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Beryllium-distribution in metallic glass matrix composite containing beryllium

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Abstract: The morphologies, sizes, compositions and volume fractions of dendritic phases in in situ Ti-based metallic glass matrix composites (MGMCs) containing beryllium (Be) with the nominal composition of $Ti_{47}Zr_{19}Cu_5V_{12}Be_{17}$ (mole fraction, %) were investigated using XRD, SEM, EBSD, TEM, EDS and three-dimensional reconstruction method. Moreover, visualized at the nanoscale, Be distribution is confirmed to be only present in the matrix using scanning transmission electron microscopy−electron energy loss spectroscopy (STEM−EELS). Based on these findings, it has been obtained that the accurate chemical compositions are $Ti_{28.3}Zr_{19.7}Cu_8V_{6.4}Be_{37.6}$ (mole fraction, %) for glass matrix and $Ti_{62.4}Zr_{18.4}Cu_{2.6}V_{16.6}$ (mole fraction, %) for the dendritic phases, and the volume fractions are 38.5% and 61.5%, respectively. It is believed that the results are of particular importance for the designing of Be-containing MGMCs.

Key words: metallic glass; composites; microstructure; electron energy loss spectroscopy; Be-distribution

1 Introduction

Metallic glasses (MGs) have attracted great attention due to their unparalleled mechanical properties [1−5] such as high strength, large elastic limit and good wear resistance. However, the lack of global plasticity, caused by the localized shear bands, has limited their engineering applications. Much effort has been put into optimizing the composition and the microstructure of MGs in order to overcome this problem [6−8]. In situ metallic glass matrix composites (MGMCs) [9−13] have been proved to be the efficient way to decrease the brittleness of MGs. It has been found that the precipitated crystalline phase can obstruct the unlimited propagation of the single shear band and promote the formation of multiple shear bands, which improves the global plasticity [9]. Since the first report of Zr-based MGMCs by HAYS et al [9], a number of such materials with varying components have been developed, e.g., Zr-based MGMCs [9−15], and Ti-based MGMCs [16,17].

In particular, Be-containing MGMCs have drawn great attention thanks to their superior mechanical properties [11,9,18], such as high strength and plasticity. Be element, as one of the great important elements to the applications in aerospace and nuclear industries [19], has been confirmed to improve the mechanical properties [20], glass forming ability [21,22] and thermal stability [15] of MGs. Meanwhile, Be element in MGMCs has been proved to improve mechanical properties [14,17] due to its low density and high elastic modulus. Therefore, the analysis of the Be distribution in alloys is of great significance. However, the current

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method to characterize the composition distribution in Be-containing MGMCs is mainly limited to energy dispersive X-ray spectroscopy (EDS), which is unfortunately not sensitive to light elements, including Be. While most compositional studies of the Be-containing MGMCs are limited to heavy elements [23], little progress has been made on Be distribution which is crucial for understanding the material. Although previous studies have predicted that Be is partitioned into the glass matrix [9,17], direct investigations of the element distribution have not yet been reported.

In this work, the Be distribution is revealed by using scanning transmission electron microscopy−electron energy loss spectroscopy (STEM−EELS). By further investigating the volume fraction of a dendrite using image quality (IQ) and EDS in scanning electron microscope (SEM), the compositions of the two phases in these Be-containing MGMCs have been quantified.

2 Experimental

2.1 Sample fabrication

Ingots with the nominal composition of $Ti_{47}Zr_{19}$-$Cu_5V_{12}Be_{17}$ (mole fraction, %) were prepared by arc-melting, a mixture of high purity elements (>99.9%) under an argon atmosphere. Rod-like composite samples were fabricated by Bridgman solidification [24].

2.2 Microstructure and composition characterization

The microstructure was characterized by using FEI Quanta 250 SEM. The crystal structure of the samples was analyzed by X-ray diffraction (XRD) using a Cu K_α radiation in Bruker D8 Advance diffractometer. The 3D study of the structure was performed on a dual beam focused ion beam/SEM(FEI Helios NanoLab 600i) via serial-sectioning procedures. The 3D reconstruction was performed by using Amira software. The chemical composition was determined by means of Hitachi S−3400N SEM with X-Flash ® 5030 EDS and the FEI Titan ChemiSTEM equipped with Gatan GIF Quantum 965. The convergent angle and collection semi-angle for STEM−EELS are of 21 mrad and 40 mrad. Electron backscattered diffraction (EBSD) analysis was conducted on JEM−6500F with EDAX-TSL EBSD. During EBSD testing, the electron beam scanned on the sample surface and generated a Kikuchi pattern composed of intersecting Kikuchi bands at each measurement point [25]. The image quality (IQ) parameter describes the quality of the Kikuchi pattern, which is the representative of lattice planes in the diffracting crystal. Thus, the amorphous matrix cannot generate the Kikuchi pattern, and the IQ is close to zero. By means of the IQ, we can get the volume fraction of the dendrites in a more accurate and efficient way. Transmission electron microscopy (TEM) analysis was performed using a JEOL−2010F equipped with a field emission gun operated at 200 kV. The TEM samples were cut from the rod-like composite samples, which were mechanically polished and then ion-milled until perforation at 5 kV using a precision ion polishing system (Gatan model 691PIPS) with a liquid nitrogen cooling system operating at −150 °C.

3 Results

Figure 1(a) shows typical SEM backscattered image of the composites. It could be seen that the crystalline phase (dark contrast) is formed and it is uniformly dispersed in the amorphous glassy matrix (bright contrast) throughout the entire cross section. In terms of the composites, XRD shows diffraction peaks from β-Ti superimposed with the broad diffuse scattering features from the glassy matrix as shown in Fig. 1(b). The volume fraction of the dendrites can be determined from the peak area ratio, which was found to be about 60%. Since the microstructure of a composite is strongly related to its mechanical properties, a statistical study of the dendritic diameter distribution has been performed using TEM images. The dendritic diameter is 0.5−2 μm, as shown in Fig. 1(c). The inter-dendritic distance is about 300 nm, as shown in Fig. 1(d), which is consistent with previous reports [16,17,24]. It is believed that a smaller inter-dendritic distance has a positive influence on the ductility of the glass [4].

The microstructure of the reinforced crystalline phases dispersed in a glassy matrix plays an important role in determining the mechanical properties of a composite. However, current morphological studies mostly rely on two dimensional (2D) images, which are insufficient for the understanding of all features and connections between the dendritic structures. A more comprehensive understanding of the dendritic structure in 3D is therefore desirable in order to provide a direct visualization of the composite. Figure 2(a) shows a snapshot of the dendritic 3D network. A serial snapshot of the 3D structure can be found in the supplementary information. The crystalline phase forms interconnecting dendritic structure which is homogeneously dispersed within the amorphous matrix. The good connectivity of such reticular structures efficiently prevents the rapid expansion of the shear zone. The volume fraction of the dendrites is almost the same in every section.

The volume fraction of the dendrites has an important effect on the performance of the material. This fraction is usually determined by analyzing the contrast in images [26] or by differential scanning calorimetry analysis [27] which may introduce a significant