Approaching the ideal elastic limit of metallic glasses

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The ideal elastic limit is the upper bound to the stress and elastic strain a material can withstand. This intrinsic property has been widely studied for crystalline metals, both theoretically and experimentally. For metallic glasses, however, the ideal elastic limit remains poorly characterized and understood. Here we show that the elastic strain limit and the corresponding strength of submicron-sized metallic glass specimens are about twice as high as the already impressive elastic limit observed in bulk metallic glass samples, in line with model predictions of the ideal elastic limit of metallic glasses. We achieve this by employing an in situ transmission electron microscope tensile deformation technique. Furthermore, we propose an alternative mechanism for the apparent ‘work hardening’ behaviour observed in the tensile stress–strain curves.

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The familiar metals and alloys in our daily lives are all crystalline materials. In recent years, amorphous alloys, or metallic glasses (MGs), have emerged as a new category of advanced materials. A striking property of MGs is their high elastic limit far above that of their crystalline counterparts. At room temperature, most MGs exhibit uniaxial yield stress ($\sigma_y$) on the gigapascal level, and the corresponding yield strain ($\varepsilon_y$) is as large as, almost universally, ~2%. In comparison with common engineering materials, the superior strength in bulk MGs is known to be controlled by the relatively easy plastic deformation on the submicron scale, which is the dominant mode of plastic deformation at temperatures well below the glass transition temperature ($T_g$). Shear bands randomly nucleate from preferential sites inevitably present in the as-prepared bulk MGs (including casting pores/flaws, surface notches and other stress concentrators). Barring such ‘heterogeneous nucleation’ assisted by extrinsic factors, the $\sigma_y$ and $\varepsilon_y$ ought to be higher. Moreover, in the amorphous structure there are internal ‘defects’ or inherently more fertile sites for shear transformations, and their coordinated organization/evolution can lead to the formation of shear bands. This is analogous to the case of crystalline metals: normally there are pre-existing dislocations and easy sources for dislocation nucleation; in their absence plastic deformation can be delayed so much that the elastic limit can be pushed towards the ‘theoretical strength’, which is at least an order of magnitude higher than the commonly observed apparent $\sigma_y$. For example, it is well known that small volume single crystalline samples, such as whiskers, nanowires and nanopillars, offer the opportunity to observe ultrahigh strength and large elastic strains close to the theoretical limit. This is because only for such micro- and nano-scale samples can pristine crystals be made to minimize those defects that are inevitable in their bulk counterparts, such that nucleation of fresh defects is often required for yielding.

The same strategy can be applied to MGs, by using micro- and nano-scale samples. In such samples the extrinsic flaws that concentrate stresses and facilitate shear banding are eliminated. In addition, internal structural defects become low in population and small in size, and are thus less probable to launch cooperative actions. As a result, shear banding tends to be delayed. This should allow us to answer the question as to what the ideal strength (and elastic strain limit) is for MGs, and observe how close to it one can reach in experiments. This ultimate ceiling is of obvious interest for applications requiring high load-bearing capability and involving storage of elastic energy. Gaining quantitative and dynamical knowledge of the elastic behaviour on the submicron scale is, from a physics perspective, of paramount importance. In fact, information on elasticity at that length scale is perhaps more important than plasticity, as they are a direct reflection of the inherent structure of the glass.

In this work, we choose to dynamically examine MG samples with the effective sample size, $d$, in the 200–300 nm regime, to uncover the ideal elastic limit at room temperature. This $d$ range was chosen because, at larger sizes (for example, $d>500$ nm), the apparent strength would depend on $d$: $\sigma_y$, increases significantly with decreasing sample size and the ceiling has not been reached yet, as revealed by the tensile test inside a scanning electron microscope (SEM)\(^{11}\). When the sample sizes are extremely small (for example, $d<100$ nm), there can be major influences from surface conditions and surface diffusion, together with changes in the mode of plastic flow to include large elongation and necking\(^{11,12}\). This would make it difficult to compare with bulk samples and to determine sample cross-sectional area via postmortem SEM measurements. Previous experiments on submicron MG samples\(^{6,11,21}\) have focused on the mode of plastic deformation and the plastic strain achievable, not dynamic elastic strain measurements and elastic limit. In addition, the in situ transmission electron microscopy (TEM) tensile tests using ‘window-frame’ rather than free-standing samples, are complicated by confinements/constraints and the lack of quantitative stress–strain curves\(^{13,22}\).

