

Paradox of strength and ductility in metals processed by severe plastic deformation

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It is well known that plastic deformation induced by conventional forming methods such as rolling, drawing or extrusion can significantly increase the strength of metals. However, this increase is usually accompanied by a loss of ductility. For example, Fig. 1 shows that with increasing plastic deformation, the yield strength of Cu and Al monotonically increases while their elongation to failure (ductility) decreases. The same trend is also true for other metals and alloys. Here we report an extraordinary combination of high strength and high ductility produced in metals subject to severe plastic deformation (SPD). We believe that this unusual mechanical behavior is caused by the unique nanostructures generated by SPD processing. The combination of ultrafine grain size and high-density dislocations appears to enable deformation by new mechanisms. This work demonstrates the possibility of tailoring the microstructures of metals and alloys by SPD to obtain both high strength and high ductility. Materials with such desirable mechanical properties are very attractive for advanced structural applications.

In this work, we report on how inducing severe plastic deformation (SPD) by equal channel angular pressing (ECAP) and high pressure torsion (HPT)^{1–3} can produce both high strength and high ductility. Both ECAP and HPT can subject a metal work-piece to arbitrarily large shear strain under high pressure without changing the work-piece dimensions. Figure 2 shows schematics illustrating both methods. In ECAP, the work-piece is repeatedly pressed through the same die. For an ECAP die with an angle $\Phi = 90^\circ$ [Fig. 2(a)], each processing pass introduces a shear strain of 2 (or a von-Mises strain of 1.15). An important merit of ECAP is its potential to be scaled-up for industrial applications.³ The HPT technique imposes large shear strain through friction between the disk-shaped sample and a rotating plunger. To date it has only been applied to produce thin samples (≤ 1 mm). The pressure imposed on the sample is over 2 GPa in both techniques.

In this investigation, pure Cu (99.996%) was processed using ECAP with 90° clockwise rotations along the billet axis between consecutive passes,¹ while pure Ti (99.98%) was processed using HPT. All processes were performed at room temperature.

Strength and ductility were measured by uniaxial tensile tests performed using samples with gauge dimensions of $5 \times 2 \times 1$ mm. Resulting engineering stress–strain curves

are shown in Fig. 3. Results for Cu tested at room temperature in its initial and three processed states are shown in Fig. 3(a). The initial coarse-grained Cu, with a grain size of about $30 \mu\text{m}$, has a low yield stress but exhibits significant strain hardening and a large elongation to failure. This behavior is typical of coarse-grained metals. The elongation to failure is a quantitative measure of ductility, and is taken as the engineering strain at which the sample broke. Cold rolling of the copper to a thickness reduction of 60% significantly increased the strength [curve 2 in Fig. 3(a)] but dramatically decreased the elongation to failure. This is consistent with the classical mechanical behavior of metals that are deformed plastically.^{4,5} This tendency is also true for Cu subjected to two passes of ECA pressing [curve 3 in Fig. 3(a)]. However, further deforming the Cu to 16 ECA passes simultaneously increased both the strength and ductility [curve 4 in Fig. 3(a)]. Furthermore, the increase in ductility is much more significant than the increase in strength. Such results have never been observed before and challenge our current understandings of mechanical properties of metals processed by plastic deformation.

Similar results were also observed in Ti samples subjected to HPT, which were tested in tension at 250°C . The coarse-grained Ti with a grain size of $20 \mu\text{m}$ exhibits

a low strength and a large elongation to failure [curve 1 in Fig. 3(b)]. After being processed by HPT for 1 revolution, the Ti material had a very high strength but significantly decreased ductility. Further HPT processing to 5 revolutions dramatically increased the ductility and slightly increased the strength [curve 3 in Fig. 3(b)].

Figure 3 shows that small SPD strains (2 ECA passes or 1 HPT revolution) significantly increase the strength at the expense of the ductility, while very large SPD strains (16 ECA passes or 5 HPT revolutions) dramatically increases the ductility and at the same time further increases the strength. This is contrary to the classical mechanical behavior of metals that are deformed plastically. Greater plastic deformation by conventional techniques such as rolling, drawing or extrusion introduces greater strain hardening, which in turn increases the strength, but decreases the ductility of the metal.^{4,5} Two representative Cu samples failed under tensile tests are shown in Fig. 4. The Cu sample processed by ECAP for 2 passes [Fig. 4(a)] shows visible necking near the fracture section. It failed at a relatively low strain [see curve 3 in Fig. 3(a)]. In contrast, the Cu sample processed by ECAP for 16 passes [Fig. 4(b)] shows no apparent necking near the fracture section, which explains its high ductility [see curve 4 in Fig. 3(a)]. Both samples show cross-section reduction over the length of the gauge section. The Ti samples (not shown here) demonstrated

