Comparative Study of Shear Fracture between Fe-based Amorphous and Ultrafine-grained Alloys Using Micro-tensile Testing

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Micro-tensile tests were employed to clarify the post-plastic-instability behavior in the shear fractures of specimens with the dimensions of 18×25×50 μm³ made from iron-based amorphous (AM) and ultrafine-grained (UFG) alloys. The AM specimen yielded by localized shear bands with an inclination angle of ~52° with respect to the loading axis, followed by sliding off almost throughout the entire specimen thickness. Micro-tensile and micro-shearing tests revealed that the Mohr failure envelope of the AM specimen could be described by a quadratic equation rather than a linear equation. Therefore, the sliding-off process is assisted by the applied normal stress, which suggests that it is caused by free-volume coalescence. For the UFG specimen, yielding set in by shear band formation with an inclination angle of ~45° with respect to the loading axis, following the Tresca criterion. Necking after shear band diffusion formed a triaxial stress state, which resulted in a final shear fracture plane via void coalescence in the UFG specimens. Voids formed along the intersection of the primary shear bands with secondary shear bands during the necking process. This indicates that the deviation of the shear fracture plane in the UFG specimen was determined by the strain development process. A comparison of the post-plastic-instability behavior between the AM and UFG specimens suggests that the external control of triaxial stress conditions is key to improving the formability of AM specimens.

KEY WORDS: amorphous alloys; ultrafine-grained alloys; shear fracture; strain hardening; micro-tensile testing.

1. Introduction

Although amorphization and grain refinement allow metals and alloys to be strengthened, amorphous¹⁻⁴) (AM) alloys as well as ultrafine-grained⁵⁻⁷) (UFG) alloys with grain sizes smaller than 1 μm have a tremendous negative impact on ductility via premature fracture due to localized shear deformation. This is because uniform elongation, which is defined as the elongation until the onset of plastic instability in a uniaxial tensile test, is often used as an indicator of ductility. In contrast, comparative studies of tensile behaviors between UFG and coarse-grained aluminum specimens⁸⁻⁹) revealed that the local elongation after the onset of necking was not significantly reduced by ultra-grain refinement. From a practical standpoint, local elongation rather than uniform elongation is a key property for determining the forming limit diagram.¹⁰) For AM alloys exhibiting poor formability, the development of an efficient processing method for the formation of complex shapes is a major challenge.¹¹⁻¹³) In fact, the Fe–Si–B AM alloy, which is attractive for high-performance motor cores, requires an improved formability. Because the deformation mechanism is directly linked to plasticity, an elucidation of the shear...
deformation process is essential for understanding plasticity from the viewpoint of formability.

The shear fracture behaviors of a Pd-based AM ribbon have been characterized by tensile tests using a specimen with gauge section dimensions of 0.5 mm (width) × 0.04 mm (thickness) × 6 mm (length), and revealed that a process of sliding off occurs almost throughout the entire specimen thickness. Similarly, another type of fracture was observed, that is, a shear fracture via sliding off with a length of ~20 μm in the width direction of the specimen, followed by the separation of the ligament without plastic strain. These findings indicate that sliding off is the elementary process of shear deformation in the AM alloy, and the relationship between the direction of sliding off and the aspect ratio of the cross-section of the tensile specimen determines the shear fracture behavior. Meanwhile, sample size effects on plasticity were reported for bulk metallic glasses (BMGs) and AM wires. For a specimen with a cross section smaller than the length of the sliding off part, the BMG deforms with necking, resulting in plastic strain. Thus, the deformation mechanism of AM alloys changes when the cross section of the specimen is smaller than the critical sliding-off length.

Interestingly, many researchers have reported that both AM and UFG alloys exhibit shear fracture planes deviating from the maximum shear stress plane with an inclination angle of 45° under uniaxial tensile stress conditions. The question that arises is whether the causes of these shear fractures are the same for both AM and UFG alloys. In this study, we focus on understanding the localized shear deformation process after the onset of plastic instability, which leads to premature shear fracture in AM and UFG alloys. Therefore, we performed uniaxial tensile tests on iron-based AM and UFG alloys using a low-aspect-ratio specimen with a cross section comparable to the critical sliding-off length, that is, a micro-tensile specimen. Recent advances in high-speed imaging techniques have helped observe the process of rapid shear fractures in BMGs. The combination of micro-tensile testing and high-speed imaging has enabled the capture of localized shear deformation processes.

