Understanding Microstructural Evolution in ZrC Inoculated Zr$_{47.5}$Cu$_{45.5}$Al$_5$Co$_2$ Via High Resolution SIMS

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Introduction

Bulk metallic glasses (BMGs) have demonstrated high yield strength, hardness and elastic strain limit, attaining values approaching the theoretical limit due to the absence of slip mechanisms [1]. However, BMGs they lack adequate fracture toughness that would make them suitable for widespread use in structural engineering applications. Upon application of load yielding, BMGs tend to form localized shear bands, that without geometric confinement, cause them to fail suddenly and catastrophically [2]. In order to address this problem, crystalline composites have been produced successfully, resulting in metallic glass-matrix composites with higher ductility and toughness [3]. In particular, zirconium based metallic glass systems have been widely used, some even showing promise towards industrial applications, especially in harsh environments [4,5]. The materials system studied here is Zr$_{47.5}$Cu$_{45.5}$Al$_5$Co$_2$.

To increase crystallinity, an inoculant induces heterogenous nucleation during melt solidification which results in partial crystallization [8]. However, the liquid-to-crystalline phase transformation is not still very well understood. Here, we introduce a method for performing high-resolution Secondary Ion Mass Spectroscopy (SIMS) to elucidate compositional gradients and the resultant changes in microstructure in the Zr$_{47.5}$Cu$_{45.5}$Al$_5$Co$_2$ system inoculated with 0.5 wt% ZrC.

SIMS Basics

Dynamic Secondary Ion Mass Spectroscopy (SIMS) is performed using a focused energetic ion beam

• The primary beam is Ne$^+$ produced via our GTS column in the NanoFab [14]

• Suction casting is used to reach the cooling rates necessary (10$^6$ – 10$^7$ K/s) to achieve a glassy microstructure

• The wedge shaped mold results in cooling rate gradients which impacts the resultant microstructure

Microstructure Investigation

Shown are the spatially resolved elemental compositions of a representative three separate areas corresponding to the top, middle, and bottom of the wedge. Interestingly, the morphology, reaction (eutectic eutectoid), reaction product 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 inhomogeneity distribution. Importantly, the high lateral resolution of the SIMS instrument reveals morphologies that would be otherwise indistinguishable with other SIMS techniques.

Spectrometer Design

Notable Specifications

• Fully retractable extractor

• Double focusing magnetic sector (Mattsch Hering design)

• Bipolar fields for detecting both positive and negative ions with minimal re-alignment

• Transmission efficiency: 40%

• TIC detector, 1 fixed detector, and 3 moveable detectors

• Mass resolution (M/ΔM) is 400

• m/z ratio is 1 to 500

Measurement Details

• While sputtering with Ne$^+$, mass spectra is obtained by sweeping the detectors along the magnetic focal plane via individually actuated piezo motors with a constant magnetic field

• Elemental maps are constructed by rastering the Ne$^+$ beam across the sample with the detector locations fixed at the position corresponding to the mass of interest. The count rate on each detector is recorded and assigned to each pixel in the image as a color intensity

SIMS Sensitivity

• One of the main concerns that becomes important when the SIMS resolution shrinks is the sensitivity. In essence, as the volume of material that is being analyzed gets smaller the number of ionized sputtered atoms likewise decreases. The sensitivity is related to the ionization probability expressed by:

  Useful Yield (UY) = # secondary ions detected

  # of sputtered ions

• Shown to the left is a depth profile performed on a silicon wafer implanted with boron to a known concentration. A crater or 10 x 10 x 0.1 µm was milled using the focused Ne$^+$ beam

• The measured UY was 6 x 10$^{-6}$ which corresponds to a detection limit on the order of 100 ppm

References