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Journal of Alloys and Compounds 434–435 (2007) 68–70

Journal of ALLOYS AND COMPOUNDS

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# Influence of air and vacuum environment on fatigue behavior of Zr-based bulk metallic glasses

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## Abstract

High-cycle fatigue (HCF) experiments in air and vacuum at room temperature were conducted on zirconium (Zr)-based bulk-metallic glasses (BMGs): $Zr_{50}Cu_{40}Al_{10}$, $Zr_{50}Cu_{30}Al_{10}Ni_{10}$, and $Zr_{50}Cu_{37}Al_{10}Pd_3$ in atomic percent. The fatigue-endurance limit of $Zr_{50}Cu_{37}Al_{10}Pd_3$ was found to be significantly greater than those of $Zr_{50}Cu_{40}Al_{10}$ and $Zr_{50}Cu_{30}Al_{10}Ni_{10}$, which indicates that the inclusions of Pd and the resulting nano structures improve the fatigue resistances of the Zr-based BMGs. The fatigue lives in vacuum and air were generally found to be comparable.

*Keywords:* Amorphous materials; Casting; Scanning and transmission electron microscopy

## 1. Introduction

After the development of bulk-metallic glasses (BMGs) during 1990s, several novel multicomponent BMGs such as Zr-based BMGs have been discovered and which exhibit exceptional glass-forming abilities [1–3]. BMGs have broken through the limitation of the specimen geometry. Many unique properties of BMGs such as high strengths (>2 GPa), low coefficients of friction, high wear resistances, low shrinkage during cooling, and almost perfect as-cast surfaces [4,5] have attracted scientific and engineering attentions. The mechanical behavior of metallic glasses is being widely studied but the real nature of the deformation mechanisms in these amorphous alloys still remain unclear [6]. Since BMGs can be applied as potential structural materials, the fatigue behavior of BMG is a very important aspect for engineering applications. However, little attention has been paid to studying the fatigue characteristics of BMGs, especially in controlled environments. And the fatigue behavior is poorly understood. In this paper, the ladle-hearth type arc-melt tilt-casting machine was used to fabricate inclusion-free BMGs samples of $Zr_{50}Cu_{40}Al_{10}$, $Zr_{50}Cu_{30}Al_{10}Ni_{10}$, and $Zr_{50}Cu_{37}Al_{10}Pd_3$ [in atomic percent (at.%)]. High-cycle fatigue behavior and the stress versus fatigue-cycle life (S/N) curves were investigated.

## 2. Experimental procedures

$Zr_{50}Cu_{40}Al_{10}$, $Zr_{50}Cu_{30}Al_{10}Ni_{10}$ and $Zr_{50}Cu_{37}Al_{10}Pd_3$ were fabricated by the ladle-hearth type arc-melt tilt-casting technique. Master-alloy ingots were prepared by arc-melting mixtures of pure Zr, Cu, Al, Pd, and Ni metals in an argon atmosphere. The cast rod samples of 8 mm in diameter and 60 mm in length were machined into the button-head fatigue specimens [7-9]. The samples were, then, polished to minimize surface effects. A computer-controlled material test system (MTS) servohydraulic testing machine was employed for fatigue experiments. Samples were tested in air and vacuum at room temperature with $R$ ratio ($R = \sigma_{min}/\sigma_{max}$, where $\sigma_{min}$ and $\sigma_{max}$ are the applied minimum and maximum stresses, respectively) of 0.1 under a load-control mode, using a sinusoidal waveform at a frequency of 10 Hz.

Thermography detection was conducted, using an Indigo Phoenix thermographic-infrared (IR) imaging system with a 320 × 256 pixels focal-plane-array InSb detector that is sensitive to a radiation wavelength of 3–5 μm. The temperature sensitivity is 0.015 °C at 23 °C. The IR camera was used at a speed of 300 Hz with a 128 × 128 pixel in the current study. The fracture surfaces of selected specimens were examined, using a Leo 1526 scanning-electron microscope to study fatigue and fracture mechanisms.

