Fracture toughness and fatigue-crack propagation in a Zr–Ti–Ni–Cu–Be bulk metallic glass

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The recent development of metallic alloy systems which can be processed with an amorphous structure over large dimensions, specifically to form metallic glasses at low cooling rates (~10 K/s), has permitted novel measurements of important mechanical properties. These include, for example, fatigue-crack growth and fracture toughness behavior, representing the conditions governing the subcritical and critical propagation of cracks in these structures. In the present study, bulk plates of a $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ alloy, machined into 7 mm wide, 38 mm thick compact-tension specimens and fatigue precracked following standard procedures, revealed fracture toughnesses in the fully amorphous structure of $K_{Ic}\sim 55$ MPa$\sqrt{}$m, i.e., comparable with that of a high-strength steel or aluminum alloy. However, partial and full crystallization, e.g., following thermal exposure at 633 K or more, was found to result in a drastic reduction in fracture toughness to ~1 MPa$\sqrt{}$m, i.e., comparable with silica glass. The fully amorphous alloy was also found to be susceptible to fatigue-crack growth under cyclic loading, with growth-rate properties comparable to that of ductile crystalline metallic alloys, such as high-strength steels or aluminum alloys; no such fatigue was seen in the partially or fully crystallized alloys which behaved like very brittle ceramics. Possible micromechanical mechanisms for such behavior are discussed. © *1997 American Institute of Physics.* [S0003-6951(97)03730-3]

The recent development of alloys for the processing of metallic glasses in bulk form[1] permits the measurement of important mechanical properties in amorphous metals, in particular the fatigue and fracture characteristics. Previous work on metallic glasses has invariably been confined to very thin ribbons or wires, thus making measurements difficult.[2,3] Few results are thus available on the toughness and cyclic crack growth properties in these alloys. Accordingly, the objective of the current study is to quantify the fracture toughness and fatigue-crack growth properties of a recently developed bulk metallic glass alloy, $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ (nominal composition in at. %).[4–6] In this note, we present results characterizing the hardness, fracture toughness, and fatigue resistance of this alloy; in addition, we compare properties in the fully amorphous structure with that of partially and fully crystallized structures in the same alloy.

As-received plates of the alloy, processed using methods described elsewhere,[1] were found to be *fully amorphous*. By heat treating at 633 K for 12 h *in vacuo*, just above the glass-transition temperature ($T_g\sim 625$ K), a *partially crystallized* structure was formed. This consists of the amorphous matrix containing finely dispersed 3–5 nm nanocrystallites of a Cu-rich, Ti-rich fcc phase with an average spacing of ~20 nm between nanocrystals.[4,5] The volume fraction of the crystalline fcc phase is estimated from x ray and TEM data to be less than 5% of the sample. By heat treating at 723 K for 24 h *in vacuo*, a third *fully crystallized* multiphase microstructure was obtained, containing a Laves Phase with the hcp ''$MgZn_2$-type'' structure,[5] a phase with the ''Al_2Cu-type'' structure, and at least one additional unidentified phase. The degree of crystallinity for each structure is indicated by the x-ray diffraction data in Fig. 1. Vickers hardness values marginally increased from 5.37 (±0.08) GPa in the glass to 6.04 (±0.04)and 6.35 (±0.10) GPa, respectively, in the partially and fully crystallized structures.

Fracture toughness and fatigue-crack growth rate properties were determined in a controlled room-air environment (22 °C, 45% relative humidity) on 7-mm thick, 38-mm wide compact-tension $C(T)$ specimens, machined from the bulk plates. Specimens were cycled under stress intensity (K) control, with a test frequency of 25 Hz (sinusoidal waveform) at a constant load ratio (ratio of minimum to maximum load) of $R=0.1$, using computer controlled, servohydraulic mechanical testing machines, in general accordance with ASTM standard E647. To obtain a wide spectrum of growth rates, samples were first cycled with a decreasing stress-intensity range (at a normalized K gradient of 0.2 mm^{-1}) until measured growth rates were less than 10^{-10} m/cycle; the value of the stress-intensity range at this point was used to operationally define the fatigue threshold stress intensity (ΔK_{TH}), below which long cracks are essentially dormant. After threshold determination, specimens were cycled under increasing ΔK conditions with the same K gradient in order to determine growth rates up to $\sim 10^{-8}$ m/cycle.