similar trend. The reduction in sample cross-sectional area is 51% for Cu (16 ECAP passes) and 43% for Ti (5 HPT revolutions). The strain-rate sensitivity of stress, defined as $m = (\partial \ln \sigma / \partial \ln \dot{\epsilon})_{\epsilon}$, where σ is flow rate, $\dot{\epsilon}$ is strain rate, were measured using the standard jump-test method.⁶ The samples with high ductility were found to have higher strain rate sensitivity. For instance, the value m was equal to 0.14 for ECAP Cu (16 passes) in contrast to $m = 0.06$ for ECAP Cu (2 passes). Higher strain rate sensitivity renders the materials more resistant to necking.^{7,8}

The extraordinary mechanical behavior in metals processed by SPD suggests a fundamental change in deformation mechanisms after the metals have been processed by SPD to very large strains.

We can begin to understand these results by examining the microstructures induced by SPD. Figure 5 shows the transmission electron microscopy (TEM) micrographs of (a) Cu processed by ECA pressing for 16 passes and (b) Ti processed by HPT for 5 revolutions and then heated at 250 °C for 10 min. As shown for both Cu and Ti, there

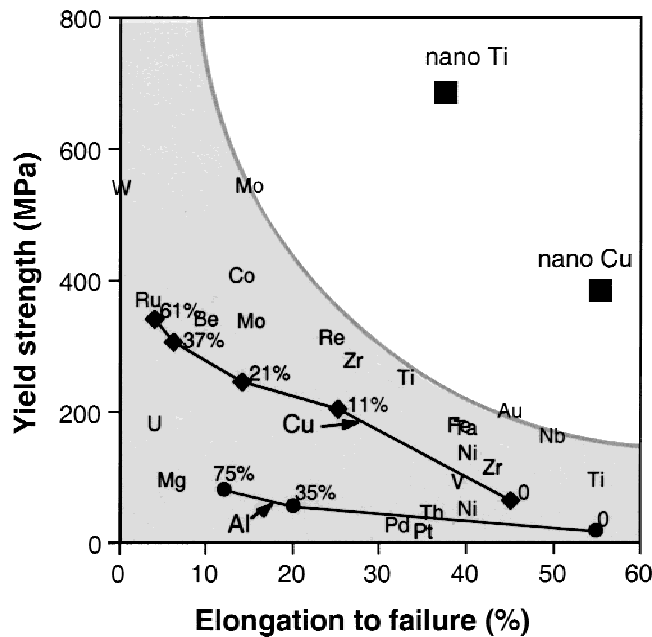


FIG. 1. Cold rolling (the reduction in thickness is marked by each data point) of Cu and Al increases their yield strength but decrease their elongation to failure (ductility).^{4,5} The extraordinary combination of both high strength and high ductility in nanostructured Cu and Ti processed by SPD clearly sets them apart from coarse-grained metals. (These metals are 99.5% to 99.9% pure. Metals with different impurity contents exhibit different strength and ductility values.)⁴

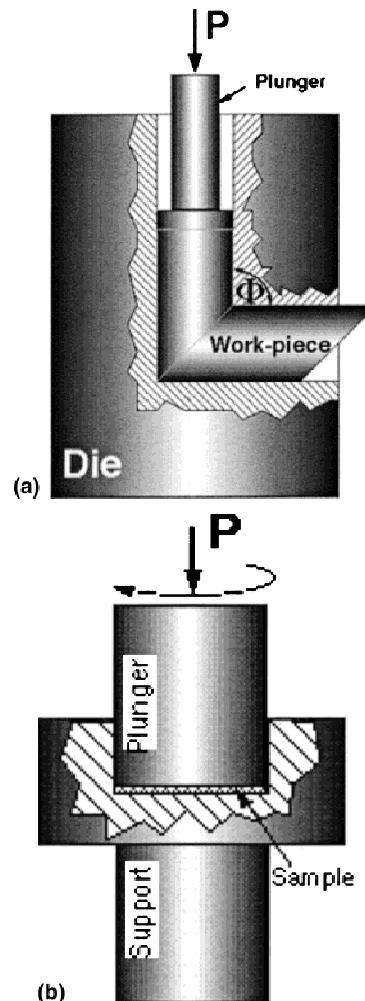


FIG. 2. Schematics of severe plastic deformation techniques: (a) equal-channel angular pressing and (b) high-pressure torsion.

is a formation of ultrafine-grained structure having a mean grain size of about 100 nm. Most grains are equiaxed without noticeable elongation. Selected-area electron diffraction indicates that a significant fraction of

