Scripta Materialia 141 (2017) 58–61

Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.elsevier.com/locate/scriptamat

Regular article

A sequential pre-cracking procedure to measure the mode-I fracture toughness of ultra pure bulk metallic glasses

C. Bernard[a], V. Keryvin[a,*], V. Doquet[b], S. Hin[a], Y. Yokoyama[c]

[a] Univ. Bretagne Sud, FRE CNRS 3744, IRDL, Lorient F-56321, France
[b] Laboratoire de Mécanique des Solides, Ecole Polytechnique, UMR CNRS 7649, Univ. Paris Saclay Palaiseau, France
[c] Laboratory for Advanced Materials, Institute for Material Research, Tohoku University, Katahira 2-1-1, Aoba-ku, Sendai City, Miyagi Prefecture 980-8577, Japan

ARTICLE INFO

Article history:
Received 20 July 2017
Accepted 23 July 2017
Available online xxxx

Keywords:
Fracture
Toughness
Bulk metallic glasses
Protocol

ABSTRACT

A dedicated fatigue pre-cracking method, performed successively under mode II and mode I, has been set up to initiate and propagate a crack close to the notch plane, which is particularly difficult to achieve using pure mode I loading on some ultra pure bulk metallic glasses, free from oxygen contamination. This method, was applied to a pure $Zr_{55}Cu_{30}Al_{10}Ni_5$ (at.%) amorphous alloy, and allowed successful pre-cracking and reliable measurement of the apparent mode I fracture toughness.

Bulk metallic glasses (BMG) are advanced materials exhibiting high mechanical properties, namely strength, yield strain, stored elastic energy and hardness [1]. They are therefore considered for engineering applications. If many studies have been devoted to these mechanical properties, relatively few papers concerning fracture toughness have been published until recently [2]. Known *plane-strain* fracture toughness (K_{IC}) values range from ~3 MPa$\sqrt{m}$ [3], comparable to those of ceramics, for Fe-based compositions, to values above 30 MPa$\sqrt{m}$ [4], comparable to those of some crystalline alloys, for Zr-based glasses.

A first requirement for assessing fracture toughness is to create an atomically sharp notch, that is a crack, usually by fatigue pre-cracking. Indeed, complications arise from the high dependence of the measured apparent toughness to the notch root radius when the specimens are not pre-cracked. As reported by Lewandowski et al. [5] or by Fujita et al. [6], notch toughness (K_{IQ}^N), even for very small root radii, may be largely higher than the real K_{IC} that requires a perfectly sharp tip.

Keryvin et al. [7] indicated that creating a straight-through-the-thickness crack may be a tricky task in Zr-based BMG samples containing very little oxygen (~300 ppm), while it was straightforward in samples containing few crystalline defects. The purpose of this paper is therefore to propose a dedicated experimental procedure to introduce a sharp crack ahead of the notch tip in ultra-pure samples (that is: free from oxygen contamination), allowing a proper measurement of their mode I fracture toughness.

Several other studies on Zr-based BMG [8] also reported that the crack may detour from the straight-through-the-thickness mode I path to adopt a tilted mixed crack growth mode. The systematic deviation, in Ref. [7], of cracks during pre-cracking at an angle close to 45° vis-à-vis the notch plane, observed on the faces, as well as the observation of helicoidal-like inner cracks, suggested that cracks follow shear bands [9] that emanate at these particular angles in flawless bulk metallic glasses [8]. An example of such a situation in another material may be found in amorphous polymers (polycarbonate) in Ref. [10, pp. 486–488] . Since, in mode II, shear bands develop in the notch plane ahead of the tip, pre-cracking in (shear) mode II could alleviate these difficulties. Indeed, Tandaiya et al. [11] analysed the notch tip plasticity in metallic glasses both experimentally and by finite-element simulations under mixed mode loadings with good agreement in terms of plastic zone size and morphology or shear bands activity. They found that the simulated shear bands are straight and extend over a long distance ahead of the notch tip under mode II dominant loading. While Narayan et al. [12] and Flores et al. [13] underlined the high variability of measured toughness for tough BMGs specimens pre-cracked in mode I, Narayan et al. [12] showed that mode II loading leads to a much better reproducibility, due to a lower dispersion in the direction of the shear bands induced

