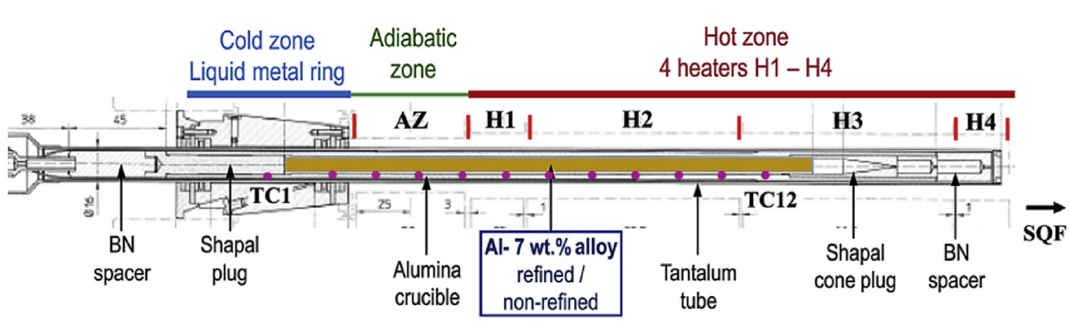

Fig. 1. Sketch of a CETSOL Batch 2a cartridge MSL-SCA integrated in the MSL-SQF furnace showing the situation before solidification with the integrated Al - 7 wt.% alloy sample.