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Ultrahigh-strength submicron-sized metallic glass wires

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In situ deformation experiments were performed in a transmission electron microscope to investigate the mechanical properties of submicron-sized $Pd_{40}Cu_{30}Ni_{10}P_{20}$ metallic glass (MG) wires. Results show that the submicron-sized MG wires exhibit intrinsic ultrahigh tensile strength of ~2.8 GPa, which is nearly twice as high as that in their bulk counterpart, and ~5% elastic strain approaching the elastic limits. The tensile strength, engineering strain at failure and deformation mode of the submicron-sized MG wires depend on the diameter of the wires.
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Metallic glasses (MGs) show good potential for applications as micro- and nanoelectromechanical devices because they possess excellent mechanical properties [1–5]. The utilization of MGs in micro- or nanotechnological systems requires a thorough understanding of their mechanical behaviour; in particular, strength and ductility are key issues. It was recently reported that the deformation mechanisms, and consequently the mechanical behaviour of crystalline materials, depend significantly on the sample size when the sample dimensions are in the micrometre and nanometre ranges [6–8]. Specifically, the tensile strength of single crystal metallic pillars has been shown to increase significantly with decreasing pillar diameter, which is attributed to dislocation starvation in small volumes [7,8]. However, this strengthening mechanism is not thought to operate in MGs due to their glassy atomic structure and the absence of lattice dislocations. Nevertheless, numerous investigations have demonstrated similar size effects on the deformation behaviour of MG pillar samples fabricated using the focused ion beam (FIB) technique [9–13]. For example, while the strength of MGs in the bulk form is controlled by shear band propagation, the strength of MGs with dimensions in the micrometre/sub-micrometre regime and in the nanometre range is determined by shear band nucleation and uniform shear transformation zones, respectively [11]. Further, when the dimensions of MG samples are smaller than 100 nm, a brittle-to-ductile transition occurs without compromising the strength of the material [14,15].

One-dimensional crystalline nanowires (NWs) have been widely and successfully used in nanodevices [16,17]. The mechanical properties of these crystalline NWs are significantly influenced by defects [18]. Theoretical predictions and experimental observations demonstrate that the surface structures of metallic and semiconducting NWs severely affect their elastic moduli [19,20]. In contrast, MGs do not possess any structural defects such as dislocations or grain boundaries, and this is widely thought to explain why they usually exhibit such good mechanical properties [1–4]. This, coupled with the stability of these properties, has stimulated much interest in exploring MGs as candidate materials for applications in nanoelectromechanical systems [21].

FIB milling has been the most successful technique used to date to fabricate nanoscale MG samples in order to investigate their mechanical properties. The Ga ion bombardment that takes place during the FIB sample preparation inevitably induces irradiation damage to

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the surface layer of the material, which significantly alters its mechanical behaviour [22,23], thereby making it difficult to determine the intrinsic mechanical properties at the nanoscale. Few alternative fabrication techniques that do not involve FIB have been used to produce nano- and submicron-sized MG samples, and very little is known about the mechanical properties of submicron-sized MG wires produced using such alternative fabrication techniques. This is an unsatisfactory state of knowledge because it suggests that we are yet to measure the true, intrinsic mechanical properties of these materials. In this work, we produced submicron-sized MG wires without FIB, and explored the mechanical properties of the submicron-sized MG wires using in situ tensile testing in a transmission electron microscope (TEM). We discovered that the ultimate tensile strength and engineering strain of MG wires depend heavily on their diameter. Reducing the diameter of such wires to ~340 nm almost doubles their tensile strength compared to that of bulk MG, with values as high as 2.8 GPa being recorded. Moreover, the reduced diameter of the wires leads to a dramatic increase in elastic strain, from ~2% in the bulk to ~5%.

Figure 1a shows typical $Pd_{40}Cu_{30}Ni_{10}P_{20}$ (at.%) MG wires with lengths of more than 200 μm. The wires appear as white lines on a dark silicon support. An enlarged image of the position indicated with an arrow in Figure 1a is presented in Figure 1b, revealing that the MG wires produced in this way have a homogeneous smooth surface structure. These microscopic images indicate that the structural quality of these submicron-sized MG wires is higher than that of the Zr-based MG NWs produced at the fracture surface of a bulk MG through a conventional mechanical testing process [24]. The very high structural quality of these MG wires further supports our objective to obtain reliable intrinsic mechanical property data from the MGs whilst avoiding the various testing-type artefacts arising from structural inhomogeneities and the FIB-induced surface defects that have occurred in previous MG NW studies [22,23,25,26].

