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The breakdown of strength size scaling in spherical nanoindentation and microcompression of metallic glasses

S. Wang^{a,*}, Y.F. Ye^a, Q. Wang^b, S.Q. Shi^c, Y. Yang^{a,*}^a Centre for Advanced Structural Materials, Department of Mechanical and Biomedical Engineering, City University of Hong Kong, Kowloon Tong, Kowloon, Hong Kong, China^b Laboratory for Microstructures, Institute of Materials Science, Shanghai University, Shanghai, China^c Department of Mechanical Engineering, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong, China

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ABSTRACT

It was previously reported that the strength of metallic glasses (MGs) would scale inversely with the size of a sample or a deformation field, commonly known as “smaller-being-stronger”. However, based on the extensive spherical nanoindentation experiments conducted across a variety of MGs, we demonstrate that such strength-size scaling breaks down at a critical indenter tip radius, which is caused by the transition of the yielding mechanism from bulk- to surface-controlled shear band initiation. Our experimental findings also provide an explanation for the unusual strength scattering observed in the micro-compression of MGs.

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It is well known that the strength of crystalline metals usually increases as their size decreases [1]. This phenomenon of strength size effect could be generally ascribed to the interplay between external sizes, such that those associated with sample dimensions or deformation field, and internal sizes, such as grain size and dislocation spacing [2–7]. To quantify the strength size effect in crystalline metals, different scaling relations were established over the past decades, such as $\sigma \sim D^{-n}$ for micro- and nano-pillar compression [1,3], where σ is the yielding strength, D the pillar diameter and n the material specific exponent; $H = H_0 \sqrt{1 + \frac{h^*}{h}}$ for nanoindentation [3,8,9], where H is the hardness for a given indentation depth h , H_0 the size-independent hardness and h^* the material specific length scale that also depends on the shape of an indenter. In theory, these scaling relations all stem from dislocations, the “plasticity carrier” in crystalline materials whose nucleation and movement are essentially size dependent [10–12].

Interestingly, metallic glasses (MGs) exhibit a similar size effect [13–29] despite the lack of a long-range crystalline structure and crystalline-like defects, such as dislocations. Many research groups [13–28] found that the strengths of MGs could increase relative to the bulk values in nanoindentation or micro- and nano-compression. To rationalize this size effect, a number of theories were proposed, such as Weibull statistics [14,19,26] and size-dependent shear band nucleation [27,30,31]. The recent molecular dynamics (MD) simulations [32] even showed

that, as the sample size reduces to the nanometer scale, the shear strength of a MG could rise up from the bulk value of about $\sim G/50$ (G = shear modulus) to an intrinsic strength limit of $\sim G/10$. However, the issue of strength size effect in MGs is not yet fully settled as shear banding is generally affected not only by the sample size but also by the sample shape [33] and stress state [34]. There were other experimental results obtained from micro- and nano-compression [35–41], which showed no significant change in the strength of MGs with the decreasing sample size. A similar finding was also reported by Packard and Schuh [42] from the spherical nanoindentation of a few kinds of MGs. Interestingly, it could be observed from the work of Packard and Schuh [42] that the hardness of the MGs slightly decreased rather than increased with the decreasing tip radius. This “inverse” size effect was then attributed to a possible surface effect [42,43].

To reconcile the seeming discrepancy among many previous findings, one possible explanation is that shear band nucleation in MGs is not only size dependent but also affected by a sample surface at the nanometer scale. Conceptually, this is analogous to homogeneous versus heterogeneous nucleation of shear bands as discussed recently in Refs [23,31]. Taking into account the possible surface effect that usually facilitates shear band nucleation, one may envision that bulk nucleation of shear bands in MGs would be “interrupted” by surface controlled shear band nucleation when the size of a sample or deformation field approaches a critical value as depicted in Fig. 1. Around this critical size, the usual scaling relation for the strength of MGs would break down with no obvious trend of size effect. Meanwhile, significant data scattering might appear around this critical size as the strength of MGs becomes very sensitive to the shear band nucleation site. At the

* Corresponding authors.

