Effects of Overload and Frequency on Fatigue Crack Propagation in Nanocrystalline Zr-Based Bulk Metallic Glass *

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A nanocrystalline (NC) bulk glass $Zr_{55}Al_{10}Cu_{30}Ni_5$ (at%) containing nano-scale crystals embedded uniformly in a glassy matrix has both high tensile strength of 1.7 GPa and high ductility. The new alloy is therefore expected to have practical applications in machines and structures. The influences of frequency and overload on fatigue crack propagation behavior of the NC bulk glass were examined. The fatigue crack propagation rate da/dn less than 3×10^{-5} mm/cycle was independent of frequency in the frequency range of 0.1 to 50 Hz at the stress ratio of 0.1 under sine and triangular waves. When the overload ratio (overload/baseline load) was large, a complete crack arrest occurred and the ΔK value just before a crack regrowth was three times larger than the threshold stress intensity factor range ΔK_{th} . The reason for the crack arrest was not explained by the crack closure effect. The overloading induced the kinking and branching of the crack. The stress reduction near the crack tip due to the kinking and branching of the crack and the crack closure effect gave an appropriate explanation for the complete crack arrest and the larger threshold stress intensity factor range. When the overload ratio was small, a temporary crack arrest occurred and the kink and branching of cracks occurred intermittently at the crack front.

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1. Introduction

Recently, nanocrystalline (NC) bulk glassy alloys containing nano-scale crystalline particles embedded uniformly in a glassy matrix have been synthesized in some Zr-based alloy systems by a squeeze casting method.¹⁾ The tensile strength and fracture toughness of the NC glassy alloys are larger than those of the single-phase bulk glassy alloys with the same composition.²⁾ Consequently, the NC glassy alloys have attracted increasing interest as a candidate of practical high-strength structural materials (tensile strength $\sigma_B = 1.7$ GPa).

It is well known that most of fractures in the strength members of real machines and structures are due to fatigue, and the most part of the fatigue life is spent for the crack propagation when a crack-like defect exists.

The fatigue behavior in Zr-based bulk metallic glasses, which have higher toughness than other metal-based alloys, has been studied,³⁻⁵⁾ and the major results are as follows; (1) a fatigue limit σ_w exists, (2) the fatigue crack propagation rate da/dn is approximately proportional to ΔK^2 , where a is the crack length, n is the number of cycles and ΔK is the stress intensity factor range, and (3) striation-like patterns are observed on the fracture surface.

In the Zr-based NC bulk glassy alloys, the present authors have examined fatigue crack growth properties, and reported the following results.^{6,7)} (1) The da/dn is approximately proportional to ΔK^2 , (2) the threshold stress intensity factor range ΔK_{th} is smaller than those of crystalline metallic materials, (3) the smaller ΔK_{th} in the NC glassy alloys is attributed to the difficulty in inducing crack closure in comparison with crystalline metallic materials, and (4) the da/dn - $\Delta K_{eff}/E$ curve of the NC glassy alloys is coincident with those of ordinary crystalline alloys, where ΔK_{eff} is the effective stress

intensity factor range and E is the Young's modulus.

It has been reported that the tip of the fatigue crack in the single-phase metallic glass is in a quasi-melting state.⁵⁾ In the NC glassy alloys, if either quasi melting or a temperature rise occurs, the influence of the frequency of cyclic loading f on da/dn would be detected even in the ordinary range of f which does not affect da/dn in the crystalline metallic materials.⁸⁾ In this report, therefore, the influence of f on da/dn was examined for the NC glassy alloy.

