Eight Routes to Improve the Tensile Ductility of Bulk Nanostructured Metals and Alloys

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Nanostructured metals and alloys are under intensive research worldwide and being developed into bulk forms for application. While these new materials offer record-high strength, their ductility is often inadequate, sometimes rendering them unusable. This article reviews recent progress in tailoring the nanostructure to achieve coexisting high strength and high ductility at room temperature. The focus is on a summary of the strategies currently being pursued as well as the outstanding issues that await future research.

INTRODUCTION

The past decade has witnessed a surge in research and development worldwide to drive the grain sizes of bulk metals and alloys down into the so-called nanostructured (NS) regime.[1] Here NS materials are loosely defined as polycrystalline solids with property-controlling microstructural features on or below the 100 nm level. Typical processing routes include severe plastic deformation (SPD),[2,3] which decomposes the grains through dislocation accumulation and reorganization (polygonization), electroplating under deposition conditions that encourage copious nucleation but little growth of the grains,[4] and consolidation of nanocrystalline powders.[5,6] One of the main motivations for such a push for the very small grain sizes stems from the expectation of unprecedented mechanical strength, as predicted by the well-known Hall-Petch relationship that projects a continuous rise of strength with decreasing grain size.[7] Experimental data accumulated so far indicate that high strength, five to ten times that of the conventional coarse-grained counterpart, is almost a given for the NS materials produced.[7,8] But their ductility, particularly uniform elongation in tension, has been rather low and in most cases nowhere close to that of the normal metals.[9,10] As pointed out in previous reviews, this drawback could be an insurmountable hurdle in bringing bulk NS materials from laboratory to commercialization.[9,10]

Compared with conventional materials, the engineering of grains on the nanoscale offers extra room for microstructural manipulation and optimization.

What has been new and exciting in the past several years is a series of breakthroughs in devising strategies that could impart decent tensile ductility while keeping the majority of the strengthening gained by nanostructuring (e.g., References 11–16). This came with the realization that one could tailor the NS grains, via various processing routes, to arrive at improved mechanical properties. In fact, compared with conventional materials, the engineering of grains on the nanoscale offers extra room for microstructural manipulation and optimization.[11–15] Unusual deformation mechanisms that accompany the nanoscale grain structures are also helpful if harnessed properly.[16–19] These have led to not only unprecedented combinations of high strength and ductility, but also impressive fatigue properties and superplastic forming advantages.[1,2] The discussions in this article focus on the microstructural designs that affect strength and ductility, while information regarding other properties can be found in several other recent overviews.[1,2,19,20]

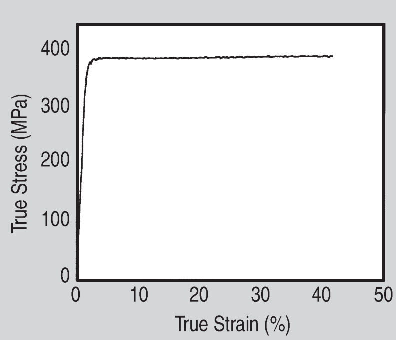

Figure 1. The compressive true stress-strain curve of a nanostructured copper processed via equal channel angular pressing.[16] The grain size is ~200 nm. Note the low strain hardening rate.

THE DUCTILITY CHALLENGE

For some applications, a sufficiently large reduction of the cross-sectional area before fracture is adequate. This is often available in bulk NS metals processed via SPD.[21,22] In many other cases, an appreciable uniform elongation under tensile stresses is required, but is rarely available in a monolithic NS material with nearly monodispersed grain size.

The reasons for the inadequate ductility have been discussed before, including a discussion on instabilities and ductility by this author in a viewpoint article in *Scripta Materialia*.[10] Briefly, strengthening at the expense of ductility is not uncommon, and it is not surprising for a high-strength material such as an NS metal/alloy to be prone to instabilities upon (large) plastic deformation. As a simple example, extensive tensile elongation is not expected for an ultra-strong material that has no capability to sustain a sufficiently high rate of strain hardening. The latter is in fact typical for NS metals, as shown, for example, in Figure 1. In this figure, an NS copper, in this case processed via SPD, exhibits an insignificant work hardening rate in a conventional compression test.[16,23] Such a material would start necking soon after yielding, leading to a plunging tensile test curve almost from the outset. Examples have been shown elsewhere (e.g., Reference 10. Note that due to space limitations, this article will only cite references without repeating the figures and plots that are already accessible in the literature).