E-mail addresses: songwang4@cityu.edu.hk (S. Wang), yonyang@cityu.edu.hk (Y. Yang).

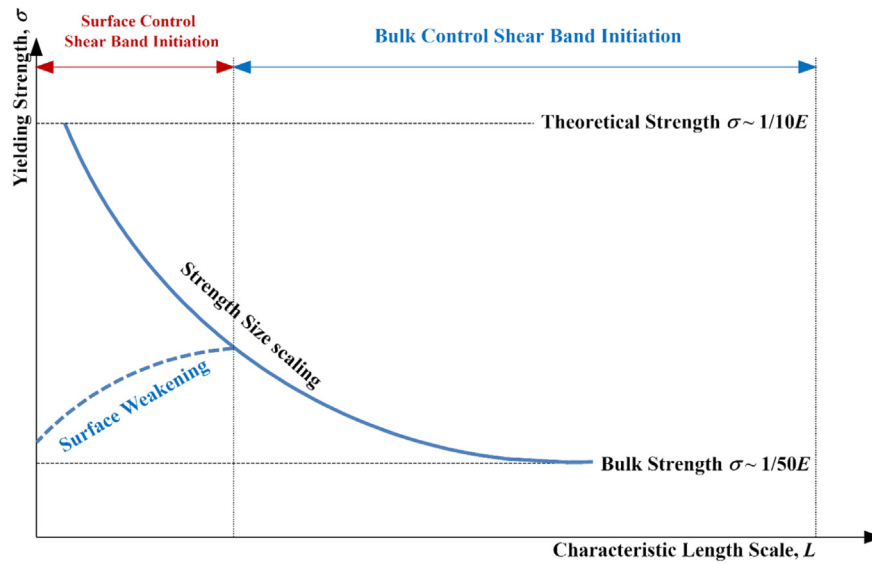


Fig. 1. The schematic illustration of the breakdown of strength-size scaling in metallic glass.

present time, however, there is no definitive answer to whether such a critical size exists and, if it does, what determines the critical size in different kinds of MGs.

To address the above issue, we chose six MGs, including $\text{Zr}_{52.5}\text{Cu}_{17.9}\text{Ni}_{14.6}\text{Al}_{10}\text{Ti}_5$, $\text{La}_{60}\text{Al}_{25}\text{Ni}_{15}$, $\text{Au}_{49}\text{Ag}_{5.5}\text{Pd}_{2.3}\text{Cu}_{26.9}\text{Si}_{16.3}$, $\text{Pd}_{40}\text{Cu}_{30}\text{P}_{20}\text{Ni}_{10}$, $\text{Fe}_{48}\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Er}_2$ and $(\text{Fe}_{44.3}\text{Cr}_5\text{Co}_5\text{Mo}_{12.8}\text{Mn}_{11.2}\text{C}_{15.8}\text{B}_{5.9})_{98.5}\text{Y}_{1.5}$ (in at.%), to study the possible break-down of the strength size scaling with spherical indentation. These alloys cover a wide range of mechanical/physical properties and their glassy nature was already identified by X-ray diffraction (XRD) (see Refs [27,44,45]) and the sample surface was mechanically polished to a mirror finish. Their basic mechanical properties, including elastic modulus and hardness, were first measured on the Hysitron™ NanoIndenter system (Hysitron Inc., Minneapolis, MN) with a Berkovich diamond tip (see Table 1). Subsequently, a series of spherical nanoindentation experiments were carried out at the constant indentation strain rate $\dot{\epsilon} = 0.2 \text{ s}^{-1}$ with the indenters of nine different radii R ($R = 0.1, 0.4 \mu\text{m}, 2 \mu\text{m}, 5 \mu\text{m}, 10 \mu\text{m}, 20 \mu\text{m}, 25 \mu\text{m}, 30 \mu\text{m}$ and $35 \mu\text{m}$). According to the Hertzian theory, we can extract the reduced modulus E_r by fitting the initial linear portion of the P - $h^{3/2}$ curve to $P = (4E_r R^{1/2} / 3)h^{3/2}$, where P is the indentation load and h the indentation depth. The results agree well with the Berkovich nanoindentation results. Following Ref. [31], the critical yielding load P_c is herein identified to be the point of departure of the P - h curve from the Hertzian solution. Here, it is interesting to note that, for the Zr-based MG in our study, the departure from the Hertzian curve appears abrupt and almost coincides with the point of the first pronounced displacement pop-in for the small indenters ($R \leq 2 \mu\text{m}$), which is consistent with the previous reports [42,46]; however, the point of departure turns into a gradual and smooth elasto-plastic transition for the large indenters ($R > 2 \mu\text{m}$). A similar phenomenon was also