It is well known that a single peak tensile overload during constant-amplitude loading induces retarded crack growth in crystalline metallic materials.⁹⁻¹⁶⁾ The physical explanations of the influence of overload given in the literature are residual compressive stresses near the crack tip and associated with yield zone interaction,^{10,14)} crack closure,^{9,11,12,15,16)} and crack branching along the shear bands generated by overload.¹³⁾ A number of reports have pointed out that the crack closure is the main mechanism. The influence of overload on da/dn was also examined for the NC glassy alloy which has different crack closure properties in comparison with the crystalline metallic materials described above.⁶⁾

2. Experimental Procedure

The NC bulk glassy alloy, $Zr_{55}Al_{10}Cu_{30}Ni_5$ (at%), was prepared by the squeeze casting method.¹⁾ The alloy has an average NC particle diameter of about 3 nm, tensile strength σ_B of 1.7 GPa and Young's modulus E of 87 GPa. A standard center-cracked-tension (CCT) specimen following ASTM E 647 was used in the fatigue crack growth rate testing. The specimen has a width of 30 mm and a thickness of about 2 mm. The specimen surface was electropolished by 30–60 μ m after machining, and the notch for crack initiation was introduced in the central part by electric discharge machining (EDM). The notch has a length of 12 mm, a width of 200 μ m

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and a tip radius of 50 μm . After the EDM, a strain gauge was attached in front of the crack tip in order to measure crack opening levels. The specimens were tested with a servohydraulic fatigue machine at room temperature.

The influence of the frequency of cyclic loading f was examined in the frequency range of 0.1 to 50 Hz and at the stress ratio R of 0.1 under sine and triangular waves. Figure 1 shows the loading spectra and load value combinations I and II (LVC I and LVC II), where ΔK_B , ΔK_{OL} and ΔK_A indicate the baseline stress intensity factor range before overloading, the overload stress intensity factor range and the stress intensity factor range after overloading, respectively. $\Delta K_{OL}/\Delta K_B$ is an overload ratio and approximately 5 in LVC I and 3 in LVC II. ΔK_B and ΔK_A were applied under constant-amplitude loading. Complete crack arrest was defined when no crack growth is observed using a metallographical microscope ($\times 500$) after 1×10^6 cycles under loading of a ΔK_A value equal to ΔK_B . After crack arrest, the amplitude was increased by 5–10% and the reloading was performed up to 1×10^6 cycles. Loading cycling was successively repeated until crack regrowth occurred.

The crack length was determined as the average of crack lengths measured with a metallographical microscope ($\times 100$ and $\times 500$) on both the specimen surfaces using plastic film replicas. The plastic film replication was conducted by stopping the testing machine at the mean load of ΔK_B or ΔK_A . A 1 mm precrack was introduced from the application of ΔK_B . For the case when the crack was propagated in a direction inclined toward the plane normal to the loading axis, the projected crack length was used to evaluate the K value of Mode I type.⁶⁾ The measurement of crack closure was performed by an unloading elastic compliance method¹⁷⁾ using the strain gauge.

Fig. 1 Condition of loading spectrum.

3. Results and Discussion

3.1 Influence of frequency

Figure 2 shows the relations between da/dn and ΔK (a) or $\Delta K_{eff}/E$ (b) under different f values. Large circles and triangles represent the present results under sine and triangular waves, respectively. The numbers by these large symbols indicate the f values. The small symbols indicate the previous results obtained at $f = 10\text{ Hz}$.⁶⁾ In the f range from 0.1 to 50 Hz under both sine and triangular waves, the large symbols, corresponding to growth rates less than $3 \times 10^{-5}\text{ mm/cycle}$, are situated within the scatter of small symbols. This implies the absence of frequency dependence on da/dn , suggesting that the rise of temperature does not affect the influence of da/dn under these conditions.

Fig. 2 Erdogan-Paris plot of fatigue crack for Zr-based NC bulk glassy alloy under different frequencies. (a) da/dn vs ΔK and (b) da/dn vs $\Delta K_{eff}/E$.

Fig. 3 Crack length a or crack opening ratio U versus number of loading cycles n . (a) load value combination (LVC) I and (b) LVC II.

3.2 Influence of overload

3.2.1 Crack growth behavior and crack closure

Figure 3 shows the crack length a and the crack opening ratio U against number of load cycles n obtained from the overload tests at the two different overload ratios. The value of U was evaluated by $(P_{\max} - P_{\text{open}})/(P_{\max} - P_{\min})$, where $P_{\max}$ is the maximum load, $P_{\min}$ is the minimum load and

Fig. 4 Load-strain hysteresis loop by the unloading elastic compliance method using a strain gauge. (a) before and (b) after overloading in LVC I.