Therefore, a key to meeting the ductility challenge is to use stabilizing mechanisms to overcome the instabilities that threaten the tensile elongation of NS metals and alloys.[10,16] This article is a brief overview of the eight different approaches that have been attempted, and demonstrated to various degrees, to reach this goal. The stabilizing strategies that the various research groups have come up with to combat the unstable deformation are, for the most part, rooted in two sub-categories of mechanisms: strain hardening and strain-rate hardening.[16] These, as in conventional metallic materials, prevent the concentration of plastic deformation in local regions that would otherwise experience excessive deformation to induce failure.

Although some success stories are cited in this article, it should be recognized that in general these strategies have not yet been fully established for NS materials, nor carefully analyzed to satisfactory sophistication. Only some preliminary demonstrations have been reported. There are numerous opportunities for future investigations. In fact, one of the objectives of this overview is to stimulate interest and encourage activities in this area by outlining open questions that should be addressed with in-depth research.

EIGHT ROUTES TO ENHANCED DUCTILITY

The presentation of the eight approaches begins with a seemingly straightforward idea, which, at first glance, may be conceived as merely sacrificing some strength for ductility (i.e., a simple trade-off between strength and ductility). For a single-phase material, such a trade-off is well known; for example, cold working increases the strength of copper, but reduces its ductility.[11,12] One could consider backing off in percent cold work or resorting to not-so-nano grain sizes, to exchange for better tensile elongation. In the first approach, the author's group demonstrated that, by mixing up the length scales, specifically by creating a bimodal (or multi-modal) grain size distribution, one could achieve simultaneously good yield strength and fairly large uniform (and total) elongation.[12]

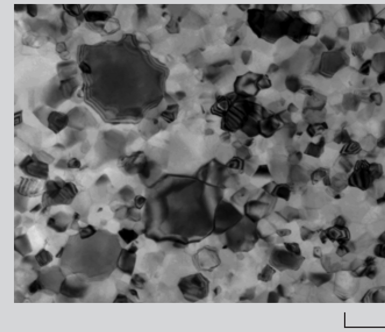

Figure 3. The bimodal grain sizes developed in-situ during tensile straining of a nanocrystalline aluminum film. The as-deposited film had a uniform grain size distribution with an average grain size of ~30 nm (Courtesy of D.S. Gianola et al.).

What turned out to be interesting is that one could do better than just a simple trade-off. When the bimodal structure was created on the nano-micro-scale, a large gain in work hardening and uniform strain was achieved, with only a (relatively) small loss of strength. The extra strain hardening ability may have something to do with the microstructural length scales involved, which are in this case close to the characteristic (or intrinsic) length scale of a material for strain-gradient plasticity to play a significant role. The storage of geometrically necessary dislocations required for compatible plastic strains may be pronounced for the unusually large strain gradient produced,[9,12] which is depicted in Figure 2. Also possibly contributing is the twinning activities triggered by stress concentrations in a highly non-uniform grain structure deforming at a high flow-stress level.[16,24]

This approach has advantages: the idea is simple and easy to grasp, the grain structure can be induced through traditional thermomechanical means (e.g., through recrystallization and secondary recrystallization) in a bulk sample, and a bimodal grain size distribution can even be produced by consolidating a simple mixture of powders of pre-selected, different grain size.[25] Also note that a functionally gradient material with a combination of good strength and ductility is beneficial for fatigue performance.[2]

