

## DEFORMATION MECHANISMS AND FRACTURE OF Ni-BASED METALLIC GLASSES

The cracking of materials and fracture surface is of great practical and academic importance. Over the last few years the development of the fractography of crystalline alloys resulted in a useful tool for the prediction or failure analysis. Many attempts have been made to observe cracks using optical microscopy, X-ray topography and transmission electron microscopy (TEM). Of these techniques, the resolution of optical microscopy and X-ray topography is too poor. By contrast, the resolution of TEM is high enough for detailed information to be obtained. However, in order to apply TEM observations, a thin foil specimen must be prepared, and it is usually extremely difficult to prepare such a specimen from a pre-selected region containing a crack.

In the present work, deformation mechanisms fracture surfaces of Ni-based metallic glass samples have been studied by specially designed experiments. In order to study the deformation mechanisms and fracture the Ni-based metallic glasses have been investigated in the tensile test. The structure and fracture surfaces after the decohesion process in tensile tests were observed using transmission electron microscope (TEM) and scanning electron microscope (SEM), respectively. The studies of structure were performed on thin foils.

Moreover the investigated tape was subjected to a banding test. Then, the tape was straightened and the thin foil from the area of maximum strain was prepared. This thin foil sample was deformed before the TEM investigation to obtain local tears.

*Keywords:* metals, transmission electron microscopy, scanning electron microscopy, fracture, shear bands

### 1. Introduction

Based on their attractive properties, such as good soft magnetism as well as mechanical behaviour (high yield strength, high hardness, good wear resistance) or excellent corrosion resistance, many metallic glasses (MGs) have potential technological applications in various areas: machinery structural materials, die materials, tool materials, hydrogen storage materials, photovoltaic materials, sporting goods materials, etc. [1-6].

Metallic glasses have a disordered structure as in traditional glasses, but their atoms are densely packed via cohesive metallic bonds as in traditional metals. This unusual combination leads to unique properties. Metallic glasses have a very high strength, hardness and elastic limit. The high strength of MGs is frequently accompanied by more or less pronounced plastic deformation and their deformation and fracture mechanisms are quite different from crystalline materials. Metallic glasses seldom exhibit tensile ductility at room temperature and are considered quasi-brittle materials. Although these amorphous metals are capable of shear flow, severe plastic instability sets in at the onset of plastic deformation, which seems to be exclusively localized in extremely thin shear bands [7].

The first studies of metallic glasses, limited by the small and irregular samples that were available, focused on thermal, electrical, magnetic and structural properties. A study of the

mechanical properties of these novel materials was first reported in 1971 by Masumoto and Maddin [8]. They conducted tensile tests over a wide range of temperature and strain rate. They found that, despite the high value of strength and the macroscopically brittle behaviour, the fracture stress was essentially independent of sample volume. On the surfaces of bent samples “cracks or deformation lines” developed, associated with surface steps; this appears to be the first observation of what are now known as shear bands, and it led Masumoto and Maddin to conclude “that plastic flow occurs in the amorphous alloys but by a process considerably different from crystalline metals”. They estimated the deformation-line thickness to be 20 nm; interestingly, this is very consistent with the values for shear-band thickness obtained in later work, mostly by transmission electron microscopy (reviewed in Ref. [9]) - Table 1.

This is comparable with the observed thickness of 10 to 20 nm, suggesting that in analysing shear in metallic glasses, attention should be paid to the possible effects of a structure based on packing of atomic clusters. The thickness of shear bands in metallic glasses indicates a structural, not thermal, origin. Local plasticity at fracture occurs in thicker liquidlike layers associated with heat generation in the bands. Shear localization within a supercooled or equilibrium liquid layer can be understood in terms of isoconfigurational viscosity changes on rapid heating [7,9]. In metallic glasses, shear bands are particularly important as they play the decisive role in controlling plasticity and failure at room temperature.