**Results**

**In situ tensile tests.** For comparison with the known properties of bulk MG samples, we first present in Figure 1 the typical range of $\sigma_y$ and $\varepsilon_y$ observed so far in compression tests of MGs, particularly those based on Cu–Zr alloys (yellow shaded region)\(^{2,23–26}\). This sets the stage for presenting the extraordinarily high-elastic limits discovered in the following from the submicron-sized MG samples.

The material used for testing is a Cu$_{49}$Zr$_{51}$ MG, prepared using melt spinning. Figure 2a is a schematic showing how a tensile specimen was prepared using focused ion beam (FIB) micromachining. The sample was FIBed from the MG parent body, and the T-shaped free end (upper part in Fig. 2b) will be grabbed by the tungsten grip for tensile test. Figure 2c shows the two very small markers (indicated by white arrows) that define the gauge length. These markers were made by electron beam-induced carbon deposition in the FIB chamber. As detailed later, these fiducial markers are critical for reliably measuring the strain actually experienced by the gauge length. A TEM image showing the overall tensile test assembly is displayed in Figure 2d.

A total of seven specimens were tested using the Hysitron PI95 TEM PicoIndenter, see the Methods section for details. The sample evolution during the tests was recorded in movies (see an example in Supplementary Movie 1). The resulting engineering stress–strain curves are shown in Figure 3a. These curves show good linearity in the early stage of loading, which is confirmed via multiple loading, unloading and reloading cycles, such as those displayed in Figure 3b. Interestingly, this apparent elastic region goes well beyond the yield point known for bulk-sized samples (including ribsbons obtained by melt spinning) of this MG and similar MGs (the strength measured for bulk samples of this MG is in the range of 1.3–1.6 GPa, and

![Figure 1 | Strength and strain limit of Cu-Zr metallic glasses.](image-url)
2.3 × 10^{−3} s^{−1}. The first loading-unloading curve (red) exhibited completely linear elastic behaviour. Deviation from linearity was observed in the second loading cycle (dark blue) after the engineering strain reached ~3.6% (marked by a red star); -5% total strain was applied in the second loading cycle and the unloading curve was quite linear. Approximately 0.3% residual plastic strain was observed at the end of this loading cycle. The sample fractured in the third loading cycle (light blue) when the strain reached ~5.9%. The yield stress indicated by the red star in the third loading cycle and the unloading curve was quite linear. Approximately ~3.6% (marked by a red star); ~5% total strain was applied in the second loading cycle. For comparison, the curves for the second and third loading cycles have been shifted.

Additional elastic strain beyond proportionality limit. Also, as indicated in Figure 4a, there appears to be an additional elastic strain that was sustained in the sample, after the onset of yielding. This was measured as follows: after fracture ($\varepsilon_y = 5.2\%$), the sample gauge length was found to have retained 0.8% plastic strain, as detailed in Figure 4b-e using the still frames extracted from the acquired TEM movie (see Supplementary Movie 1). Therefore 4.4% of the total strain was recovered upon unloading, significantly higher than the $\varepsilon_y = 3.3\%$ shown in Figure 4a. In other words, after the stress–strain curve deviates away from linearity, part of the ensuing deformation is from additional elastic deformation. This extra elastic strain is 4.4–3.3% = 1.1%, and the (accompanying) plastic deformation is 5.2–4.4% = 0.8%. The observation that the unloading curve shown in Figure 3b (second loading cycle, dark blue colour) is quite linear suggested that nonlinear elastic strain may not have a major role in the total strain. No obvious crystallization was observed in the regions near the fractured surface; see the selected area diffraction pattern (inset in Fig. 4e).