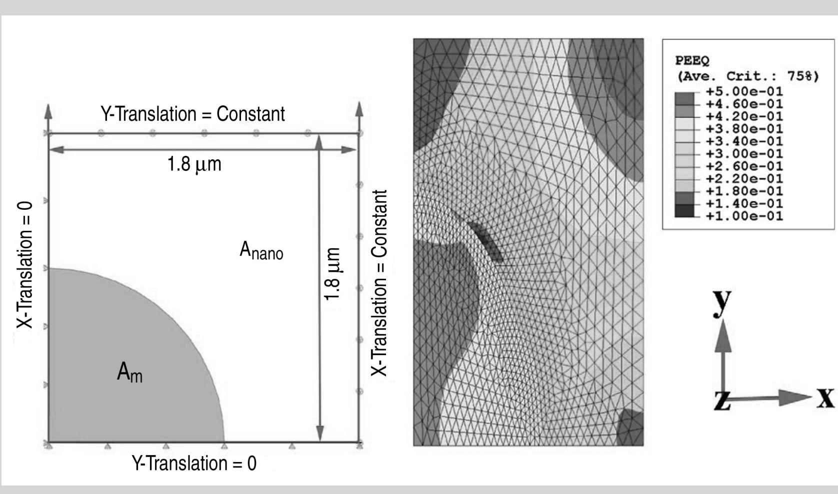

Figure 2. The finite-element method modeling of the bimodal nanostructured copper:[12] (a) micrometer-sized grains embedded inside a sea of nanocrystalline grains. Note the heterogeneity is on a length scale (1 to 2 micrometers) that, according to the strain gradient plasticity theory, would lead to strong strain gradient hardening effects (geometrically necessary dislocations) for copper. (b) The large strain gradient observed across this characteristic length scale. More details are in the on-line supplemental material of Reference 12.

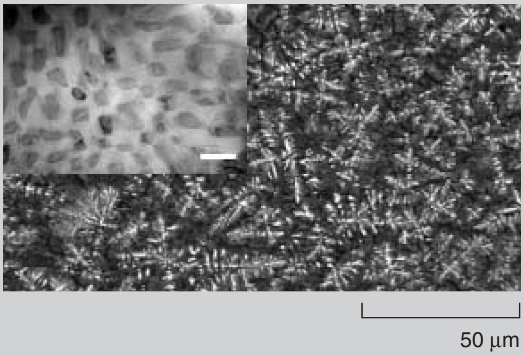

Figure 4. A scanning-electron microscope image of an in-situ composite formed via liquid casting of a titanium alloy.[27,28] The matrix has a rod eutectic microstructure with eutectic spacing less than 100 nm (see the TEM micrograph in the inset; the white scale bar represents 100 nm). Embedded in the matrix are dendritic primary body-centered cubic titanium solid solution micrometers in size.

There are also inconveniences: the exact distributions of the grain size, grain shape, and spatial locations depend on many processing parameters and may be difficult to reproduce each time, and the overall material response hence becomes tricky to predict/model. The outcome properties therefore span a relatively wide range depending on the microstructural variations. Note that abnormal grain growth is not uncommon for NS materials and can even be stress-induced in-situ during low-temperature deformation. For example, a recent tensile test of an NS aluminum showed ductile behavior; the large tensile elongation turned out to be the result of a bimodal grain distribution (Figure 3) grown out of an initially uniform NS grain structure upon loading.[26]

Industrial applications usually employ multiphase alloys rather than simple elemental metals. In this case, the parallel to the described bimodal microstructure approach is the second route to ductility: a mixture of two or multiple phases with varying size scales and properties. This is then the familiar composite idea. Figure 4 shows such a composite of micrometer-sized ductile phase embedded in an ultra-strong (but brittle) eutectic product with NS spacing.[27,28] This composite microstructure was formed in-situ during casting (solidification) of the corresponding liquid titanium alloy. As perhaps expected, the microstructure shown in Figure 4 simultaneously provided impressive strength due to the large number of interfaces, a high strain-hardening rate due to the dislocation accumulation in the micrometer-sized dendrites, and large plastic strains due to the large number of slip bands and profuse dislocation activities.[27,28]