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Summary of observations of shear-band thickness in metallic glasses [9]

| Metallic glass, composition in at. [%]   | Mode of observation* | Shear-band thickness [nm] | Reference |
|--|----------------------|---------------------------|-----------|
| Pd <sub>80</sub> Si <sub>20</sub>  | TEM                  | 20                        | [8]       |
| Fe <sub>40</sub> Ni <sub>40</sub> B <sub>20</sub>  | TEM                  | 10–20                     | [10]      |
| Zr <sub>56.3</sub> Ti <sub>13.8</sub> Cu <sub>6.9</sub> Ni <sub>5.6</sub> Nb <sub>5.0</sub> Be <sub>12.5</sub> | TEM                  | ≤10                       | [11]      |
| Zr <sub>57</sub> Ti <sub>5</sub> Cu <sub>20</sub> Ni <sub>8</sub> Al <sub>10</sub>                             | HRTEM                | ≤20                       | [12]      |
| Al <sub>90</sub> Fe <sub>5</sub> Gd <sub>5</sub>   | HRTEM                | 10–15                     | [13]      |
| Al <sub>86.8</sub> Ni <sub>3.7</sub> Y <sub>9.5</sub>  | HRTEM                | 10–15                     | [13]      |

\*TEM, transmission electron microscopy; HRTEM, high-resolution TEM

Due to the ribbon geometry of rapidly quenched glasses, early experimental work on fracture typically employed notched or unnotched bending tests to evaluate the fracture behaviour of metallic glasses [14,15]. Mostly, these studies were directed at determining whether a particular metallic glass is ductile or brittle and how the fracture behaviour depends on composition, processing (including quenching rates, structural relaxation and hydrogenation), and temperature [15]. The shear nature of plastic deformation in metallic glasses was established by Leamy et al. [16]. Their studies of the outer surface of bent samples showed primary and secondary steps corresponding to significant shear displacements with, remarkably, no sign of tensile cracks. They noted that samples failing in tension do so on the plane of maximum shear stress, at roughly 45° to the loading axis.

The fracture surfaces have two regions, corresponding to the two stages shown in Fig. 1.

The initial offset gives a smooth sheared surface, and is followed by localized necking giving a characteristic pattern of raised ridges: this is the vein pattern as originally named by Leamy and co-workers. The final fracture stage in Fig. 1 should give patterns on both opposing surfaces that roughly match each other. Leamy et al. noted that for the heating to be significant, the flow must be confined to thin shear deformation bands, and they considered that their operation would be close to adiabatic. Then as now, fracture was considered “ductile” if it involved large plastic strains, at least locally, due to inhomogeneous flow. In this case, the fracture surface morphology exhibits a characteristic “vein” or “river” pattern. In contrast, brittle fracture is reflected as a relatively smooth fracture morphology [14,17]. A major drawback, which hinders the application potential, is the lack of plasticity metallic glasses [18,19]. This also hinders the precise study of some fundamental issues in glasses, such as the deformation mechanism and the dynamics of plastic deformation, in which large plasticity is needed for detailed analysis [20].

In the present work, deformation mechanisms and fracture of Ni-based metallic glass samples have been studied by specially designed experiments. In order to study the mechanisms of deformation and fracture in the Ni based metallic glass have been investigated in the tensile test. The structure and fracture surfaces after the decohesion process in tensile tests were observed in transmission electron microscope (TEM) and scanning electron microscope (SEM), respectively. The studies of structure were performed on thin foils. This thin foil sample before the TEM investigation was deformed to obtain local tears.

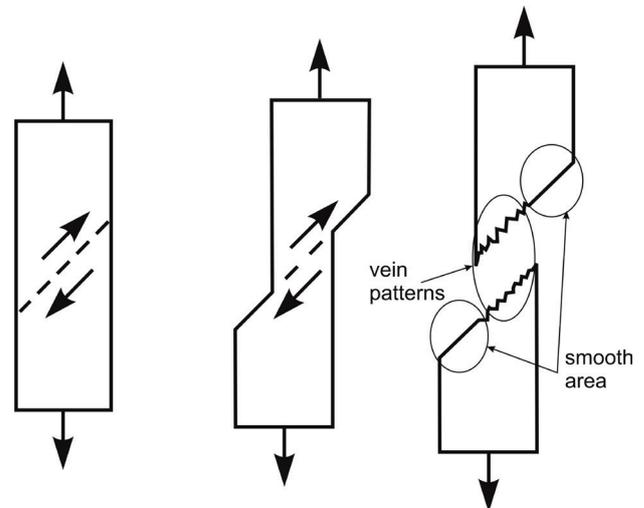


Fig. 1. The flow and fracture process by which metallic-glass samples tested in tension have fracture surfaces with two distinct areas: smooth and veined (circled). At the two ends of the shear offset the surface is smooth and shiny, suggesting that the temperature has not been very high. In the middle are the typical vein patterns, indicating the high local temperature that has been reached during fracture [16]

