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# Shear striations and deformation kinetics in highly deformed Zr-based bulk metallic glasses

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**Abstract**

Detailed microscopic observations of the shear surfaces of a deformed, but unfractured, bulk metallic glass sample reveal a wealth of information on the deformation characteristics, kinetics and influence of temperature during serrated flow. The shear surfaces exhibit shear striations, which are similar to those resulting from viscous-like flow in rock-forming minerals. On the shear surface only a few areas show typical vein patterns, the thicknesses of which are less than those known from fracture surfaces. Combined with estimates for adiabatic heating, this indicates that sufficiently high temperatures are already present during shear banding before fracture, though instigated by non-purely adiabatic effects. A kinetic model based on an energy variable which reflects the structural relaxation ability is proposed that accounts for the occurrence of serrated flow combined with negative strain rate sensitivity, and the transition to non-serrated flow, i.e. positive strain rate sensitivity, below a critical temperature and strain rate.
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*Keywords:* Bulk metallic glasses; Adiabatic heating; Deformation kinetics; Structural relaxation; Strain rate sensitivity

## 1. Introduction

Despite the lack of significant plasticity in bulk metallic glasses (BMGs), deformation is highly localized in a few shear bands, where microscopically large shear strains $\gg 1$ take place. Similar to the discrete displacement bursts at shallow depth or low loads observed in single-crystalline metals, shear strain increments of several tens of nanometers are also measured by nanoindentation or microcompression tests in BMGs [1,2]. In single-crystals these "pop-ins" are associated with dislocation nucleation and propagation [3–6], while in BMGs they are associated with the nucleation and percolation of shear transformation zones (STZ) (clusters of several tens of atoms), which lead to macroscopic shear steps [7]. Despite the fundamental structural difference between a single crystal and an amorphous structure, they show some similarities in their mechanical properties: in micropillar compression experiments both materials exhibit similar inhomogeneous flow behaviour with shearing along the angle of the maximum resolved shear stress, which, in contrast to polycrystalline materials, shows a unidirectional orientation of the shear plane in both materials. While the shear surface of a single slip glide plane in crystalline materials is expected to be atomically smooth, a rougher surface can be envisioned in amorphous metals due to their structural disorder. Recent work in micropillar experiments on single-crystalline samples indicates, as expected, smooth glide/shear surfaces [8]. Apart from the interatomic binding potential, it is this configurational atomic arrangement that defines the resistance to shearing (decoupling and recoupling of atomic bonds). Hence a smoother atomic surface exhibits a lower shear resistance/stress than a rougher one. That is why the shear stress for single dislocation events in a single-crystalline sample of conventional size is difficult to discern with a standard mechanical testing setup. Only at sufficiently small sample size and high force resolution (nano-Newton range) are discrete displacement bursts detectable in single-crystals [3–5,9,10]. In BMGs, on the

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other hand, the shear resistance is up to two orders of magnitude higher than in single crystals, and thus a shear event can also be discerned in a macroscopic specimen. One focus of this work is to characterize such shear events in detail.

So far only a few literature reports have paid attention to the surface morphology of shear planes in bulk amorphous metals [11,12]. This is mainly related to the generally small amount of plastic strain occurring before failure, which would otherwise result in large enough shear offsets and thus allow for a more detailed microscopic examination. The observation of the surface morphology of shear planes is important information for the description of possible deformation mechanisms in metallic glasses. Fracture surfaces of metallic glasses, on the other hand, are well known and described by their typical river, vein or other flow patterns [13,14]. These flow features suggest that temperatures well above the glass transition temperature ($T_g$) are reached at the moment of fracture. Since their first observations, several thermographic experiments have supported the idea of such high-temperature bursts at fracture [15–18]. Some ambiguity with respect to adiabatic heating and the amount of temperature rise associated with shearing before fracture, however, remains, stimulating recent debate as to whether the temperature might be sufficiently high to instigate a localized drop in viscosity and thus promote easy glide along this softened shear zone [17,19–22].