The third approach is to use nanoscale growth twins in lieu of the nanograins for strengthening. The reasons for doing so were discussed recently.[14,29] It has been argued that a coherent twin boundary, while not much of a defect in terms of interface (grain boundary) energy, is very effective in blocking dislocations to require high stresses for slip transmission across this special grain boundary. Meanwhile, the twin boundaries do not encourage dynamic recovery as general high-angle grain boundaries or dislocation cells do. A transmission-electron microscopy (TEM) picture of such a nano-twinned copper after tensile deformation is shown in Figure 5. It shows that dislocations accumulated in regions where the twin spacing is large, the thin twin ribbons were cut into pieces by dislocations tangles, and the twin boundaries had large numbers of dislocations and eventually became dislocation sources. In other words, the originally low defect content (with only coherent, low-energy twin boundaries) saved room for dislocation storage to further strengthen the material upon tensile straining.[29]

The fourth approach involves dispersions of nanoparticles and nano-precipitates, which are widely used in engineering alloys. Precipitation hardening is in fact the most potent strengthening method for many alloys. In NS grains, especially those on the larger side (on the order of 100 nm), the hard precipitates also initiate, drag, and pin dislocations such that dynamic recovery is reduced. The result is a significant dislocation storage required for compatible plastic strains,[9] allowing a high strain-hardening rate that leads to larger uniform strains while elevating strength. To promote the precipitation of a large population of nanoparticles, SPD is often involved in the processing to pump in extra defects to increase nucleation sites. Several groups are exploiting nanoscale second-phase precipitates in acquiring adequate ductility for ultrafine-grained steels and aluminum alloys.[30–35] The authors' recent attempt in a 2024 aluminum alloy corroborates that this approach is effective,[35] as reflected by the tensile properties shown in Figure 6. Note that a new concept is advocated here: starting from an NS alloy, one can get decent ductility without sacrificing strength at all. In fact, the strength can be made higher due to precipitation (ageing), even though this processing step partly recovers the grains and improves the ductility. Other dual-phase microstructures, such as those in steels[36] involving ultrafine martensites, are also advantageous in offering a good combination of strength and ductility.

The fifth approach, using transformation-induced plasticity (TRIP) and twinning-induced plasticity (TWIP), is well established for conventional metals and alloys. It seems that TRIP is operative in NS or at least ultrafine grains a few hundred nanometers in size,[37] albeit at relatively high flow stresses. In tensile deformation, martensitic transformation was reported in an ultrafine steel, Fe-0.1%C-10Cr-5Ni-8Mn, at a rate similar to that in its coarse-grained counterpart so that the strain-hardening rate is also almost identical.[38] Twinning-induced plasticity in some materials of low stacking fault energy is also likely.[39]

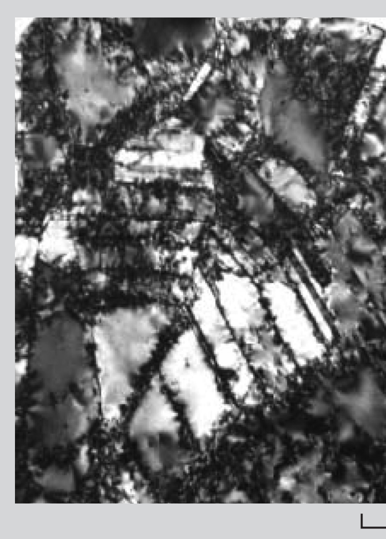

Figure 5. A TEM micrograph of an electrodeposited copper with a high density of growth twins. After plastic deformation a large number of dislocations are stored in the microstructure (courtesy of L. Lu).

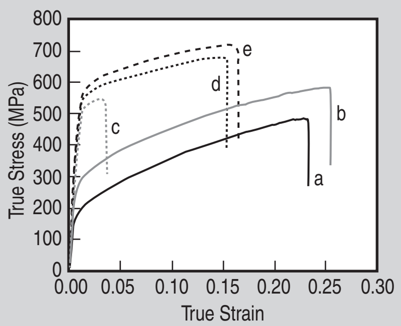

Figure 6. The tensile true stress-strain curves of the 2024 Al alloy after different processing (S. Cheng and E. Ma, unpublished): a–493°C solution treatment (ST); b–ST and then aged at 160°C for 10 h; c–ST + cold rolling at room temperature (CR-RT); d–ST + CR-RT, followed by aging at 160°C for 10 h; e–ST + liquid-nitrogen-temperature rolling + 100°C aging for 100 h. Note the low ductility of curve c, and the coexisting high strength and uniform ductility after aging (curves d and e).