Crack initiation was facilitated using a half-chevron-shaped starter notch; prior to data collection, samples were fatigue precracked for several millimeters beyond this notch. Thereafter, crack lengths were continuously monitored using unloading elastic-compliance measurements with a 350 Ω

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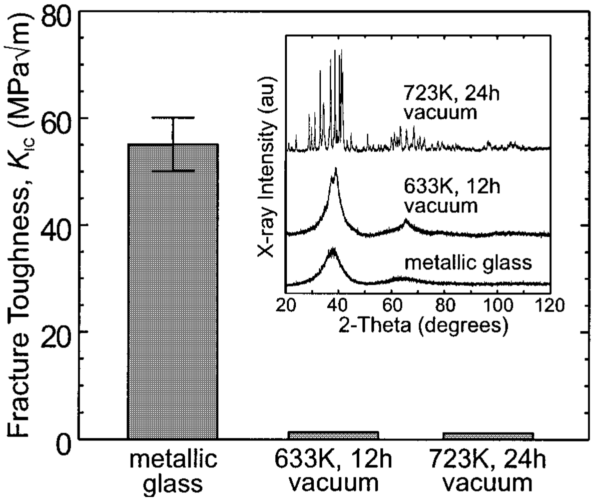

FIG. 1. While the fracture toughness of the amorphous alloy was ~55 MPa√m, thermal exposure resulting in partial or full crystallization leads to a 50-fold reduction in K_{Ic} values to ~1.21 and 1.04 MPa√m, respectively. X-ray diffraction data corresponding to each of these microstructures are included in the inset.

strain gauge attached to the back face of the specimen; crack lengths were also checked periodically using a traveling microscope. Optical and compliance measurements were always found to be within 2%. Data are presented in terms of the growth rate per cycle, da/dN, as a function of the alternating stress intensity, $\Delta K(=K_{\max}-K_{\min})$, the latter being computed using standard linear-elastic handbook solutions.

Following growth-rate measurements, fracture toughness, K_{Ic}, values were determined by monotonically loading the fatigue precracked specimens to failure; procedures were in general accordance with ASTM standard E399. However, fatigue cracking was unstable in the partially and fully crystalline structures due to their extreme brittleness; correspondingly, here toughness values were obtained using Vickers indentation methods, with measurements averaged from at least five indents under an indentation load of 49 N.

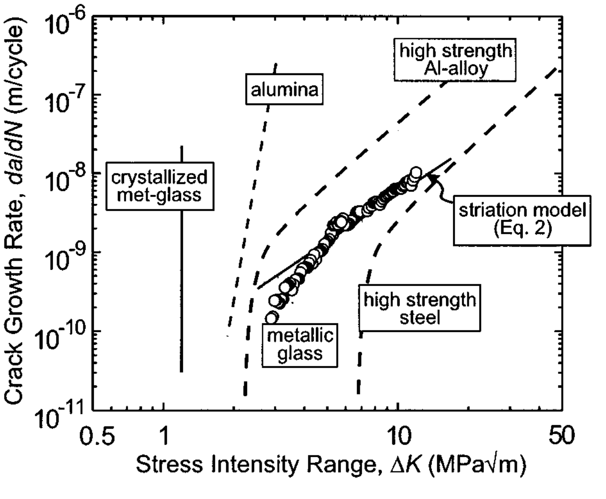

FIG. 2. Fatigue-crack growth rate, da/dN, is plotted as a function of stress-intensity range, ΔK, for the amorphous and crystallized $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ alloy. Data are compared to a range of engineering materials, including fine-grained alumina, 2.25Cr–1Mo steel and 2124 aluminum alloy. Growth rates in the amorphous structure were found to be comparable to that in many ductile crystalline metals such as high-strength aluminum alloys and steels.

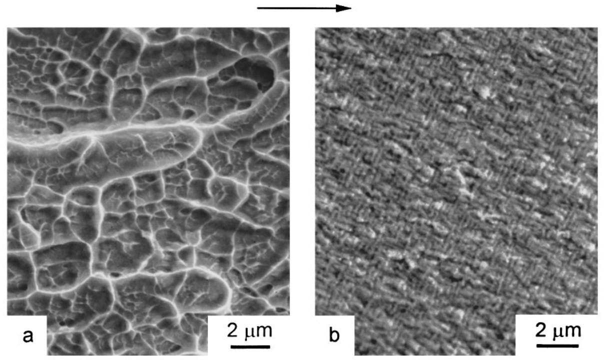

FIG. 3. Scanning electron micrographs of fracture surfaces developed during (a) overload fracture and (b) fatigue-crack growth. Arrow represents direction of crack extension.