Comparison with bulk values. The measured strain limits, $\sigma_f$ and $\varepsilon_f$, as well as the $\sigma_y$ and $\varepsilon_y$, have been added into Figure 1. On average, these data points indicate an impressive $\varepsilon_y$ of 3.5±0.3%, and $\sigma_f$ of 5.3±0.4% (this latter value contains a small plastic strain, for example, a fraction of ~1%, as discussed above). The strengths achieved for this material are unprecedented as well, except for confined conditions under nanoindentert27,28. For example, $\sigma_f$ is ~2.7 GPa, and $\sigma_y$ is of the order of 3.8 GPa (Fig. 4a). These properties are about twice as high as those observed for bulk samples of this MG (yellow region in Fig. 1).

Comparison with theoretical elastic limit. We next compare the measured properties with the theoretical elastic limit estimated from modelling. For bulk MG samples, Johnson and Samwer proposed an equation to describe the temperature-dependent shear strain limit, $\gamma_s = \gamma_0 - \gamma_1 (7T)^{\gamma_1}$, where $\gamma_s$ is the elastic strain limit in shear, $\nu$ is a scaling constant (2/3 in their model). Our comparison will be made at a typically used strain rate of ~10^{−3} s^{−1} and
T < T_g. In such a regime γ_C0 = 0.036 and γ_C1 = 0.016 are constants obtained by fitting experimental data of elastic limit \( \varepsilon \), which, as discussed earlier, were determined by shear band propagation in bulk samples (the blue curve in Fig. 5 represents this scenario). The same equation has recently been confirmed by analysis of atomistic calculations to be applicable for ideal elastic limit for yielding, but the fitting parameters were found to change to \( \gamma_C0 = 0.11 \) and \( \gamma_C1 = 0.09 \) (ref. 29). As MGs are isotropic and the Poisson’s ratio for our MG is \( \nu = 0.36 \) (ref. 23), the above equation can be used to obtain ideal strain limit in uniaxial loading using \( \varepsilon_y = \frac{T}{T_g} (1 + \nu) \):

\[
\varepsilon_y = 0.081 - 0.066 \left( \frac{T}{T_g} \right)^{\frac{1}{3}}.
\]

This prediction of ideal elastic limit is shown using the red curve in Figure 5 for the Cu–Zr MG. At room temperature \( (T = 300 K) \), and taking \( T_g = ~730 K \), the ideal \( \varepsilon_y \) of this MG is predicted to be 4.5%, and this corresponds to a \( \sigma_y \) of 3.7 GPa for \( E = 83 \) GPa. This point is also marked in Figure 1 (red solid circle).

Compared with the ~2% measured in bulk samples (see the room-temperature value on the blue curve in Fig. 5), the proportionality limit of ~3.5% observed in our 200–300 nm samples is closer to the ideal elastic limit of the MG. In addition, the \( \varepsilon_y \) of ~5.3%, when the small (less than 1%, Fig. 4) accompanying plastic strain is deducted from it, in fact gives a total elastic (reversible) strain that is very close to the theoretical prediction from equation (1), that is, 4.5% (see the room-temperature value on the red curve in Fig. 5).

Discussion

The small plastic strain involved between \( \varepsilon_y \) and \( \varepsilon_f \) is very likely related to the fact that our free-standing samples all have surfaces. A surface is an imperfection in itself that may eventually become the preferred site for the initiation of plastic flow, such that the theoretical \( \varepsilon_y \) of 4.5% may not be reached. In a molecular dynamics simulation, it was found that a MG with free surfaces yields at a strength ~15% lower than the same MG under periodic boundary conditions.

