

The Pursuit of the Small: From Grain-Boundary Cavities to Nanocrystalline Metals

Julia R. Weertman

Abstract

The following article is based on the Von Hippel Award presentation given by Julia Weertman of Northwestern University on December 3, 2003, at the Materials Research Society Fall Meeting in Boston. Weertman received the award for “her lifelong exceptional contributions to understanding the basic deformation processes and failure mechanisms in a wide class of materials, from nanocrystalline metals to high-temperature structural alloys, and for her inspiring role as an educator in materials science.” It has been said that “the best things come in small packages,” and that is certainly in Weertman’s mind in this presentation. She has spent much of her career “in pursuit of the small.” In this article, she first looks back at her experiences studying grain-boundary cavities and life in the spaces between grains. She then fast-forwards to modern work on nanocrystalline mechanical behavior, confirming that such nanocrystalline materials are indeed strong, but also brittle. Some of her experiences in studying these phenomena are also described.

Keywords: grain-boundary cavitation, mechanical behavior, nanocrystalline metals.

Introduction

First, I want to thank the Materials Research Society for this great award. When I look at the past recipients, I am daunted by being in their company. But unlike certain movie stars, I have no intention of giving this award back! Again, I’m very grateful for this honor.

A good deal of my materials science career has been involved in looking at phenomena associated with small entities: first, grain-boundary cavities; later, defects and structures in nanocrystalline metals. I am going to talk first about the grain-boundary cavity work, which took place some time ago, then move on to the nanocrystalline mechanical behavior and characterization work, which has stretched over about three academic units of time. (I count an academic unit of time as the “lifetime” of a PhD graduate student, about

five years. So, I have been working on this latter subject for about 15 years.)

Grain-Boundary Cavitation Studies

When I joined the Materials Science Department at Northwestern University, I was making a big switch from my past life—really, my past *past* life, which was in physics. I did not know a lot about materials science then, and I still shudder at some of the things I said in class off the top of my head when I felt I had to answer a question. I’m afraid I have led a whole generation astray on certain topics. I had been raising a family for 13 years (this was my past life), so it was quite a change when I came back to a technical career, and in a different field. I had no idea what sort of research to work on. Hans Weertman, my

husband, was developing a theory of grain-boundary cavitation at the time and suggested that I study that phenomenon.

At the time I started working on grain-boundary cavitation in the early 1970s, it was a hot area of research. The phenomenon is an important damage mechanism in materials subjected to stresses at elevated temperatures, and it presents interesting scientific problems. An important paper by Raj and Ashby¹ that had just been published calculated the high localized stresses that arise at serrations in grain boundaries and the effect of these stresses in driving cavity growth by diffusion. Under creep conditions, the high stresses rapidly smooth out.

We decided to examine cavity growth under fatigue loading.² At a brisk, fully reversed cycling of 17 Hz, the initial stresses have little chance to relax. Instead, they tend to produce voids at the predicted position near the tips of the grain-boundary serrations, as seen in the shadowed replica of Cu in Figure 1. The replica technique also proved useful in a study of the nucleation and growth of grain-boundary cavities at hard particles in an internally oxidized alloy of copper and silicon.³

The relation between dislocations and grain-boundary cavitation was explored in the high-voltage electron microscope (HVEM) at Argonne National Laboratory. The HVEM was chosen so that we could use relatively thick specimens with the voids totally enclosed. As expected, it was observed that voids produced both in creep and fatigue tend to be sited at ledges or bends on the grain boundaries. Less obvious, heavy tangles of dislocations were frequently seen feeding into the cavities (Figure 2).⁴

About that time, one of our very distinguished (and now sadly missed) faculty members, the late Jerry Cohen, spent a summer at the Institut Laue-Langevin in

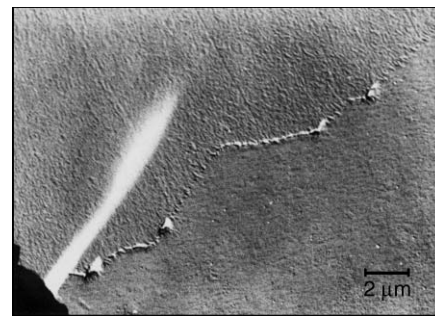


Figure 1. Shadowed replica of Cu fatigued at 17 Hz for 1.5×10^6 cycles. Strain amplitude = 0.035%, $T = 405^\circ\text{C}$. Note voids at the peaks. (From Reference 2.)