## 2. Material and methods

### 2.1. Material

Experiments were performed on tapes of amorphous Ni<sub>68.7</sub>Cr<sub>6.6</sub>Fe<sub>2.65</sub>Si<sub>7.8</sub>B<sub>14</sub>C<sub>0.25</sub> alloy. The alloy composition represents nominal atomic percentages. The tapes were produced by method of “chill - block - melt- spinning” - it is method of continuous casting of the liquid alloy. Tapes had 0.03 mm of thickness.

### 2.2. Experimental procedure

In order to study the mechanisms of deformation and fracture in the Ni based metallic glass have been investigated in the tensile test. Tensile tests at ambient temperature by an Instron-type testing machine were performed with tension rate 5 mm/min. Tensile-test specimens were 50 mm mm length.

The structure and fracture surfaces after the decohesion process in tensile tests were observed in transmission electron microscope Tesla BS 540 and scanning electron microscope (SEM) Jeol JSM-35, respectively. The studies of structure were performed on thin foils.

Moreover the investigated tape was subjected to banding test, which resulted in surface contact expressed as  $\varepsilon = 1$ . Then, the tape was straightened and the thin foil from the area of maximum strain was prepared. This thin foil sample before the TEM investigation was deformed to obtain local tears.

### 3. Results and discussion

The amorphous state of samples acknowledges both, the structure observations and investigation by method of selective electron diffraction. Generally there are typical pictures for amorphous structure. The broad diffraction rings formed as a result of electron beam dissipation, characteristic for amorphous state, showed electron diffraction pictures (Fig.2).

Investigations of fractures after decohesion in tensile test confirmed high plasticity of  $\text{Ni}_{68.7}\text{Cr}_{6.6}\text{Fe}_{2.65}\text{Si}_{7.8}\text{B}_{14}\text{C}_{0.25}$  tapes and revealed their ductile character with vein pattern morphology, standard for ductile metallic glasses.

Areas of narrow shear bands parallel to the fracture are visible on the external side of the tape, near the fracture (Fig. 3-5). Shear bands can be locally contorted and may have different thickness of the interlayer. Moreover areas of wedge-shaped zones connecting adjacent plastic strain areas are visible (Fig. 4,5). These effects of localized inhomogeneous strain can indicate that forming of shear band can proceed by stages. In the first stage the shear band was formed; in the second stage its hampering and successive activation only in the part of the band took place. This effect is accompanied by the formation of a transition zone from one band to the neighboring band or coupling of the shear bands in the inhomogeneous strain activation process (Fig. 4,5).

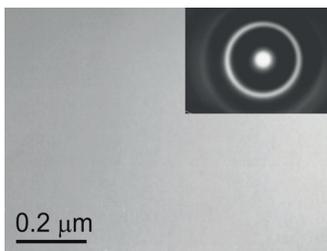


Fig. 2. TEM, with electron diffraction; Amorphous structure of samples in as quenched state

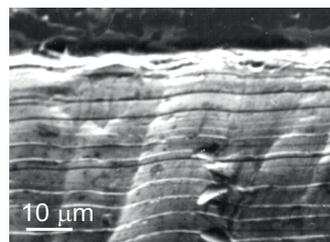


Fig. 3. SEM image of fracture morphology on the external side of tapes after decohesion in tensile test. Areas of narrow shear bands parallel to the fracture are observed near the fracture