Results indicated that the fracture toughness of the amorphous alloy was a remarkable 55 (±5.0) MPa√m; however, thermal exposure resulting in partial or full crystallization led to approximately a 50-fold reduction in K_{Ic} values to 1.21(±0.04) and 1.04 (±0.05) MPa√m, respectively (Fig. 1). Whereas the toughness of the metallic glass is comparable to that of a typical (crystalline) aluminum or high-strength steel alloy, the toughness after partial or full crystallization is comparable to that of silica glass or very brittle ceramics.

This drastic embrittlement upon crystallization meant that fatigue cracking was not observed in these structures; any attempt to grow stable cracks from the machined notches led to catastrophic failure of the sample. Such behavior is typical of very brittle (untoughened) ceramics, and is consistent with the precipitous drop in toughness on crystallization. However, stable fatigue-crack growth behavior was characterized in the amorphous structure under cyclic loading. Results in the form of growth rates as a function of ΔK are plotted in Fig. 2 and compared to results for a range of ceramic and metallic materials, including polycrystalline alumina, high-strength steel, and high-strength aluminum alloys; these data pertain to plane-strain conditions, unlike all previous data for metallic glasses which were measured on very thin sheets.[7,8] It is apparent that cyclic crack growth rates in the amorphous metal lie between that of high-strength steel and aluminum alloys. When regression fit to a simple Paris power-law equation:[9]

$$da/dN=C\Delta K^{m}, \tag{1}$$

the crack-growth scaling constants are found to be $C=1.46\times10^{-11}$ and $m=2.7$ (units: m/cycle, MPa√m). The exponent m is typical of ductile metallic alloys, which usually lie between 2 and 4 over this regime of growth rates; this is in contrast to brittle materials like alumina where m is typically 20 or higher.[10] The threshold for fatigue-crack growth in the metallic glass, ΔK_{TH}, is ~3 MPa√m; this is again comparable to many aluminum and steel alloys, although it does not appear to be as distinct as in the polycrystalline metals.

The corresponding fracture surfaces display a morphology quite distinct from anything seen in crystalline metals, with the fracture surface roughness increasing markedly with increasing crack velocity. Surfaces vary from a rough morphology exhibiting ridgelike features in the fast-fracture region [Fig. 3(a)] to a mirrorlike surface in the near-threshold fatigue region. However, closer examination of the fatigue surfaces, particularly in the higher growth-rate region, reveal clear evidence of classic fatigue striations [Fig. 3(b)], i.e., ''beach markings'' parallel to the crack front representing the cycle-by-cycle advance of the crack. Indeed, such striations have been reported in the past for the fatigue of metallic glasses in thin sheet.[7,8]

The mechanism of striation formation in amorphous alloys is as yet unclear. However, in ductile crystalline metals, some degree of irreversible slip at the crack tip is required alternately to blunt the crack on the loading cycle and subsequently resharpen it on unloading,[11] and tensile experiments in this[6] and other amorphous metals[2,3] indicate that slip bands readily form. Models for striation formation[11] indicate that growth rates should scale with the range of crack-tip opening displacement, $\Delta\delta$, which using simple continuum mechanics arguments is given by:[11]

$$\Delta\delta=\beta\frac{\Delta K^2}{\sigma_Y E'}, \tag{2}$$

where σ_Y is the flow stress, $E'=E$ (Young's modulus) in plane stress and $E/(1-\nu^2)$ in plane strain (ν is Poisson's ratio), and β is a scaling constant ($\sim$0.01–0.1 for mode I crack growth) which is a function of the degree of slip reversibility and elastic-plastic properties of the material. The fact that Eq. (2) provides a reasonable description of the experimentally measured growth rates for the metallic glass, with $m\sim 2.7$ and $\beta\sim 0.01$ (Fig. 2), together with the presence of fatigue striations on the fracture surfaces [Fig. 3(b)], strongly suggests a mechanism for crack advance involving repetitive blunting and resharpening, i.e., a mechanism similar to that commonly observed in crystalline metals.

In conclusion, measurements of fracture properties on a *bulk* metallic glass, $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$, reveal a high fracture toughness of $\sim$55 MPa$\sqrt{m}$ in the amorphous alloy, which is drastically reduced by a factor of over 50 upon crystallization, i.e., from levels comparable to high-strength steel and aluminum alloys when fully amorphous to levels comparable to brittle ceramics when partially or fully crystallized. Although no stable cracking was seen in the partially or fully crystalline structures, the amorphous alloy was susceptible to fatigue-crack growth under cyclic loads; behavior in terms of fractography and the dependency of growth rates on the stress-intensity range is similar to traditional crystalline metals.

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