Electroplated NS-cobalt showed a tensile ductility similar to that of conventional cobalt.[4] The author's group also tested such a cobalt, which showed both high strength and good ductility, as shown in Figure 7. A very high density of extremely thin twins, often only two to several atomic layers thick (Figure 7), is observed in the tensile-tested sample. Some mechanical twinning and twin boundary migration may be ongoing to contribute to strength and strain hardening, although the majority of these observed twins must be growth twins formed during deposition of this metal of low stacking-fault energy. The propensity of the martensitic transformation and deformation twinning in very small NS grains is being debated at present.[24] Prior experience with conventional materials at room temperature point to the other direction: both martensitic transformation and twinning would be difficult when the grains become very small.[1,40]

In addition to tailoring the microstructure, one could also change deformation conditions to encourage strain hardening mechanisms during deformation. The sixth approach, lowering of dynamic recovery at low-temperature (cryogenic temperatures) and/or dynamic strain rates, is known for coarse-grained face-centered cubic (fcc) metals and has been demonstrated to be applicable for NS fcc metals produced via SPD.[16,41,42]

All the described approaches, in one way or another, improve strain hardening. The alternative is the seventh approach, which is to improve strain rate hardening, as known from the Hart's instability criterion.[16] A material with a strain rate sensitivity, m, of the order of unity could be superplastic. For fcc metals, going to the NS grain size does increase the strain rate sensitivity,[43,44] especially at slow strain rates where grain boundary mechanisms help mediate deformation.[16,41] The authors have used this idea to rationalize the near-perfect plastic behavior observed in copper over a range of nearly uniform strains, in absence of strain hardening.[16,45] Several reports also suggest that after many SPD rounds, copper, titanium, and aluminum alloys appear to show enhanced ductility.[11,19] The reasons are yet to be fully clarified, but the refined grain sizes and the highly non-equilibrium grain boundary structures may be promoting grain boundary deformation mechanisms,[11] which offer a relatively high m. As a result, uniform tensile deformation is stabilized to relatively large strains. At room temperature, NS metals and alloys are usually not superplastic, except perhaps for NS-zinc[20,46] and SPD-processed magnesium for which room temperature is already a high homologous temperature. A copper had a high m after 16 SPD passes but it is an exception to all the other NS copper tested so far.[11,47]

All of the described approaches assume bulk materials free of flaws such as porosity, which obviously can ruin ductility prematurely and completely.[48] Porosity in consolidated NS materials may also lower strength[49] and help initiate shear localization; this may be partly responsible for the high propensity for shear banding in irradiated alloys or consolidated NS metals.[50–53] Therefore, for consolidated NS materials, the paramount requirement, or the foremost strategy, is the eighth approach: to strive for truly flaw-free materials. Very recently, there was a breakthrough in obtaining NS copper of such quality at least for pieces on the order of 1 cm in diameter.[8,15,54] The copper was in-situ consolidated to full density from ball-milled NS powders, rendering a final average grain size of only 23 nm and a narrow grain size distribution. This copper is remarkably strong (stronger than all previous NS copper[55,56]) and at the same time ductile, while strain hardening at high rates. Its tensile curve[15] is reproduced in Figure 8. The mechanisms for the effective accumulation of dislocations reported[15] and the apparent work hardening observed for the tiny grains[13,15] are not yet fully understood.