P_{open} is the crack tip opening load. P_{open} was obtained from the hysteresis loop as typically shown in Fig. 4 for a LVC I spectrum.

In the case of the high overload ratio, LVC I (a), a complete crack arrest occurs after overloading, and the crack regrowth occurs when ΔK_A reaches about 1.5 times as large as ΔK_B . The $(\Delta K_A)_{\text{th}}$, which is the value of ΔK just before crack regrowth, is about 3 times larger than ΔK_{th} , which was obtained at about $1 \text{ MPa}\sqrt{m}$ in a previous test.⁶⁾ Almost the same result was obtained for a different specimen. No clear difference in the U values is observed among the steady crack growth region before overloading, the crack arrest region after overloading and the crack regrowth region.

In the case of the low overload ratio, LVC II (b), after overloadings, temporary crack arrests are observed and crack regrowth occurs when $\Delta K_A \cong \Delta K_B$. The U value remains nearly constant during the test period, similarly to that for LVC I. Thus, it is evident from the crack closure data shown in Fig. 3 that crack arrest cannot be explained by the crack closure mechanism.

3.2.2 Fracture surfaces and mechanism

Figure 5 shows the crack morphology on the specimen side-surfaces near the overloading region. In the case of the high overload ratio, LVC I (a), the crack grows to about $150 \mu\text{m}$ in the inclined direction after overloading and then gradually returns in the original direction. In the case of the low overload ratio, LVC II (b), the crack appears to be almost straight. The plastic zone sizes for the overloadings are calculated at $19 \mu\text{m}$ in LVC I and $5 \mu\text{m}$ in LVC II.¹⁸⁾ These sizes correspond to the length of slip lines indicated by the arrow lengths at the crack tips in the figures.

Figure 6 shows highly magnified SEM images around the crack tip using plastic film replicas taken just after overloading. The overloading in LVC I induces kinked small crack growth (Kink type, about $1 \mu\text{m}$) and branched small crack growth (Fork type, about $1 \mu\text{m}$) as shown in (a), while the overloading in LVC II does not show such kinking or branching in (b).

Figure 7 shows the fracture surface micrographs of the overloading region in LVC I. The longitudinal sections in the width direction of the specimen are drawn in the micrographs using stereo-matching observation. The crack profiles are mainly Kink type (a) and Fork I type (b), as is the case

Fig. 5 Crack growth morphology on specimen surfaces. (a) LVC I and (b) LVC II.

for the crack shapes observed on the specimen surface in Fig. 6(a). Another Fork II type branching is also partly observed in (c).

Kinked or branched crack growth induced by overloading in LVC I reduces the ΔK values. These ΔK values under the test conditions in LVC I are shown in Table 1. Here, ΔK_A and $(\Delta K_A)_{th}$ are the Mode I type stress intensity factor ranges based on the crack length which does not include kinked and branched small crack growth (about 1 μm), just after overloading and just before crack regrowth, respectively. The asterisked ΔK_A and $(\Delta K_A)_{th}$, ΔK_A^* and $(\Delta K_A^*)_{th}$, indicate the values considered for kinked and branched small crack growths, respectively, and $(\Delta K_A^*)_{eff}$ and $((\Delta K_A^*)_{th})_{eff}$ are obtained by $U \times \Delta K_A^*$ and $U \times (\Delta K_A^*)_{th}$, respectively. The kinking or branching angle θ of the cracks is assumed to be 75 degrees for both the Kink and Fork types. This angle was determined on the basis of the observation results of cracks on specimen surfaces (70–80 degrees, Fig. 6(a)) and fracture surfaces (70–90 degrees, Fig. 7). When a crack kinks or branches, the Mode I and Mode II stress intensity factors, K_I and K_{II} , respectively, occur, and the equivalent stress intensity factor K^* at the tip of the crack is calculated as $K^* = \sqrt{K_I^2 + K_{II}^2}$,¹⁹⁾ which is defined by the crack energy release rate. The K_I and K_{II} for the Kink and Fork type cracks are obtained from $F_I \times \Delta\sigma \sqrt{\pi a}$ and $F_{II} \times \Delta\sigma \sqrt{\pi a}$, respectively, where $\Delta\sigma$ is the stress range. Here, $F_I (= K_I / (\Delta\sigma \sqrt{\pi a}))$ and $F_{II} (= K_{II} / (\Delta\sigma \sqrt{\pi a}))$ are the non-dimensional stress intensity factors of the kinked or branched crack, which are given as a function of θ at $b/2c \rightarrow 0$ in the literature,^{20–24)} where b and c are defined in Table 1.