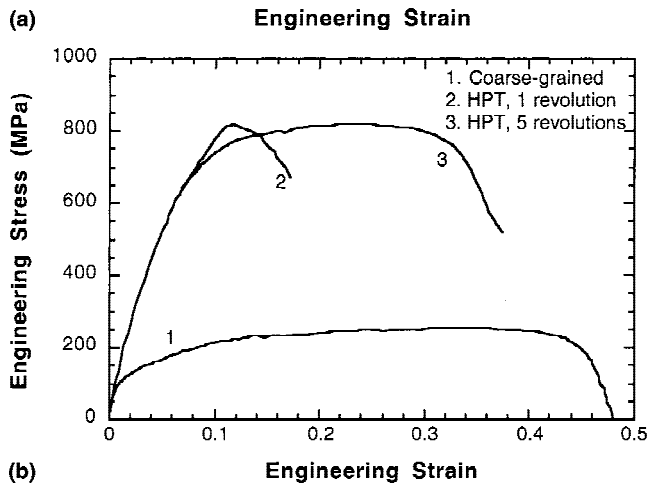
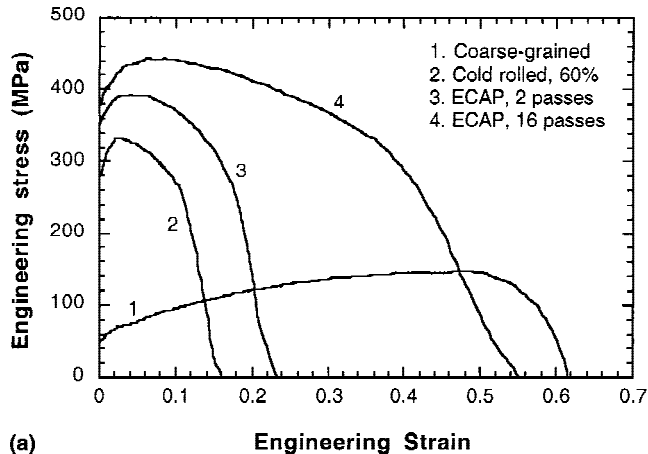


FIG. 3. Tensile engineering stress-strain curves of (a) Cu tested at 22 °C and (b) Ti tested at 250 °C. Both were tested at a strain rate of 10^{-3} s^{-1} . The processing conditions for each curve are listed on the figure.

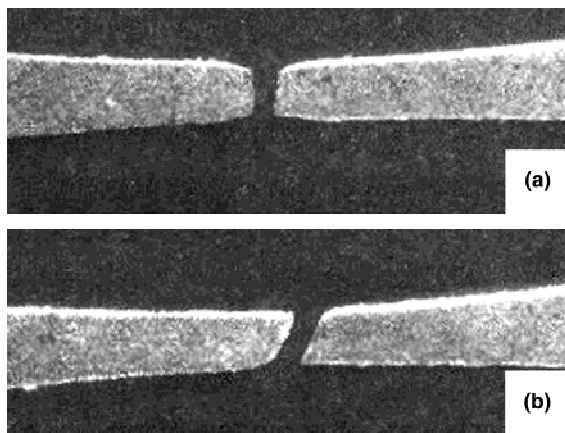


FIG. 4. Representative Cu samples pulled to failure at room temperature. The samples were processed by ECAP for (a) 2 passes and (b) 16 passes.

grains are highly misoriented. This is consistent with the recent TEM/high-resolution electron microscopy observations that large severe plastic deformation mostly introduces high angle grain boundaries.¹ In addition to creating ultrafine grains, the SPD process also introduces high densities of dislocations and internal elastic strains.¹ The grain boundaries are usually in a nonequilibrium state, as evidenced by their distorted shape (Fig. 5). As a comparison, conventional deformation techniques such as rolling usually cause the formation of low-angle subgrain structures (cell blocks) in metal,^{9,10} although some low- and high-angle grain boundaries may form with sufficient strain. The subgrains/grains are usually elongated (or filamentary) and larger than the grains shown in Fig. 5.