We note that the flow stress rises continuously with continued straining, as shown in Figure 3. Such behaviour was not observed in bulk samples and could be perceived as the material undergoing fast ‘work hardening’. A likely origin of this behaviour, we speculate, is the delayed shear band nucleation, due to the small sample size. Specifically, in a free-standing sample without pre-existing flaws, the measured \( \varepsilon_y \) (corresponding to the onset of plastic flow catalysed by the surfaces) would approach, but not reach, the ideal elastic limit. Different from a crystal, where dislocation nucleation is a highly localized event, a well-defined shear band from the surface in a free-standing MG may require a certain length scale to develop. As discussed by Shimizu et al. and Shan et al. such a length scale is of the order of 10\(^2\) nm, below which the groups of shear transformation zones (STZs) do not achieve the large aspect ratio needed for sufficient stress concentration to launch shear bands beyond their embryonic stages. As a result, our samples (200 nm ~ 300 nm) may not undergo shear banding immediately after the onset of STZ activities from some local regions (for example, softer regions at or near surfaces). The sample is still dominated by harder and confined regions that continue to deform elastically, long after some STZs have started changing their configurations in a small fraction of the sample volume to bend the stress–strain curve (contributing small plastic strains, of the order of a fraction of 1% before fracture, see discussions earlier). One typical example is shown in Figure 3b. Most of the preferential STZ sites used up are not recovered after unloading (for example, Fig. 3b, second loading cycle). Upon reloading, higher yield stress is required to activate harder sites gradually to carry plastic relaxation through shear transformations (for example, Fig. 3b, third loading segment). The bending of the curve involving harder and harder regions can thus be envisioned as a manifestation of the inherently non-equilibrium and non-uniform (different degrees of local order) glassy structure. The increasing stress (from

Figure 4 | Tensile behaviour and corresponding strain evolution of the d = 220 nm sample. (a) Engineering stress–strain curve from the tension test. The average strain rate was about 2\( \times 10^{-3} \) s\(^{-1}\). Mechanical properties extracted from the stress–strain curve include Young’s modulus (E), \( \varepsilon_y \), \( \varepsilon_f \), and \( \varepsilon_y \) (defined at the proportionality limit), fracture strength (\( \sigma_f \)) and total elongation to failure (\( \varepsilon_f \)), as well as the plastic strain (\( \varepsilon_{\text{plastic}} \)) remained in the gauge length. (b–e) These still frames extracted from the recorded movie (see corresponding points in a) demonstrate that the virgin sample (b) was elongated to a strain of \( \varepsilon_y = 3.3 \pm 0.2\% \) (c) when global yield began, and then fractured upon a total strain of \( \varepsilon_f = 5.2 \pm 0.2\% \) (d), among which \( \varepsilon_{\text{plastic}} = 0.8 \pm 0.2\% \) was measured from e, and the rest \( 4.4 \pm 0.2\% \) was the total recoverable elastic strain. These strains were determined with high accuracy because there were no rough/irregular fracture surfaces to reconnect back, as the fracture occurred outside the section between the two markers. No crystallization was observed near the fractured region, as further confirmed by the selected area electron diffraction pattern (inset of e) taken from the fractured area. The scale bar in b represents 200 nm, and the magnification was same for all the TEM images.
was milled into the plate, and the sample gauge was trimmed to approach the

\( \sigma_N \)

condition. To keep the cross-section of the sample gauge uniform in
table 30. Johnson, W. & Samwer, K. A universal criterion for plastic yielding of metallic

gaseous He at 30 kV/5 pA as the final cutting condition. To keep the cross-section of the sample gauge uniform in
to the thinning of both sides of the sample. An SEM

\( \varepsilon_f \)

molecular dynamics calculations.

\( \varepsilon_f \)

under uniaxial
to the tensile elongation of the sample were captured in situ

\( \varepsilon_f \)

in situ

\( \varepsilon_f \)

in microelectromechanical systems and nanoelectro-
micromechanical systems and nanoelectromechanical systems.