The ΔK_A^* values under the test conditions in LVC I are nearly equal to $\Delta K_{th} = (\Delta K_{eff})_{th} \cong 1 \text{ MPa}\sqrt{m}$.²⁵⁾ Since the $(\Delta K_A^*)_{eff}$ values are slightly less than the ΔK_{th} value, the $((\Delta K_A^*)_{th})_{eff}$ values may be approximated by the ΔK_{th} value. Table 2 shows the relation between ΔK_A^* for the Fork I type crack and crack branching angle θ . The ΔK_A^* values are considerably smaller than the ΔK_A value shown in Table 1, over the whole crack branching angle θ range. These results indicate that the crack arrest induced by overloading is explained mainly by taking account of the kinking and branching of cracks.

Figure 8 shows the fractography of the overloading region in LVC II. The longitudinal sections in the width direction of the specimen are also shown in the micrographs. The Kink type, Fork I type and Fork II type cracks are also observed in (a), (b) and (c), respectively. It is found that the kinked and branched cracks are intermittently mixed with the comparatively flat and nonbranching crack regions as observed in enlarged views of these figures. The striations on the fracture surfaces indicate that the local crack growth occurred in the direction as shown by the arrows. Thus, it is probable that the cracks grew from the flat regions, which would sustain a small stress reduction, to the regions of the large stress reduction induced by the kinking or branching of cracks. Before overloading, however, the local crack growth occurred nearly coincidentally with the macroscopic growth direction as shown by the arrows. Based on these observations, it is presumed that a temporary crack arrest or retardation occurs until the local crack growth direction restores to the macroscopic crack growth direction.

Table 1 Stress intensity factors for kinked or branched cracks.

Type of branching	ΔK_A	$(\Delta K_A)_{th}$	ΔK_A^*	$(\Delta K_A^*)_{th}/MPa\sqrt{m}$	$(\Delta K_A^*)_{eff}$	$((\Delta K_A^*)_{th})_{eff}$	Ref.
Kink	$\theta = 75^\circ$	2.3	3.4	1.1	1.7	0.83	20, 21)
	$\frac{b}{2c} \rightarrow 0$	2.1	3.1	0.97	1.4	0.89	
Fork I	$\theta = 75^\circ$	2.3	3.4	1.1	1.6	0.82	22, 23)
	$\frac{b}{2c} \rightarrow 0$	2.1	3.1	0.93	1.4	0.86	
Fork II	$\theta = 45^\circ$			A_1	A_2		22)
	$\frac{b}{2c} \rightarrow 0$	2.3	3.4	1.3	1.3	1.9	1.9
	$\frac{d}{b} = 0.7$	2.1	3.1	1.1	1.1	1.6	1.6
						1.0	1.0

$$K_A^* = \sqrt{K_I^2 + K_{II}^2}, (K_A^*)_{eff} = U \cdot K_A^*$$

Fig. 6 Microscopic crack tip morphology just after overloading by SEM using plastic film replicas. (a) LVC I and (b) LVC II.

The ΔK_{th} value in this NC glassy alloy is smaller than that in crystalline metallic materials. It is of interest to evaluate the maximum $(\Delta K_A)_{th}$ value in the NC glassy alloy which maintains crack arrest after overloading.