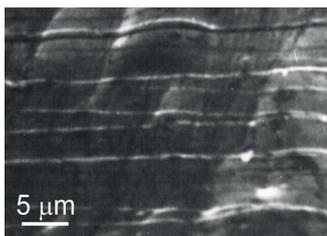


Fig. 4. SEM image of fragment of fracture morphology presented in Fig. 3. The shear bands areas and areas of wedge-shaped zones connecting bands are visible

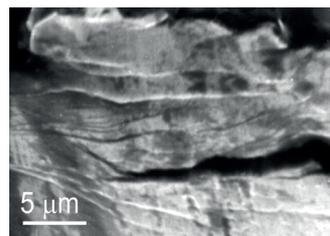


Fig. 5. SEM image of fracture morphology on the external side of tapes after decohesion in tensile test

All local effects of inhomogeneous plastic deformation occur above a limit tensile stress and lead to the decohesion of metallic glasses. A total plastic deformation through the development of shear bands gives the plastic component of the deformation of metallic glasses.

Two different regions (Fig. 6) in the fracture surface formed from the divergence of one of the shear band (Figs. 2,5). Smooth area region occurs at one of the fracture edge. This region changes in to the vein pattern area in the remaining part of the fracture. It is accepted, that smooth region morphology is a result of the feed due to the slip mechanism of the part of the sample beyond the side surface of the neighbouring region. It leads to a decrease in a cross-section of the sample, a significant increase of stress at a critical region and finally to a viscous divergence of the remaining part of the sample and formation of the vein pattern area. Leamy et al. [16] suggested that this type of fracture is due to the adiabatic heating created by plastic flow. This, in turn, leads to the localized deformation and eventually the specimen fails and leaves the vein pattern. A somewhat similar view although with a different mechanism is that of Spaepen [21]. The fracture pattern formed is considered to be the result of the breaking of a fluid layer between two solid surfaces. The fluid layer (the slip band) is formed because of the increased "free volume" created by the hydrostatic tension existent at the head of a growing band. This lowers the viscosity within that region down to values typical of a fluid. In both models an important role is played by the rapid have been performed.

The vein pattern area of characteristic morphology containing smooth regions and typical buckles (Fig. 7) testifies that at the moment of the divergence and fracture formation the change of the viscosity and local viscous material flow take place. It results in tearing off viscous medium layer.

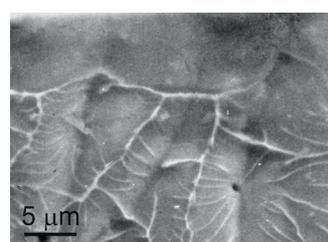


Fig. 6. SEM image of fracture surface of sample - tape. From the one edge of the smooth area fracture passes to the vein pattern area

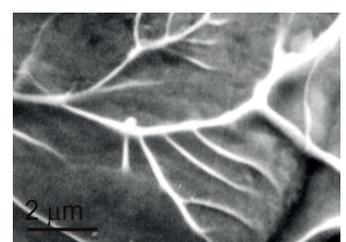


Fig. 7. SEM image of fracture surface of sample - tape. Vein-like morphology

The thin foil sample parallel to surface - tape prepared and deformed before the TEM investigation reveals an amorphous structure with areas of band form with a different bright contrast when compared to an amorphous matrix (Fig. 8). Band width is from 10 to 30 nm. A review of measurements and atomistic modelling shows that shear bands in metallic glasses have a characteristic thickness of 10 nm [9].

The TEM micrograph with contrast changes of amorphous structure indicates that in the areas of bright contrast exists the reduction of atom packing density with presumable topological and chemical changes. These changes influence a reduction of the viscosity of these

areas. It seems probable that the unique image of shear band in cross-section was achieved (Fig. 8). A reduction of the viscosity in these areas; as suggested by one of the theory of plastic deformation of the metallic glass, would result in an atomic distance dilation similar to observed effects, which should confirm the occurrence of slip mechanism in the shear bands. According to Sethi, Gibala et al., the regions around the cracks are brighter than the rest of the foil because of localized necking that precedes crack propagation [22].