## 3. Results and discussion

Fig. 1 presents the fatigue results for $Zr_{50}Cu_{40}Al_{10}$, $Zr_{50}Cu_{30}Al_{10}Ni_{10}$ and $Zr_{50}Cu_{37}Al_{10}Pd_3$ in air and vacuum. The

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doi:10.1016/j.jallcom.2006.08.291

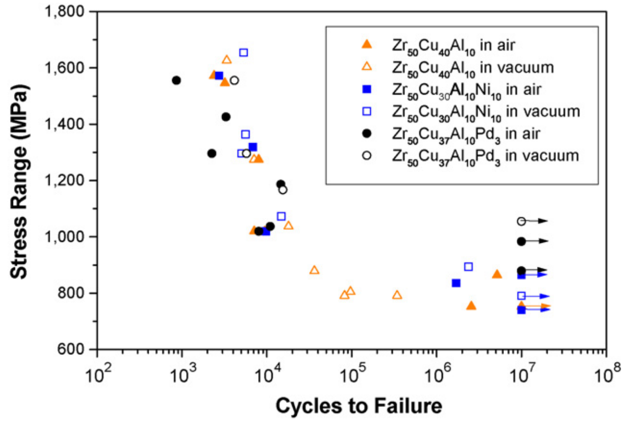

Fig. 1. Stress-range/fatigue-life data of notched specimens tested in air and vacuum with $R = 0.1$ and a frequency of 10 Hz.

stress-range values reflect the stress-concentration factor ($K_t$) of 1.55 [10,11] at the notched section for this kind of specimen.

The fatigue lifetime of these BMGs shows no difference at high stress levels. Nevertheless the fatigue-endurance limits ($\sigma_L$), based on the applied stress range, for $Zr_{50}Cu_{40}Al_{10}$, $Zr_{50}Cu_{30}Al_{10}Ni_{10}$, and $Zr_{50}Cu_{37}Al_{10}Pd_3$ tested in air, were approximately 752, 865 and 983 MPa, respectively. Transmission-electron microscopy (TEM) results (Fig. 2) show that $Zr_{50}Cu_{37}Al_{10}Pd_3$ includes some nano structure that could result in its higher fatigue-endurance limit, because nano structure might block shear bands and crack propagation. Results indicate that the fatigue-endurance limits of Zr-based BMGs can be improved by changing the chemical compositions.

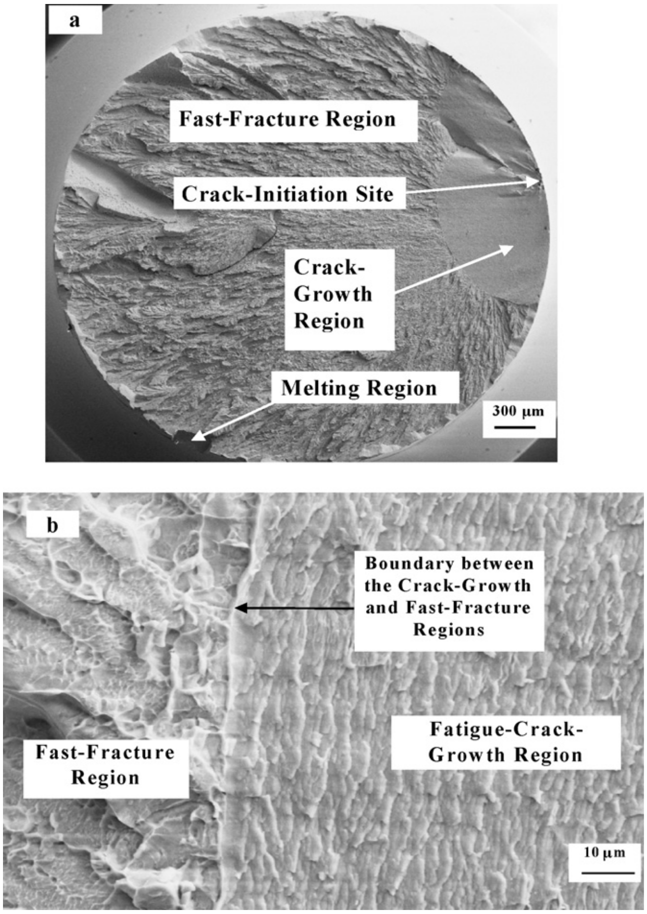

Fig. 3. (a) Overall fatigue fractography of the $Zr_{50}Cu_{37}Al_{10}Pd_3$ specimen tested at $\sigma_{max} = 1152$ MPa; (b) fatigue-crack-growth and fast-fracture region.

Fig. 3 shows the fatigue-fracture surface of the $Zr_{50}Cu_{37}Al_{10}Pd_3$ specimen tested at $\sigma_{max} = 1152$ MPa in air. The whole fatigue fracture surface consisted of four main regions: the fatigue-crack-initiation, crack-propagation, fast-fracture, and apparent-melting areas (Fig. 3(a)). The fracture surface was basically perpendicular to the loading direction. The propagation region was of a thumb–nail shape and relatively flat and showed the fatigue striation (Fig. 3(b)). The final-fast-fracture region was very rough and occupied most of the fracture surface (Fig. 3(b)). Moreover, the final fracture surface exhibits a clear vein-like structure, which is identical to the tensile fracture surface of most of BMGs [12]. Along the propagation path of the fatigue crack, however, there is no vein-like structure. This trend demonstrates that the melting phenomenon of BMGs does not occur at the tip of the fatigue crack during the crack-propagation stage. In fact, it means that the released elastic energy due to crack propagation is too low to melt the metallic glass locally. This result is consistent with observations reported in the references [6,13]. However, in the melting region, the vein-like structure and droplets appear [8]. The distinct melting marks, droplets, and vein patterns were observed in the melting region at a high magnification by SEM.