2. Materials and Methods

The materials used in this study were a ribbon of commercially available Fe–Si–B AM Metglas® 2605HB1M and a UFG Fe-0.01 mass% C alloy composed of 0.01 Si, 0.012 P, 0.015 S, 0.23 Mn with a balance of Fe. The UFG alloy was obtained through high-pressure torsion (HPT) processing followed by annealing at 573 K for 0.5 h. The details of the HPT process conditions are described elsewhere. The surfaces of the UFG samples were polished with emery paper of up to #4000 and then finished using colloidal silica paste. The crystal orientations of the UFG samples were measured using automatic electron beam scanning at a step size of 0.06 μm under an accelerating voltage of 15 kV. Electron backscatter diffraction (EBSD) analysis was conducted using crystallographic orientation analysis software (OIM 7.1.0). Micro-tensile specimens with gauge section dimensions of 18 μm (width) × 25 μm (thickness) × 50 μm (length) were fabricated using a focused ion beam (FIB). Figures 1(a) and 1(b) show the shapes of the AM and UFG specimens, respectively, and an inverse pole figure map is overlaid onto the scanning electron microscope (SEM) image of the UFG specimen. The average grain size of the UFG specimen was determined to be approximately 246 nm. The loading directions (LDs) of the AM and UFG specimens were perpendicular to the rolling direction of the ribbon and the radial direction of the HPT disc, respectively. Tensile tests were conducted at a displacement rate of 0.1 μm s⁻¹, corresponding to a strain rate of 2 × 10⁻³ s⁻¹ under room-temperature atmospheric conditions. The gauge sections of some tensile specimens were monitored at a frame rate of 6 × 10⁴ fps during tensile testing using a high-speed camera (FASTCAM Mini AX200, Photron, Ltd.) for the AM’ and UFG’ specimens for a dynamic observation of the growth process of the shear bands. The specimens observed with the high-speed camera were distinguished by adding a prime symbol. For the AM’ specimen, the three-dimensional (3D) geometry of the fracture surface was analysed using Alicona MeX software. The 3D geometry images were constructed using three the SEM micrographs taken at tilt angles of 0° and ±5°. A micro-shearing test was conducted on the AM specimen to evaluate the critical shear fracture stress. Micro-shearing specimens with gauge section dimensions of 3 μm (depth) × 2 μm (width) × 8 μm (height) were fabricated using FIB. The conditions of the micro-shearing test are described elsewhere.

3. Results

3.1. Nominal Stress–nominal Strain Responses of AM and UFG Specimens

Figure 2 shows the nominal stress–nominal strain curves obtained through micro-tensile testing of the AM and UFG
specimens. The AM specimen fractured under a tensile stress of ~3.20 ± 0.06 GPa without plastic strain, whereby the errors across three stress-strain measurements were within ±5%. By contrast, the UFG specimen exhibited strain hardening after the onset of yielding at ~1.13 GPa (Fig. 2). The tensile fracture stress and elongation-to-failure (ε_F) were ~1.65 ± 0.07 GPa and 2.7%, respectively. These values are comparable to the results for a millimeter-sized UFG specimen in a previous report.\(^7\)

This indicates that the mechanical properties obtained using the micro-tensile test were equivalent to those of the bulk specimen. Both the AM and UFG specimens exhibited high tensile strengths while exhibiting low elongation-to-failure.

### 3.2. In-situ Observation of Shear Deformation Processes of AM’ Specimen

Figure 3 shows the deformation processes captured through in-situ high-speed imaging and the matching of 3D geometry images of the fracture surfaces of an AM’ specimen. A single shear band with an inclination angle of 52° with respect to the LD was generated from the bottom surface (Fig. 3(a)) and grew towards the top surface without an increase in the thickness of the band (Fig. 3(b)). After the sliding-off process (Figs. 3(c) and 3(d)), the final fracture occurred by ligament separation. Most of the fracture surface was covered with a smooth feature (indicated by the red arrow in Fig. 3(e)), whereas a wavy undulation formed in other parts (Fig. 3(e)). The matching analysis of the fracture surfaces reveals that the directions of the wavy undulation features do not correspond with each other, and the pattern on the fracture surface on the left side shifts downward (Fig. 3(e)). This indicates that the wavy undulation features are formed after the sliding-off process. Considering that the distance the sliding-off process covers corresponds to the area of the smooth feature,\(^15\) SEM observations perpendicular to the fracture surface revealed a sliding-off length of ~22.8 μm for the Fe–Si–B AM ribbon. Figure 4 shows the relationship between the shear strain, |γ_{xy}|, and the normal strain, ε_x, during the deformation of the AM’ specimen, which was obtained by measuring the distance between the gauge marks indicated by the yellow boxes in Figs. 3(a)–3(d). The shear strain–normal strain relationship of the AM’ specimen was |γ_{xy}|/ε_x = tan 52°, which corresponded to the inclination angle of the shear band (Fig. 3). This indicates that the shear fracture process of the AM’ specimen was dominated by the sliding off process.

