HRTEM study of rejuvenation in metallic glasses under cryothermal cycling

Ivanov, I.[1], Ketov, S.[2], Louzguine-Luzgin, D.V.[3], Eckert, J.[2,4] and Greer, A.L.[1]

[1] Department of Materials Science & Metallurgy, University of Cambridge, United Kingdom, [2] Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria, [3] WPI Advanced Institute for Materials Research, Tohoku University, Japan, [4] Department Materials Physics, Montanuniversität Leoben, Austria

Ivanov Yu. P.[1,4], Ketov S.V.[1], Louzguine-Luzgin D.V.[2], Eckert J.[1,3], Greer A.L.[4]

1Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Jahnstraße 12, A-8700 Leoben, Austria, 2WPI Advanced Institute for Materials Research, Tohoku University, Sendai, 980-8577, Japan, 3Department Materials Physics, Montanuniversität Leoben, Jahnstraße 12, A-8700 Leoben, Austria, 4Department of Materials Science & Metallurgy, University of Cambridge, Cambridge CB3 0FS, UK

Bulk metallic glasses(BMG) are materials of interest for both functional and structural applications [1]. However, the lack of macroscopic ductility limits their application fields. It was established that metallic glasses with the structure closer to the liquid exhibit higher plastic strain than relaxed ones..Nowadays many methods for enhancement of structure of metallic glasses are known. Recently, a new method for metallic glass rejuvenation was found [2].Heterogeneous structure of metallic glass on nano scale results in heterogeneous thermal expansion or contraction upon temperature changes which induce local stresses high enough for anelastic strains to occur. Cryothermal cycling appears to be an attractive and simple method to rejuvenate metallic glasses. It is non-destructive, avoids macroscopic deformation of the sample, can be applied to samples of any shape and size, is uniform and changes the whole sample etc. Method appears also to be usefull for wide variety of materials. However, the applicability boundaries of the method for metallic glasses are still unknown. Here we report the High Resolution Transmission Electron Microscopy (HRTEM) study of the chemical and crystal structure changes in BMGs of Zr60Cu20Fe10Al10, Zr60Cu20Co10Al10, and Zr60Cu20Ni10Al10 after cryothermal cycling treatment.
TEM studies were carried out with a Titan G2 60 - 300 (FEI, Netherlands), equipped with X-FEG and Cs image corrector. The estimated spatial resolution is about 0.08 nm at 300 kV. A dual beam system (FEI Helios 450) was employed to fabricate the cross-sections of the as-cast samples as well as those cycled in Liquid Nitrogen. The EELS and EDX experiments were performed with a probe size of around 0.2 and 0.5 nm accordingly. The structural characterization of the amorphous materials was performed by evaluation of the atomic radial distribution function (RDF) which was directly computed from the fast Fourier transform (FFT) patterns of the HRTEM images. The results obtained from conventional Selected Area Electron Diffraction show a similar behavior to ones extracted from FFT.
It was established that cryothermal cycling is a universal method for triggering structural changes in pure metallic glasses. BMGs of chosen compositions behave differently under the cryothermal cycling treatment. Cryothermal cycling treatment can work as a method for rejuvenation as well as relaxation of metallic glasses leading to increase or deterioration of mechanical properties The bond structure in metallic glasses appears to have a significant influence on the treatment outcome as well as the maximal achievable plastic strain. Analysis of HRTEM images revealed that after cryothermall cycling all RDF peaks of the Fe containing sample are shifted towards higher values which mean that the average atomic bond distances increased. This indicates the increase of general volume and rejuvenation takes place in Fe containing glass. Interestingly, after cryothermal cycling treatment copper tends to form islands on top of the native oxide. Affinity of the noble metals for oxygen is very low and bond strength is not high. Copper also tends to segregate from iron during cryothermal cycling. This does not happen in the glass containing Ni.
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