* Corresponding author.
E-mail address: vincent.keryvin@univ-ubs.fr (V. Keryvin).

http://dx.doi.org/10.1016/j.scriptamat.2017.07.027
1359-6462/

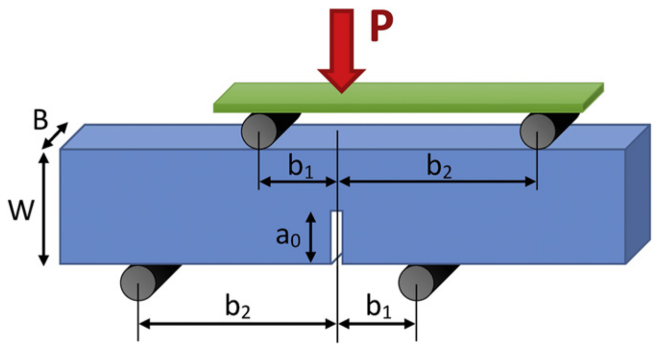

Fig. 1. Asymmetric four-point bending test used for pre-cracking in mode II. A pure shear force is induced along the notch plane.

at the notch tip [9,11]. This advantageous phenomenon may allow for an improvement in the pre-cracking procedure of defect-free glasses. In what follows, we propose a sequential pre-cracking procedure first in mode II then in mode I allowing a correct assessment of the mode I fracture toughness of ultra-pure Zr-based BMG.

The specimens were made from a quaternary eutectic composition $Zr_{55}Cu_{30}Al_{10}Ni_5$ (at.%) with a super low oxygen content Zr (<50 ppm). Oxygen concentration of the bulk amorphous alloys was measured by infrared absorption method after fusion in helium gas. Samples contain about 300 ppm of oxygen. No crystalline defects were spotted. The rods produced had the dimensions $\phi 8 \times 60$ mm^2 and were machined by EDM (electron discharge machining) into single edge-notched bending specimens (SENB, $a_0 = 2$ mm, B = 4 mm, W = 5.5 mm, cf. Fig. 1) with a straight-through-the-thickness notch. A sharper notch (with a root radius of about 30 µm) was created using a 0.2 mm-thick abrasive disk, and finally with a razor-blade covered with 1 µm diamond paste. The elastic properties of these samples were measured by ultrasonic echography, giving E ~ 85 GPa and $\nu \sim 0.365$, as described elsewhere [14].

All samples were first fatigue pre-cracked in mode II (in-plane shear mode; frequency f = 5 Hz, with a load ratio $R = P_{\min}/P_{\max} = 0.15$) by means of an asymmetric four-point bending apparatus (see Fig. 1, $b_1 = 5$ mm, $b_2 = 10$ mm) that induces a pure shear force and no bending moment along the notch plane. The stress intensity factor in mode II is given as a function of crack length a, applied force P and geometry of the SENB specimen:

$$K_{II}\,(a,P) = \frac{(b_2 - b_1)\,P}{(b_2 + b_1)\,B\sqrt{W}}\,\frac{\left(\frac{a}{W}\right)^{\frac{3}{2}}}{\left(1 - \frac{a}{W}\right)^{\frac{1}{2}}}\,F_{II}\left(\frac{a}{W}\right) \tag{1}$$

The polynomial function F_{II} is described in Ref. [15]. Initial ΔK_{II} values[1] were ~8 MPa$\sqrt{\text{m}}$. The notch region was monitored by optical microscopy. After several thousands of cycles, straight cracks appeared on both faces of the specimens (see Fig. 2). Let us note that pre-cracking in mode I was sometimes reported to be successful as well at this stage; yet the inspection of fracture surfaces revealed that the crack did not develop straight through the thickness [7]. Therefore the development of straight cracks along the faces is a necessary but not sufficient condition of successful pre-cracking. Final $K_{II}^{\max}$ values were ~30 MPa$\sqrt{\text{m}}$, well below the reported values of fracture toughness in mode II for Zr-based alloys [16], and final a/W values ~0.5. The final mode II plastic zone size, r_p^{II}, in which residual stresses are present, can be estimated [17] by:

$$r_p^{II} = \frac{3}{2\pi}\left(\frac{K_{II}^{\max}}{\sigma_Y}\right)^2 \tag{2}$$

where σ_Y (~1600 MPa [18]) is the tensile yield strength.