Studies of the crack propagation produced on the thin foil (Fig. 9,10) indicate that the development of cracks must not be straight; the crack may consist of straight and curved sections. Development stages of a crack are also likely and its retention leading to relaxation of stresses at the edge of the cracks and the formation of dense random packing of the atoms changes. The characteristic decay at some distance from the cracks, the areas of reduced contrast of amorphous structure are visible in the neighbourhood of a crack (Fig. 9). As in the case of the image traces of the shear bands, the lowered contrast indicates the formation of dense random packing of the atoms changes in an amorphous structure. The crack front may have several alternative directions of crack development in the areas of contrast changes, visible in Fig. 9, or have one clear direction of propagation - Fig. 10. As a result, along the cracks lateral areas of reduced contrast not participating in the development of the slit are also visible.

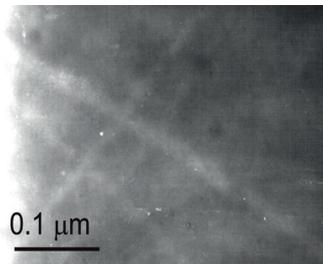


Fig. 8. TEM micrograph; areas of band form with different bright contrast when compared to an amorphous matrix

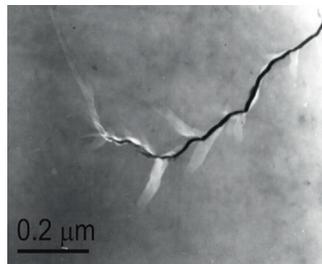


Fig. 9. TEM micrograph around a crack and at its neighbourhood. The crack front with contrast changes of amorphous structure

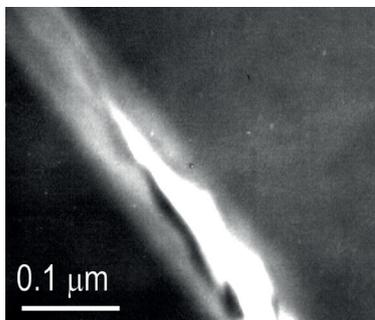


Fig. 10. TEM micrograph around a crack and at its neighbourhood. The crack front with contrast changes of amorphous structure

#### 4. Conclusions

The presence of different morphologies on the fracture surface raises basic questions about the conditions as well as the

time sequence of their formation. The fracture surface analysis of the Ni-based metallic glasses reveals two morphologies: vein-like pattern and smooth regions.

The presence of areas of different morphologies in the fracture of examined specimens is related to an operating deformation mechanism, which is characteristic for metallic glasses. The tensile stresses above the proof stress and corresponding to tensile strength of a glass leads to activation of shear bands resulting in located parallel (or close to parallel) bands and slips visible on the sample surface. It seems probable that the shear bands generated at the angle of 45° according to an acting force leads to parallel small displacement of a part of the sample resulting in the interposition of the part beyond an outer surface preferentially along one of bands. This displacement of a part of the sample leads to reduction of a true section of the sample near the band and a rapid increase in stress. Finally, the sample cracks and the cross section shows a vein pattern area which is possible to obtain under conditions of changes in the density of atoms in the shear band and reduction of the viscosity. This part of the fracture morphology shows similarities to separation of the viscous surface of the substance. On the other hand the side part of a sample ejected by shearing off the surface has a smooth surface. From this point further crack spreads passing to the vein-pattern morphology by the mechanism which was described above. The present mechanism of cracking with the formation of dense random packing of the atoms changes in the shear band was confirmed by the results of the TEM tests. Thin foils made of deformation areas are the narrow bandwidth of the reduced contrast which is associated with a dense random packing of the atoms reduction of those areas and, accordingly changes in the positioning of atoms. The presumption is that these areas from neighbouring places to spawn the free volume of the overhead incurred by reducing the viscosity of glass in the shear band. The shear bands consist of a thin layer of low viscous material with chemically and atomically changed short-range order with excess of free volume.

Interesting images were also obtained for the thin film possessing a local tear. As in the case of shear bands the microscopic image with distinct changes of reduced contrast was observed in the area of developing cracks and fissures created in front of the cracks. The studies have indirectly revealed the significant impact of density changes of atom arrangement in the shear band and in front of the cracks on the process and mechanism of plastic fracture of metallic glass under tensile stress conditions.

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