![Fig. 2. (a) Nominal stress–nominal strain curves of the AM and UFG specimens.](image)

![Fig. 3. (a–d) Optical micrographs showing the shear fracture processes captured using a high-speed camera and (e) matching of 3D geometry images showing the fracture morphology of the AM’ specimen. (Online version in color.)](image)
3.3. **In-situ Observation of Shear Deformation of UFG′ Specimen**

Figure 5 shows the deformation processes and the typical fracture morphology of a UFG′ specimen. The first event of plastic deformation was the generation of shear bands at an inclination angle of 45° with respect to LD (Fig. 5(a)), which is in accordance with the Tresca criterion. This was followed by the growth of shear bands with an increase in their thickness, which led to the occurrence of diffuse necking, as indicated by the arrows in Fig. 5(b). Furthermore, the inclination angle of the shear bands increased from 45° to 52° at this stage. Thereafter, diffuse necking proceeded via the generation and growth of secondary shear bands (indicated by red lines in Fig. 5(c)). Finally, a shear fracture occurred via local necking at the intersection of the primary shear bands with the secondary shear bands (Fig. 5(d)). Consequently, the shear fracture plane was inclined at an angle of 52° with respect to the LD in the UFG′ specimen (Fig. 5(e)). Therefore, the deviation of the shear fracture plane from the maximum shear stress plane in the UFG′ specimen was a result of the plastic deformation under the triaxial stress state.

Figure 6 shows the fracture surface and the matching of the transmission electron microscopy (TEM) images of the post-tensile UFG′ specimen. The fracture surface of the UFG′ specimen was covered by elongated dimples (Fig. 6(a)), indicating that the final fracture occurred through the coalescence of voids. The TEM samples were obtained from the longitudinal sections indicated by the broken lines in Fig. 6(b). TEM observations revealed that the crystal grains were significantly deformed in the necking region (Fig. 6(c)). Close observation by TEM revealed that the crystal grains were elongated along the shear fracture direction in the local necking region, whereas the elongation directions were parallel to the LD in the diffuse necking region (Figs. 6(d) and 6(e)). Hence, the elongation trends were asymmetric with respect to the fracture surface. Furthermore, the selected-area electron diffraction patterns obtained from regions F and G in Fig. 6(c) exhibit directional and random features, respectively (Figs. 6(f) and 6(g)), indicating that the micro-texture develops in the necking region through alternating shear. These findings suggest that the crystal grains were elongated along the shear direction during the growth process of the primary shear bands (Fig. 5(b)), which was followed by crystal elongation along the LD during the diffuse necking process via secondary shear bands (Fig. 5(c)), resulting in void nucleation. Deformability after plastic instability is attributed to dislocation-based slip deformations and slip transfer inhibition by grain boundaries. In addition, the bright area indicated by the red arrows in the TEM bright-field (TEM-BF) image revealed that multiple voids were formed underneath the fracture surface on the right-hand side of the specimen (Figs. 6(h) and 6(i)), which suggests that voids preferentially formed in the region in which primary shear bands developed (Fig. 5). Therefore, it is anticipated that the shear fracture of an UFG specimen occurs via the coalescence of voids formed along the damage-evolved region under the triaxial stress state.
4. Discussion

In-situ observation of deformation in the AM specimen revealed that yielding occurred by shear banding with an inclination angle of 52° with respect to the LD. If shear stress dominates the yielding, a shear band should be generated along the maximum shear stress plane with an inclination angle of 45° with respect to the LD. This indicates that the generation of shear bands in the AM specimen did not follow the Tresca criterion. Donovan proposed that the tensile fracture of the BMG conforms to the Mohr–Coulomb (M–C) criterion rather than the Tresca criterion. The M–C criterion has been applied to brittle materials, such as soil, rock, and polymers, and considers the contribution of normal stress to a fracture. The criterion under the uniaxial tensile loading condition is expressed using the following equations:

\[ \tau_\theta = \tau_0 - \mu \sigma_\theta, \quad (1) \]
\[ \sigma_\theta = \sigma_T^2 \sin^3(\theta), \quad (2) \]
\[ \tau_\theta = \sigma_T^2 \sin(\theta) \cos(\theta), \quad (3) \]
\[ \mu = -\frac{1}{\tan(2\theta)}, \quad (4) \]
where \( \tau_0 \) and \( \sigma_0 \) are the resolved shear and normal stresses applied to the shear plane, respectively; \( \tau_0 \) is the critical shear stress; \( \mu \) is a constant that reflects the effect of the normal stress; \( \theta \) is the angle of the shear plane with respect to the normal direction of the principal stress plane; and \( \sigma_F^T \) is the tensile fracture stress. The use of the tensile test results and the M–C criterion revealed a critical shear stress under uniaxial tensile loading, \( \tau_0^T \), of 2.05 GPa.

To examine the validity of the critical shear stress value, a micro-shearing test was performed on the AM specimen. Figures 7(a) and 7(b) show the shear stress–displacement curve obtained through the micro-shearing test and the SEM image of the deformed gauge part of the AM specimen, respectively. The micro-shearing test revealed an abrupt stress drop at a shear stress of \( \sim 1.79 \) GPa concurrent with the formation of a large step in the gauge section. Furthermore, the step was slightly inclined with respect to the LD. Meanwhile, using the maximum shear stress, \( \tau_M \), and the mean normal stress, \( \sigma_M \), the M–C criterion is given by:

\[
\tau_M + \sigma_M \cos(\theta) = \tau_0 \sin(\theta).
\] .......................... (5)

As the value of \( \sigma \) is zero under the pure shear loading condition, the M–C criterion can be expressed by the following equation:

\[
\tau_M = \tau_0 \sin(2\theta). \quad \text{.......................... (6)}
\]

Figure 7(c) shows the Mohr’s circles in \( \tau-\sigma \) coordinates, which were obtained from the results of the micro-shearing and tensile tests of the AM specimen. Mohr’s circle of the micro-shearing test reveals that the angle between the principal stress plane and the maximum shear stress plane is \( 45^\circ \), which indicates that the normal to the principal stress plane is inclined by \( 45^\circ \) with respect to the LD, as shown in Fig. 7(b). Therefore, using the M–C criterion, the critical shear stress under pure shear loading, \( \tau_0^S \), was determined to be 1.85 GPa. Thus, applying the M–C criterion to the uniaxial tensile test results overestimated the critical shear stress of the AM specimen (Fig. 7(c)). These findings suggest that the Mohr failure envelope in the AM specimen can be described using a quadratic equation rather than a linear equation. It has been reported that the Mohr failure envelopes of unsaturated soils are described by a quadratic equation.
equation, whereas those of saturated soils are described by a linear equation. This implies that the generation of shear bands in the AM specimen is related to a dilatation of the space between atoms.

Figure 8 shows the deformation microstructure of the AM specimen. Figures 8(a) and 8(b) show the fracture morphology and sampling positions for the TEM observations of the AM specimen. A shear band with interrupted growth was observed in the region several microns from the fracture surface of the AM specimen (Fig. 8(a)). TEM samples 1 and 2 were prepared in the post-shear-banding and shear-band-interrupted regions, respectively, as shown in Fig. 8(b). Figure 8(c) shows the TEM-BF image of sample 1, and Fig. 8(d) shows the high-angle annular dark-field scanning TEM (HAADF-STEM) image obtained of the boxed region in Fig. 8(c). A straight shear band with a thickness of approximately 10 nm was formed in the region of sample 1. Meanwhile, the TEM images of sample 2 (Figs. 8(e) and 8(f)) reveal a dotted shear band in the shear-band-interrupted region, which implies that the cores of the shear band were formed just before the growth of the shear band. Considering that the dilatation of space between atoms is a key factor for the generation of shear bands in the AM specimen, as discussed above, it is plausible that the shear bands are generated by the multiplication of free volume, which requires normal stress.²⁷