CONCLUSIONS

In the eight approaches to achieve enhanced ductility for the NS metals and alloys, the common theme is the tailoring of microstructure by manipulating the nanoscale features present in the NS grains, be they twins, second phases, precipitates, or (nonequilibrium) grain boundaries. The alternative is to take advantage of the dependence of the flow mechanism on deformation conditions so that appropriate deformation conditions can be employed to make use of the elevated strain-rate hardening capability of some NS materials. The end result is a simultaneous strength and

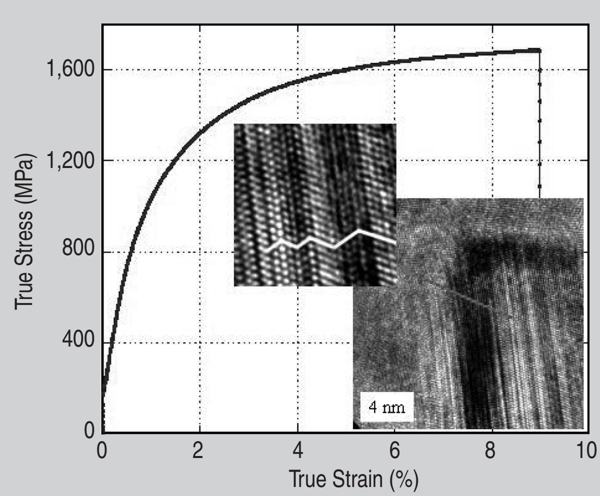

Figure 7. The tensile true stress-strain curve of an electrodeposited NS-Co foil tested in the authors' laboratory. The inset displays high-resolution TEM micrographs showing high densities of nanoscale twins (see white markers) in a local region inside an NS grain (Y.M. Wang, M.W. Chen, and E. Ma, unpublished).

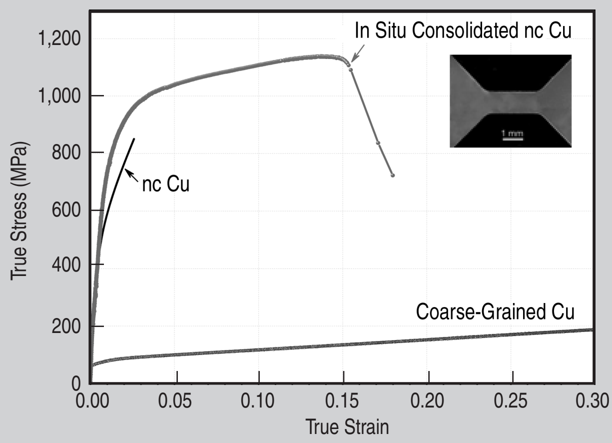

Figure 8. A typical tensile stress-strain curve[15] for the bulk in-situ consolidated nanocrystalline (nc) copper sample, with a relatively narrow lognormal grain size distribution with an average grain size of 23 nm, in comparison with that of a coarse-grained polycrystalline copper sample (an average grain size larger than 80 μm). A curve representative of previous nanocrystalline copper samples[55,56] is also included for comparison.

ductility, rather than a high strength alone.[56]

The author's group has participated, to varying extents, in the studies of all these eight strategies and concluded that these are research directions where new opportunities abound. As seen in the previous discussions, there are many poorly explained observations as well as unexplored territories. For almost each and every approach, there are qualitative and even hand-waving arguments that need to be substantiated and better analyzed. Other examples include the role of textures and the changes in slip planes in magnesium alloys where strong anisotropy exists, the effects of impurities on the stabilization/growth of the NS grain size and the tensile behavior,[57] and the influence of pre-existing twins on TWIP.[58] By and large, the optimization of properties and the cost associated with it has not yet made bulk NS alloys viable on commercial scales. Many scientific issues regarding the deformation mechanisms remain unsettled.[59–64] The author hopes that this short overview will stimulate interest in developing and understanding novel NS and ultrafine-grained metals and alloys, as well as in realizing the strength/ductility optimization potentials unavailable in coarse-grained metals and alloys.

Note that some applications in fact would benefit from the propensity for localized plastic deformation in NS materials. In that case, one need not go the extra mile to stabilize plastic deformation, but can directly take advantage of the NS behavior. Recently, the authors showed a prominent example in that direction: by driving the grain structure toward the NS regime, polycrystalline tungsten was made to shear in Kolsky bar uniaxial compression tests.[65,66] The shear localization and the subsequent discard of material would be desirable for certain critical applications. This is yet another example demonstrating the new opportunities brought forth by nanostructuring in bulk metals and alloys for desireable mechanical properties and engineering applications.[67]

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