In addition, a comprehensive picture of the deformation kinetics during inhomogeneous deformation in metallic glasses is still not available [23]. Apart from the temperature increase during localized shearing, the shear dilatation models (the STZ model [7] and the free volume or diffusive-jump-like model [24,25]) have found wide acceptance in explaining the drop in viscosity. As also recently reviewed by Schuh et al. [23], these two models, based on a drop in viscosity, contrast with the dislocation approach first adapted to metallic glasses by Gilman and later supported by others ([26–29] and references therein). In contrast to the model of Argon, where the nucleation of STZs (i.e. local clusters of atoms designated as the carriers of motion) reflects the critical step for the activation of shear, in Gilman's model, dislocation-like structures are responsible for the shear deformation. These create the necessary stress field and thus generate the resistance to shear propagation. According to Gilman, the description of the energy needed to cause dilatation and consequently allow for motion of the dislocation over the energy barrier is derived by treating the dilatation as point defects [26]. Therefore, with respect to the dilatational force needed to activate shear, common agreement is found between the various models. In addition, despite the difference in describing the structure and motion of the flow defect, similar thermally activated rate equations have been derived. On the other hand, however, none of the constitutive equations for inhomogeneous plastic flow ascribed by these models addresses sufficiently well the serrated flow as a kinetic phenomenon, this being partially due to the lack of experimental evidence. In this respect we were recently able to show that serrated flow in metallic glasses is strongly correlated with a negative strain rate sensitivity (SRS, $m = \partial \ln \sigma / \partial \ln \dot{\varepsilon}|_{\varepsilon}$) and that the stress–strain curve becomes smooth and the SRS positive below a critical strain rate and temperature [30–32], analogous to the Portevin–LeChâtelier effect known for crystalline solids [33,34].

This paper focuses on the shear morphology of unfractured samples and, based on the experimental observations, discusses the possible influence of local temperature increases during shearing and before fracture. A constitutive equation accounting for the presence and absence of serrated flow as a function of temperature and SRS is presented in light of the microscopic findings. An estimate of the adiabatic heating and the spatial and temporal resolution during compression testing of macroscopic samples is also presented.

## 2. Materials and methods

$Zr_{57.9}Cu_{22}Fe_8Al_{12}Pd_{0.1}$ and $Cu_{50}Zr_{50}$ prealloys were prepared by arc melting the pure elements (purity >99.995%) in a Zr-gettered argon atmosphere from which cylindrical rods were suction cast into a copper mold having length of ~30 mm and diameters of 3 and 2 mm, respectively. A second batch of compression samples was produced from the same $Zr_{57.9}Cu_{22}Fe_8Al_{12}Pd_{0.1}$ prealloy using an electromagnetic levitation drop-casting technique. Compared to the alloy $Zr_{58}Cu_{22}Fe_8Al_{12}$ [35] the effect of Pd on the glass-forming ability and the amorphous state is not noticeable for the sample sizes studied here. Compression test specimens with a length- to -diameter ratio of 1.7 were cut from these rods and subsequently polished. The amorphous structure of the specimens was confirmed by means of X-ray diffraction (XRD) using a PANalytical X'pert diffractometer with Cu Kα radiation, and by differential scanning calorimetry (DSC) using a Setaram Labsys DSC. The deformed specimens were examined by scanning electron microscopy (SEM) using a LEO 1530 microscope equipped with a field emission gun. Constant cross-head displacement tests and strain rate jump tests in compression were performed on a four-column Schenck–Trebel machine equipped with a 100 kN load cell. Tests were performed at various cross-head velocities ranging from 1.0 to 0.1 mm $min^{-1}$, resulting in initial strain rates of $3 \times 10^{-3}$ to $3 \times 10^{-4}$ $s^{-1}$. The strain was measured from the cross-head displacement and a strain gauge positioned on the pistons above and below the specimen. Acquisition rates of 20–1200 Hz were used to provide information on the shear band velocity. Oxygen contents ranging from 200 to 300 ppm were measured using a LECO TC-436 oxygen analyzer with 80–100 mg samples. Transmission electron microscopy (TEM) experiments were performed with Philips CM20 and CM300 microscopes operating at 200 and 300 kV, respectively. TEM samples of the undeformed material were prepared by mechanical dimpling for ~30 min followed by ion milling with a 3 keV argon beam. One TEM sample cut from the deformed material of the

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