Fig. 8. (a) SEM image showing the fracture morphology and (b) schematic illustration showing the positions of the TEM samples of the AM specimen. (c) TEM bright-field image of sample 1 and (d) HAADF-STEM image for the boxed region in (c). (d) TEM bright-field image of sample 2 and (f) HAADF-STEM image for the boxed region in (d). (Online version in color.)
Although the angle of the shear fracture plane with respect to the LD deviated from the maximum shear stress plane in both the AM and UFG specimens, yielding of the UFG specimen occurred via shear banding along the maximum shear stress plane. The deviation phenomenon of the shear fracture plane in the UFG specimen, which is different from that in the AM specimen, is explained below. Figure 9(a) shows the relationship between the shear strain, $|\gamma_{xy}|$, and the normal strain, $\varepsilon_n$, during the deformation of the UFG specimen, which was obtained by measuring the distance of the gauge marks indicated by the yellow boxes in Figs. 5(a)–5(d). Figures 9(b)–9(e) show schematic illustrations of the deformation processes corresponding to Fig. 9(a), with observation results shown in Figs. 5 and 6. In the UFG specimen, the initial deformation proceeded according to the shear strain–normal strain relationship $|\gamma_{xy}|/\varepsilon_n = \tan 45^\circ$, which corresponds to the inclination angle of the primary shear bands (Figs. 9(a) and 9(b)). The critical shear stress of ~0.57 GPa for shear banding could be estimated by resolving the yield stress of the UFG specimen into the shear component. Because the shear strain–normal strain relationship follows the Tresca criterion, it is suggested that the primary shear bands in the UFG specimen originated in the weakest part of the crystalline region containing dislocations. After the generation of the primary shear bands, the shear strain gradually decreased (Fig. 9(a)), which corresponds to the growth process of the primary shear bands. It should be noted that the growth region of the primary shear bands was local, as shown in Fig. 5(b). Localized shear produces a plastic constraint stemming from the surrounding undeformed region, so that the geometric shape can be retained, which leads to the onset of diffuse necking. Consequently, the inclination angle of the shear bands increased (Fig. 9(c)). After this process, a plateau regime without shear strain appears in Fig. 9(a), which corresponds to a progression of the diffuse necking process via the generation and growth of secondary shear bands (Fig. 5(c)). Considering that normal stress is recognized as a necessary component for the dilatation of space, it is suggested that voids were generated during the diffuse necking process along the damage-evolved region, as shown in Figs. 9(a) and 9(d). After this regime, $|\gamma_{xy}|$ tended to increase again until the final fracture (Fig. 9(a)), which corresponds to the local necking process in Fig. 5(d). This implies that the voids coalesced through localized shear, as shown in Fig. 9(e). Therefore, it was revealed that the deviation of the shear fracture plane from the maximum shear stress plane in the UFG specimen was determined by the development process of the strain under the triaxial stress state.

Close examination of the shear deformation process revealed that the angle of the shear fracture plane in the UFG specimen was determined by damage evolution via dislocation-based shear deformation and plastic deformation under a triaxial stress state. This was attributed to the strain hardenability of the UFG specimen owing to the dislocation-based slip deformation and slip transfer inhibition by the grain boundaries. However, the shear deformation of the AM specimen, which is based on a combination of normal stress and shear stress, does not lead to strain hardening. This implies that a deformation after plastic instability is quite difficult for AM specimens. Therefore, it is suggested that the external control of multiaxial stress loading conditions is an effective strategy for improving the formability of AM specimens.

5. Conclusions

Micro-tensile tests in combination with the in-situ high-
speed imaging technique were performed on iron-based AM and UFG alloys to clarify the post-plastic-instability behavior in shear fractures. For the AM specimen, a micro-shearing test was performed to examine the yield criterion. The conclusions can be summarized as follows:

1. The Fe-based AM specimen exhibited brittle stress–strain behavior with a tensile fracture stress of ~3.20 ± 0.07 GPa. Application of the M–C criterion to the results of the micro-shearing test revealed a critical shear stress of ~1.65 ± 0.07 GPa via strain hardening, and the critical shear stress for yielding was ~0.57 GPa.

2. In-situ observation of the AM specimen using a high-speed imaging technique revealed that yielding occurred via the formation of localized shear bands with an inclination angle of ~52° with respect to the loading axis. Followed by the sliding-off process. The Mohr failure envelope in the AM specimen can be described by a quadratic equation rather than a linear equation, which suggests that the generation of shear bands can be attributed to the multiplication of the free volume, which requires the assistance of applied normal stress.

3. In the Fe-based UFG alloy, yielding occurred with dislocation-based shear banding at an inclination angle of 45° with respect to the loading direction, which followed the Tresca criterion. Shear fracture of the UFG specimen occurred through the coalescence of voids formed in the damage-evolved region during the necking process. The deviation of the shear fracture plane in the UFG specimen was attributed to the development of strain under the triaxial stress state.

A comparison of the post-plastic-instability behavior between the AM and UFG specimens suggested that the formability in the triaxial stress state can be attributed to the potential strain hardenability, and it is suggested that the external control of triaxial stress conditions is effective in improving the formability of AM specimens.

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REFERENCES