It is assumed that the reduction rate r of ΔK induced by kinking or branching of the crack is constant when the ΔK_{OL} value is large enough to satisfy the condition of LVC I. It

Fig. 7 Fractography near the overloading regions in LVC I.

is considered that the crack arrest occurs under the following condition:

$$(\Delta K_{eff})_{th} \cong 1 MPa\sqrt{m} \geq r \cdot U \cdot \Delta K.$$

From this equation,

$$U \leq \frac{1}{r} \cdot \frac{1}{\Delta K} \quad (1)$$

can be derived. The maximum $(\Delta K_A)_{th}$ is the maximum value of ΔK which satisfies eq. (1). As the U value depends on ΔK_B whether or not there is overloading, it is possible to es-

Table 2 ΔK_A^* for Fork I type crack versus crack branching angle θ under the LVC I condition ($b/2c \rightarrow 0$).

θ	45°	60°	75°	90°
$\Delta K_A^{*,22,23})$	1.3	1.3	1.1	0.95
/MPa $\sqrt{m}$	1.1	1.1	0.93	0.80

Fig. 8 Fractography near the overloading regions in LVC II.

timate eq. (1) using the relation between U and ΔK under the constant-amplitude loading. The r value is about 1/2.7 which was estimated on the basis of a complete crack arrest condition induced in LVC I ($U \cong 0.9$, $(\Delta K_A)_{th} \cong 3 \text{ MPa}\sqrt{m}$). Equation (1) is derived using the solid line in Fig. 9, which shows the relation between U and ΔK under the constant-amplitude loading reported previously.⁶⁾ If the U value is small, it is expected that the maximum $(\Delta K_A)_{th}$ will be approximately $4 \text{ MPa}\sqrt{m}$. Figure 10 shows a and U against number of load cycles n obtained in the overload test under the conditions of $\Delta K_B = 3.9$ and $\Delta K_{OL} = 11.5 \text{ MPa}\sqrt{m}$. Only a temporary crack arrest is observed. The reason for this is presumably because the U value is large in this test ($U = 0.9$). From these results and estimation, the upper limit of the $(\Delta K_A)_{th}$ is evaluated to be approximately $3 \text{ MPa}\sqrt{m}$, which is almost equal to the ΔK_{th} value for typical structural crystalline metallic materials, as shown in Fig. 2(a).

4. Conclusions

The influences of frequency and overload on the fatigue crack growth behavior were examined in a nanocrystalline

Fig. 9 Prediction of maximum threshold stress intensity factor range influenced by overloading.

Fig. 10 Crack length a or crack opening ratio U versus number of loading cycles n under $\Delta K_B \cong 3.9 \text{ MPa}\sqrt{m}$ and $\Delta K_{OL} = 11.5 \text{ MPa}\sqrt{m}$.

(NC) bulk glassy $\text{Zr}_{55}\text{Al}_{10}\text{Cu}_{30}\text{Ni}_{5}$ alloy. The results are summarized as follows.

(1) The fatigue crack growth rate below $3 \times 10^{-5} \text{ mm/cycle}$ was independent of frequency in the frequency range of 0.1 to 50 Hz at the stress ratio of 0.1 under the sine and triangular waves.

(2) When the overload ratio is low, a temporary crack arrest occurs. When the overload ratio is high, a complete crack arrest occurs, and the ΔK value just before crack regrowth is three times larger than the fatigue threshold stress intensity factor range ΔK_{th} .

(3) The crack arrest cannot be explained by the crack closure effect. The overloading induces the kinking and branching of the crack. The stress reduction near the crack tip due to the kinking and branching of the crack and the crack closure effect can explain the complete crack arrest and the large fatigue crack threshold stress intensity factor range (about $3 \text{ MPa}\sqrt{m}$).

(4) When the overload ratio is low, the kinking and branching of the crack does not occur uniformly at the crack front. The crack regrows from the region where the stress reduction is small. It is presumed that a temporary crack arrest or delay occurs until the local crack growth direction returns to the macroscopic crack growth direction.

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- 25) In the NC glassy alloy, as the U value is nearly equal to 1 at the low da/dn range near ΔK_{th} , ΔK_{th} is equal to $(\Delta K_{eff})_{th}$.