observed in other MGs, which suggests that local plasticity should have set in before the first pronounced displacement pop-in in the indentation of MGs, particularly so for the indentation with the large indenters. In the literature [26,31,47], a similar behavior of incipient plasticity disconnected with the first conspicuous pop-in event was also found in various experiments and/or atomistic simulations. Theoretically, this could be attributed to the formation of constrained shear bands or the activation [31,48] of shear transformation zones (STZs) in the early stage of incipient plasticity [31,32,42,49].

Once E_r and P_c are obtained, the hardness H or critical pressure p_c for yielding under the indenter can be obtained as $H = p_c = (\frac{6PE_r^2}{\pi^3 R^2})^{1/3}$ for various indenter tip radius R and different MG compositions. As an estimation, the yield strength σ of the MGs is approximated as the measured hardness H divided by three [50,51]. Interestingly, regardless of the chemical composition, the yield strength σ exhibits a sharp and positive size effect when R is above a critical value while a negative size effect when below, as shown in Fig. 2(a)–(f). This critical indenter size is found to be $\sim 2 \mu\text{m}$ for the Zr-based MG but seen to vary with the chemical composition of the MGs studied [Fig. 2(a)–(f)].

To quantitatively understand the positive size effect, we first apply the scaling relation derived in our previous work [31] to fit the experimental data. By assuming that yielding is triggered by homogenous shear-band initiation [23–25,52,53], it can be derived $H = H_0 + 0.54E_r (l_c/R)^{3/2}$, where H is the size dependent hardness, H_0 the “bulk” hardness and l_c the critical size for homogenous shear-band initiation defined in Ref. [31]. Therefore, we obtain:

$$\sigma = \sigma_0 + 0.18E_r \left(\frac{l_c}{R} \right)^{3/2} \quad (1)$$

Table 1
The reduced modulus (E_r), bulk hardness (H_0) and the critical length scale (l_c) extracted from nanoindentation for various MGs; the Poisson's ratio (ν) for various MGs from literature.

Composition	E_r (GPa)	H_0 (GPa)	l_c (μm)	ν
$\text{Zr}_{52.5}\text{Cu}_{17.9}\text{Ni}_{14.6}\text{Al}_{10}\text{Ti}_5$	100 ± 2	5.4 ± 0.3	0.54	0.370 [59]
$\text{La}_{60}\text{Al}_{25}\text{Ni}_{15}$	49 ± 2	2.7 ± 0.2	0.44	0.325 [58]
$\text{Au}_{49}\text{Ag}_{5.5}\text{Pd}_{2.3}\text{Cu}_{26.9}\text{Si}_{16.3}$	97 ± 2	4.0 ± 0.3	0.45	0.406 [57]
$\text{Pd}_{40}\text{Cu}_{30}\text{P}_{20}\text{Ni}_{10}$	107 ± 3	5.7 ± 0.3	0.49	0.404 [56]
$\text{Fe}_{48}\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Er}_2$	177 ± 5	9.9 ± 1.3	1.7	0.305 [56]
$(\text{Fe}_{44.3}\text{Cr}_5\text{Co}_5\text{Mo}_{12.8}\text{Mn}_{11.2}\text{C}_{15.8}\text{B}_{5.9})_{98.5}\text{Y}_{1.5}$	222 ± 9	12.1 ± 1.5	2.1	0.33 [26]

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