A sparking phenomenon [7,8] was found via an IR-camera system, when the $Zr_{50}Cu_{30}Al_{10}Ni_{10}$ specimen was cyclically

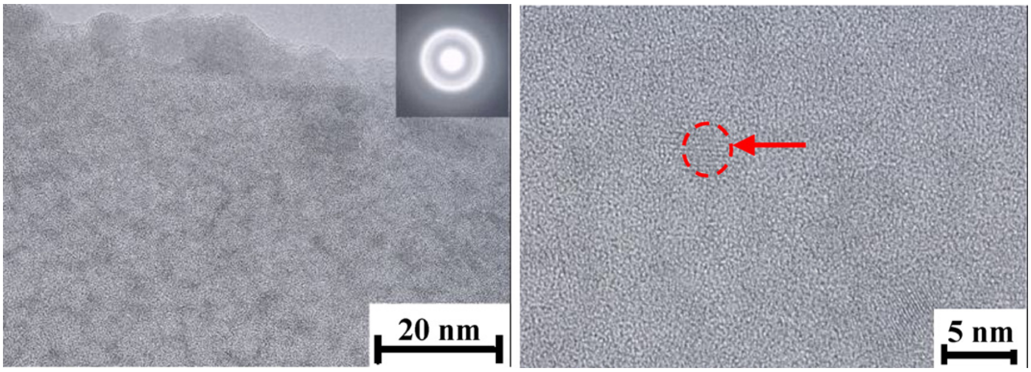

Fig. 2. TEM micrograph of the microstructure of $Zr_{50}Cu_{37}Al_{10}Pd_3$. The arrow indicates a nano structure.

loaded at higher levels ($\sigma_{max} > 1500$ MPa) in air. Though the sparking phenomenon was not detected for $Zr_{50}Cu_{40}Al_{10}$ and $Zr_{50}Cu_{37}Al_{10}Pd_3$ in air and vacuum and $Zr_{50}Cu_{30}Al_{10}Ni_{10}$ in vacuum, the fracture sections were very bright at the moment of the specimen fracture, which meant that the temperature was very high. This trend is identical to the melting phenomena that were observed on the fracture surfaces of these BMGs by SEM.

The fatigue behavior of crystalline materials has been well studied, and many theories, such as grain-boundary deformation and dislocation kinetics, can be used to understand their fatigue-damage mechanisms. However, since BMGs are amorphous, no grain boundaries and dislocations exist. Their fatigue-crack initiation and growth mechanisms could be different from the crystalline alloys. The basic fatigue-damage mechanism of BMGs is still unknown. However, it is possible that shear bands form during fatigue. And shear bands could result in the formation of shear steps on the outer surface of BMGs [7,12]. Due to the stress concentration, microcracks could form from the shear steps or shear bands on the outer surface. This trend could be the reason why the fatigue crack initiated from the outer surface.

## 4. Conclusions

The fatigue-endurance limit of $Zr_{50}Cu_{37}Al_{10}Pd_3$ is greater than $Zr_{50}Cu_{40}Al_{10}$ and $Zr_{50}Cu_{30}Al_{10}Ni_{10}$. However, there is no apparent difference in fatigue lives at higher stress levels. Vacuum has no distinct effect on the fatigue lives of BMGs. The fatigue-damage mechanism of BMGs needs further study.

## Acknowledgements

We would like to acknowledge the financial support of the National Science Foundation, the Division of Design, Manufacture, and Industrial Innovation, under Grant No. DMI-9724476, the Combined Research-Curriculum Development (CRCD) Programs, under EEC-9527527 and EEC-0203415, the Integrative Graduate Education and Research Training (IGERT) Program, under DGE-9987548, and the International Materials Institutes (IMI), under DMR-0231320, to the University of Tennessee, Knoxville, with Dr. D. Durham, Ms. M. Poats, C. Dr. J. Van Hartesveldt, Dr. D. Dutta, Dr. W. Jennings, Dr. L. Goldberg, and Dr. C. Huber as Program Directors, respectively.

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