Note that not all SPD-processed metals exhibit the high ductility as observed in this work. For example, commercially pure Ti processed by ECAP for 8 passes has only 14% elongation to failure.¹¹ We believe that for a metal to exhibit a combination of extraordinary

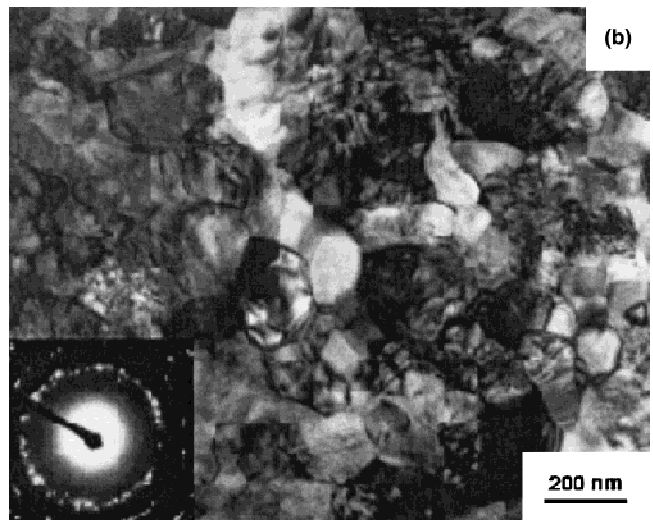
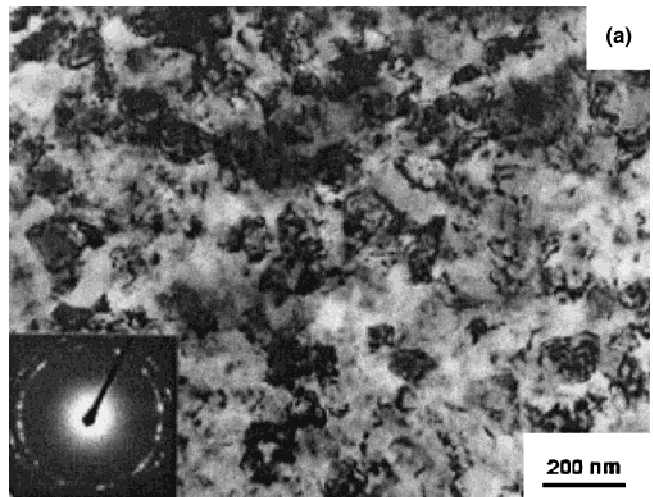


FIG. 5. TEM micrographs of (a) Cu after ECA pressing (16 passes), and (b) Ti after HPT (5 turns) and being heated at 250 °C for 10 min.

ductility and high strength, it has to be severely deformed beyond a certain level of strain. Increasing strain decreases grain size, but only to a minimum size that appears to depend on the SPD processing conditions.¹² However, after the grain size saturates, the fraction of high-angle grain boundaries continues to increase, and the microstructure becomes more homogeneous with further SPD straining.^{1,13} We also believe that the high pressure applied during SPD may play a role in the formation of nanoscale microstructures shown in Fig. 5. It has been reported that higher pressure during HPT produces finer grains.¹⁶

For coarse-grained metals, dislocation movement and twinning are the primary deformation mechanisms. Ultrafine, equiaxed grains with high-angle grain boundaries impede the motion of dislocations and consequently enhances strength. At the same time, these grains may also facilitate other deformation mechanisms such as grain boundary sliding and enhanced grain rotation,¹⁴ which improves ductility. A recent report attributed the room-temperature superplasticity of electrodeposited Cu with a grain size of 28 nm to grain-boundary sliding.¹⁵ In addition, the grain boundaries generated by SPD are usually in a nonequilibrium state, with many dislocations that are not geometrically necessary to form the grain boundary.^{1,16} These dislocations, as well as dislocations piled up near the grain boundaries, could move to facilitate grain boundary sliding¹⁷ and grain rotation, and therefore increase the ductility. Significant grain boundary sliding was experimentally observed in ultrafine-grained copper deformed at room temperature.¹⁴ The enhanced strain rate sensitivity observed in this work also indicates an active role of grain-boundary sliding.⁶ On the other hand, a possible change of the deformation mechanisms from dislocation slip (and also twinning in Ti) to grain-boundary sliding in SPD metals may explain the unusual stress-strain curves of Cu and Ti (Fig. 3). This prospect is supported by the strain rate sensitivity measurements. Deformation entirely by grain boundary sliding would ideally result in a strain rate sensitivity of 0.5 whereas conventional dislocation slip typically results in strain rate sensitivities less than 0.1. The higher values of m observed experimentally suggest that a mixture of grain boundary sliding and crystallographic slip occurs in nanostructured metals.

This work demonstrates the possibility of tailoring microstructures by SPD techniques to produce ultrafine (or nanostructured) metals and alloys that have a combination of high strength and high ductility. Figure 1 shows that the trend of higher strength accompanied by

lower ductility is not only followed individually by the work-hardened Cu and Al, but also followed collectively by 21 other coarse-grained metals. The nanostructured Cu and Ti are clearly separated from coarse-grained metals by their coexisting high strength and high ductility. Further more, the ECAP technique has the potential to provide nanostructured materials in sufficiently large product forms to enable their use in advanced structural applications.

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