Such residual stresses might bias the results, should the fracture toughness had been directly measured after mode II pre-cracking. To avoid such history effect, the samples were *subsequently* cyclically loaded in mode I by classical three-point bending (R = 0.15, f = 5 Hz) until the crack propagated well beyond the mode II plastic zone (r_p^{II} ~75 µm). The mode I stress intensity factors were computed as a function of the crack length a, applied force P and geometry of the SENB specimen (span length $S = 20$ mm) by:

$$K_I\,(a,P) = \frac{3\,P\,L\sqrt{\pi\,a}}{B\,W^2}\,\frac{F_I'\left(\frac{a}{W},\frac{L}{W}\right)}{\left(1 - \frac{a}{W}\right)^{\frac{3}{2}}} \quad \text{with} \quad S = 2L \tag{3}$$

The values of F_I' are tabulated in Ref. [19]. Final $K_I^{\max}$ values were ~25 MPa$\sqrt{\text{m}}$ and final a/W values were ~0.6.

All samples were eventually monotonously loaded in three-point bending up to failure at a displacement rate of 0.05 mm/min. All curves were of type I (ASTM E399 [20]) as shown in Fig. 3. The force used to calculate the fracture toughness, P_Q, was defined by the intersection between the load-displacement curve and the 95% secant line. For all the tests, the ratio $P_{\max}$ / P_Q did not exceed 1.10. The critical crack length of all the broken specimens was obtained from Scanning Electron Microscopy (SEM) images of fracture surfaces, such as those presented in Fig. 4. A tilted view of the fracture surface is provided to appreciate the three dimensional propagation of the crack, that should stay, according to the ASTM standards, in the notch plane. It permits to check that i) propagation occurred along the notch plane; ii) the crack front was reasonably straight and parallel to the notch root. Since the crack front, highlighted by a white line, was not perfectly straight, the crack length after pre-cracking, a_c, was taken from the SEM images as the mean of five values measured at five equidistant points through the thickness. Conditional fracture toughness, K_{IQ}, was then calculated using Eq. (3). Values were 85 ± 2 MPa$\sqrt{\text{m}}$.

Narayan et al. [12] have already observed on a Zr-based BMG that mode II loading tends to generate numerous shear bands close to the notch root and mainly oriented along the notch plane. Afterwards, inside the dominant shear band, a straight crack initiates, then propagates steadily and remains close to the notch plane, as it can also be observed on the specimen in Fig. 2. Conversely, the shear bands appearing under the mode I loading are more widely spread around the notch tip and without any favoured direction. As a consequence, the probability that the crack, emerging from the dominant shear band, is close to the notch plane is very low. This is in accordance with the numerical simulations of Tandaiya et al. [9]. However, if mode I fatigue crack growth follows the mode II pre-cracking stage, it just extends further the coplanar crack initiated by mode II. This is due to the singular crack tip stress field that largely exceeds the unfavourable notch root stress concentration — provided that the crack has already propagated far enough from the notch to no longer be perturbed by it. However, a crack path characterization based only on observations of the sample faces is not sufficient to validate a test. A three dimensional observation of the crack path from the fracture surface, as allowed by the tilted view of Fig. 4, is necessary to confirm that the measured fracture toughness really corresponds to mode I loading.

This preparation method uses the mode II loading advantages that were underlined in [9], i.e. the tendency to generate straight shear

[1] $\Delta K_{II}^0 = K_{II}^{\max}(a_0, P_{\max}) - K_{II}^{\min}(a_0, P_{\min})$.