Methods

Sample preparation. The following procedure has been adopted to prepare the
tensile samples: first, a thin plate with dimensions of 400 nm (thickness) x 2.5 μm

\( \varepsilon_f \)

were exhaustively etched, the stress eventually becomes suf-

\( \varepsilon_f \)

is taken to be

\( \varepsilon_f \)

of the order of 700 K

\( \varepsilon_f \)

are being exhausted, the stress eventually becomes suf-

\( \varepsilon_f \)

the predicted ideal elastic strain limit in Fig. 3a. It is worth noting that the displacement data plotted in Supplemen-
tary Figure S2 also contained the contributions from outside the gauge part and

\( \varepsilon_f \)

to the tensile test. The

\( \varepsilon_f \)

to FIBed from its bulk parent body. Then a tensile sample

\( \varepsilon_f \)

of the strain rate is assumed to be 10\(^{-3}\) s\(^{-1}\) under uniaxial

\( \varepsilon_f \)

in the bulk parent body. Then a tensile sample

\( \varepsilon_f \)

was FIBed from its bulk parent body. Then a tensile sample

\( \varepsilon_f \)

stress, compared with bulk samples. Before reaching \( \sigma_f \),

\( \varepsilon_f \)

of the number of electrons in the sample should be identical. The variation in diameter

\( \varepsilon_f \)

of this MG is significantly higher than room temperature. Moreover, the electron

\( \varepsilon_f \)

that define the gauge length, strains were accurately determined by measuring the changes between the deposited carbon markers (see Fig. 2c and Fig. 4b-e), from the still frames extracted from the corresponding movies. For samples without markers, the uniform middle section of the sample is taken as the gauge length. Its change in the recorded movie was used to measure the strain. As the two ends in such gauges were not as well defined as in the marker cases, the strains determined would not be as accurate. For the determination of Young's modulus, we rely on the stress–strain curves (that is measuring the slope of their linear elastic portion) achieved from the samples with markers.

Electron beam effect. It should be noted that our experiments ruled out electron beam effects on the measured properties. For the comparative experiment on electron beam effect, three samples (see Supplementary Fig. S2) with identical projected geometry (along electron beam direction) were selected. Therefore, the gauge length of these three samples should be identical. The difference in strain is due to the thickness difference. Consequently, if there is any obvious beam effect, the stress versus displacement curves of these three samples should be different. However, as one sees clearly from Supplementary Fig. S2, the two samples tested with beam off displayed curves very similar to the one with beam on. In other words, the electron beam made no obvious difference for the sample size range we tested in this work. This is understandable, as the temperature rise in the sample is small for several reasons. First of all, the electron beam irradiating on the samples has a relatively low intensity. In addition, there is a large heat-conducting tungsten grip in intimate contact with the small sample. Also, the \( T_g \) of the order of 700 K of this MG is significantly higher than room temperature. Moreover, the electron beam effect on the metallic bonds in the MG is not expected to be as significant as that on the directional and localized covalent bonds. The possible contribution of beam-enhanced diffusion on surfaces is insignificant to alter the overall behaviour for the sample sizes used in this study. Therefore, for the beam-off samples, it is reasonable to convert the stress versus displacement curves to stress versus strain curves by assuming that their moduli remain identical to the sample with beam on (Fig. 2a). It is worth noting that the displacement data plotted in Supplementary Figure S2 also contained the contributions from outside the gauge part and therefore should not be used directly to calculate the strain. Otherwise, the strain would be overestimated.

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Author contributions
E.M. and Z.W.S. designed the project. L.T. carried out the tensile experiments and the data analysis. Y.Q.C. conducted the modelling. C.C.W. assisted in the tensile experiments. Z.W.S. and J.S. supervised the analysis and presentation of the tensile results. X.D.H. analysed the melt-spun MG, E.M. and J.L. benefited from an adjunct professorship at XJTU. J.L. also acknowledges support by NSF CMMI-0728069, DMR-1008104 and DMR-1120901, and AFOSR FA9550-08-1-0325. Y.Q.C. and E.M. were supported at JHU by US-NSF-DMR-0904188. X.D.H. was indebted to a 973 program of China (2009CB623700). We thank W.H. Wang’s group for providing the MG samples, and D.G. Xie and B.Y. Liu for useful discussions of the results.

Additional information
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