doi:10.1017/S1431927619005191
Microsc. Microanal. 25 (Suppl 2), 2019

Understanding Microstructural Evolution in ZrC Inoculated $Zr_{47.5}Cu_{45.5}Al_5Co_2$ Via High Resolution SIMS

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Since their discovery at Caltech in the late 1950s [1], bulk metallic glasses (BMGs) have demonstrated high yield strengths attaining values approaching the theoretical limit [2] due to the absence of slip mechanisms. However, BMGs lack adequate fracture toughness that would make them suitable for widespread use in structural engineering applications. Upon yielding, BMGs tend to form localized shear bands that, without geometric confinement, cause them to fail suddenly and catastrophically [3]. In order to address this problem, crystalline composites have been synthesized, resulting in metallic glasses with higher ductility [4]. These so-called bulk metallic glass matrix composites have attracted attention because they maintain the superior strength, hardness, and large elastic strain limit of traditional BMGs while simultaneously increasing the plasticity and fracture toughness. One such system and the one studied here is the $Zr_{47.5}Cu_{45.5}Al_5Co_2$ system. Zirconium based metallic glass systems have been widely used, some even showing promise towards industrial applications, especially in harsh environments [5, 6]. Cobalt is added in order to enhance the corrosion resistance of the resulting material even though the glass forming ability (GFA) is slightly inhibited with its inclusion [7].

One way to introduce crystallinity into a BMG system is to introduce inoculant material into the melt solution to induce heterogenous nucleation during melt solidification which can result in partial crystallization [8]. Ceramics such as ZrC, SiC, and carbon fibers are often used as inoculants. Previously [9, 10], such inoculation has been shown to increase the ductility and facture toughness of BMG composites. However, the liquid-to-crystalline phase transformation is still not very well understood. Here, we introduce a method for performing high-resolution Secondary Ion Mass Spectroscopy (SIMS) to elucidate compositional gradients in the $Zr_{47.5}Cu_{45.5}Al_5Co_2$ system inoculated with ZrC.

Suitable ZrC inoculant is added and the melt solidifies via vacuum suction casting [11]. This process results in a wedge of varying thickness which represents variable cooling rates (faster rates occur at the thinner edge of the sample). After solidification, the sample is cut, mounted, and polished. Figure 1 i) shows a schematic of the sample. In this paper, the resulting microstructure is examined using scanning electron microscopy (SEM) in backscattered imaging mode and the phase composition is studied via high-resolution SIMS. The compositional analysis of the resulting microstructures allows for new insights into the nucleation and growth of different phases facilitated by ZrC inoculants. It was found that a 0.5 wt% addition of inoculation casts an effect on the evolution of morphology of phases observed over a range that also corresponds to the cooling rate of the melt.

The ZEISS ORION NanoFab was used to spatially resolve the elemental composition of the Zr-based BMG. A specially designed magnetic sector spectrometer [12] was integrated with the NanoFab and used to enable analytical capabilities to the already impressive high resolution secondary electron imaging Gas Field Ion Source (GFIS) [13]. A highly focused Ne^+ primary beam was used to produce secondary ions (SIs) in a localized region. These SIs were then extracted and mass separated via magnetic field towards four separate detectors lying on the same magnetic focal plane. This tool enables the analysis of surface elemental compositions with a resolution approaching 10 nm. Shown in Figure 1 are the elemental compositions of a representative three separate areas corresponding to the top, middle, and bottom of the wedge. Interestingly, both the morphology and elemental distribution varies as a function of position on the wedge. Of note is the non-homogenous distribution of Zr in the thin region of the wedge. It is hypothesized that preferential nucleation due to crystallographic orientation is the cause of this distribution.

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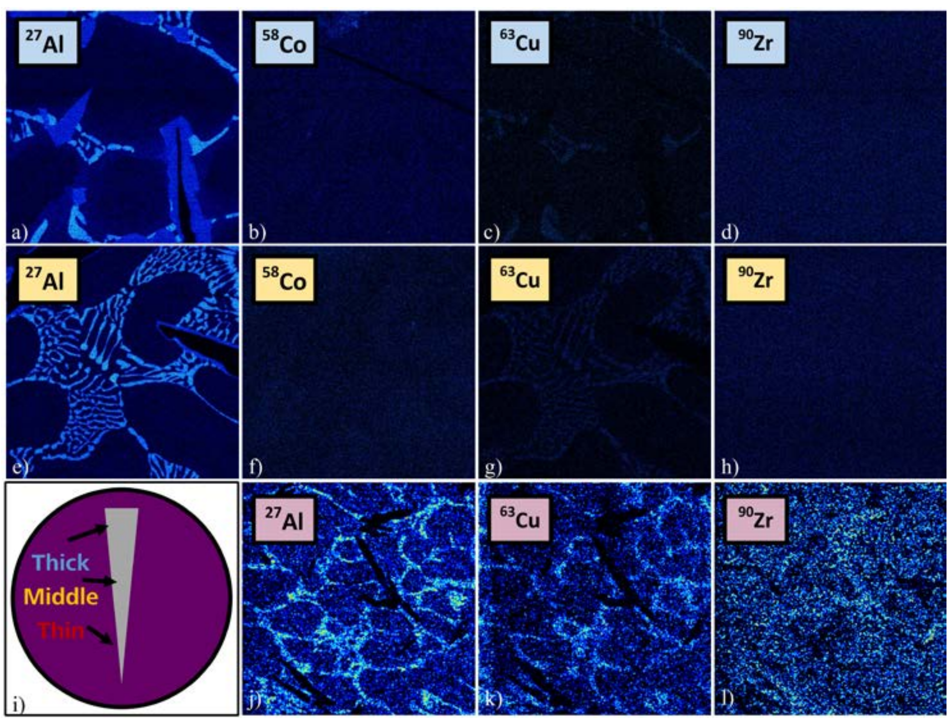

Figure 1: SIMS elemental maps of various regions on the Zr-based BMG composite with 0.5 wt% ZrC inoculant. Shown are representative mass separated images for the thick part of the sample a – d, the middle of the sample, e – h, and the thin part of the sample j – l. The elements measured were aluminium, cobalt, copper, and zirconium as labelled. All fields of view are 10 µm.

https://doi.org/10.1017/S1431927619005191 Published online by Cambridge University Press