Figure 2a is a scanning electron microscope (SEM) image of an MG wire of diameter ~1230 nm bridging the gap of a push-to-pull (PTP) module. The two ends of the wire were welded onto the module using Pt under an FIB. An enlarged view of the welded joint is provided in the inset SEM image, which clearly shows that the joint is sound and defect free. Figure 2b, which was extracted from Movie 1 in the Supporting information, shows that the fracture of the wire caused by in situ tensile deformation occurred via several shear banding events. The fracture angles relative to the wire axial direction (indicated by white lines in Fig. 2b) are about 55° and 45° at each side of the wire, which is a typical brittle fracture feature in BMG samples, as previously reported [27]. After retracting the applied external force, the two fractured ends of the wire are in contact owing to the reversible elastic deformation of the PTP module. However, the fracture is still visible, as shown in the circled position in the inset SEM image. Figure 2c presents the corresponding force–displacement curve, which combines the contributions from the PTP module and the wire. The force increased linearly up to ~2035 μN (as indicated by a black arrow), when kinking occurred. The kink corresponds to a crack initiation on the left side of the wire that approached the lower welding point, as shown in Movie 1. After the kink, the force continued to increase linearly until the wire fractured at ~2150 μN. Further pushing of the PTP led to an immediate drop in force down to ~150 μN, coupled with some force vibration induced by the detachment between the two parts of the wire, then a further drop to ~60 μN. After that, the force increased linearly again because of the stiffness of the empty PTP. The engineering stress–strain curve of the wire shown in Figure 2d was obtained by removing the contribution of the PTP module from Figure 2c and then converting the net force applied to the wire and the displacement into stress and strain, respectively. The tensile strength of ~1.75 GPa shown in Figure 2d is slightly higher than the 1.6–1.7 GPa reported for bulk Pd-based MGs [28,29]. There was no notable plastic strain in the wire.

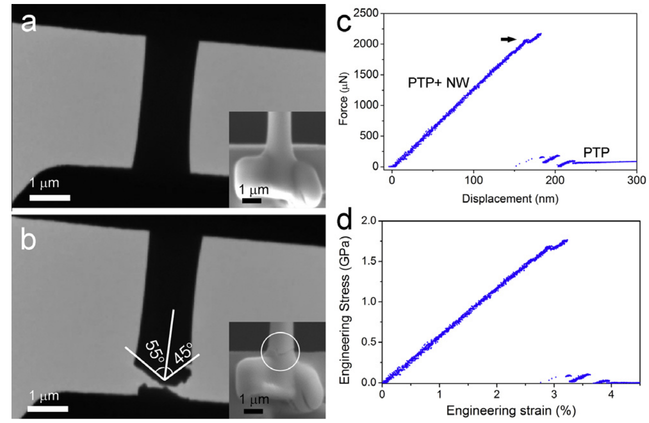

Fig. 2. (a) A TEM bright-field image showing a $Pd_{40}Cu_{30}Ni_{10}P_{20}$ MG wire bridging the gap of a PTP module. The inset SEM image shows one end of the wire before tensile straining. (b) A TEM image extracted from Movie 1 showing a brittle fracture caused by in situ straining. The inset SEM image shows the fracture after deformation. (c) The force–displacement curve from the combined contribution of the PTP module and the wire. The kink indicated by the black arrow suggests a crack initiation followed by fracture. (d) The engineering stress–strain curve for the wire after subtracting the contribution from the PTP module.

The mechanical behaviour of MG wires varies with their diameters. Figure 3 shows a typical mechanical behaviour of a submicron-sized MG wire with a diameter of 340 nm. The in situ tensile straining process was recorded in Movie 2 in the Supporting information and the wire images before deformation and after frac-

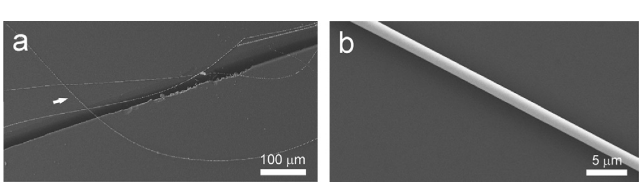

Fig. 1. (a) A SEM image of $Pd_{40}Cu_{30}Ni_{10}P_{20}$ metallic glass wires. (b) An enlarged image of the wire indicated by the white arrow in (a), revealing a high-quality wire with a homogeneous smooth surface structure, with no defects.

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