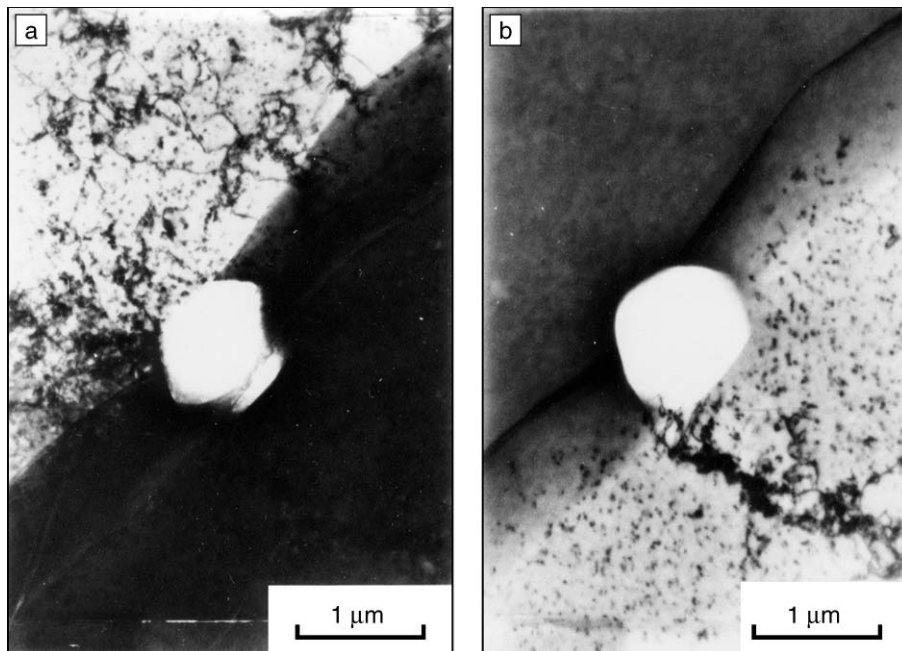


Figure 2. High-voltage electron microscopy picture of a grain-boundary void in 99.999% Cu fatigued at a strain amplitude of 0.032%, 17 Hz, 405°C (one-half the melting temperature T_m), for 3.5×10^4 cycles. The sample was tilted to bring the upper left-hand grain into contrast (a) and the lower right-hand grain into contrast (b). Note the bend in the grain boundary at the void and the intense dislocation activity in the void vicinity. (From Reference 4.)

Grenoble to carry out various neutron scattering experiments. He took along some of our cavitated samples and demonstrated that the cavities could be picked up by small-angle neutron scattering (SANS). This finding launched a series of studies by our research group to determine the evolution of grain-boundary cavitation during high-temperature creep and fatigue.⁵⁻⁷ While the use of SANS for such studies is commonplace now, at the time the idea was quite novel. This was an example of Jerry's remarkable forward thinking.

Among our research group's most noteworthy grain-boundary cavitation studies were those carried out by a number of outstanding visiting scholars from Japan with whom I had the privilege to work. In a series of studies, they examined the stress field around hard grain-boundary carbides in nickel-based superalloy specimens that had been subjected to deformation.⁸⁻¹² Their calculations showed that the deformation leaves intense internal stresses around the ends of the carbides, with the possibility of creating microcracks at grain-boundary carbide-alloy interfaces. At elevated temperatures, vacancies would be driven into those microcracks that are adjacent to material that experiences a tensile internal stress, and thus cavities would grow. No cavitation would be seen at the ends of carbides under compressive internal

stress. Strong shear stresses at the ends of the carbides produce localized slip that propagates from the carbide ends into the matrix. Observations made using shadowed replicas confirmed the analysis. This body of work represents a nice combination of theoretical mechanics and experiment.

Nanocrystalline Metals

In the immortal words of Monty Python, "And now for something completely different...."

My involvement in nanocrystalline research started with a dinner at a meeting of the Minerals, Metals, and Materials Society in New Orleans some 20 years ago. Hans and I were eating barbecued shrimp with Dick Siegel, and he was very enthused about this wonderful new field of nanocrystalline materials. He described a novel technique that permitted the synthesis of material with an extremely small grain size. Of course, like any other materials scientist interested in mechanical behavior, I immediately thought "Hall-Petch" and possibilities of great strength. (The Hall-Petch equation relates yield strength to the inverse square root of the grain size.) This dinner marked the beginning of a very fruitful collaboration between my research group and, first, Dick Siegel and then, after Dick left Argonne National Laboratory, Jeff Eastman, a collaboration that still continues.

There are many ways to synthesize nanocrystalline metals, none totally satisfactory. Currently, it is popular to make nanocrystalline samples by electrodeposition or some form of severe plastic deformation.¹³ The synthesis method used by Gleiter in his pioneering work on nanostructured materials¹⁴ is inert gas condensation (IGC); that is the method we have used to make most of our samples.

This is how it works: Metal that is to end up in the nanostructured form is vaporized (we use resistive heating) in a chamber evacuated to a high vacuum and back-filled with a low pressure of helium. The collision of atoms from the molten pool of metal with cooler helium allows the metal atoms to linger long enough to form nanoparticles. The particles travel by thermophoresis to a cold finger, where they are collected. The cold finger is continually scraped by rotating blades, and the little coils of scrapings fall down into a small bucket. At the end of an evaporation run, the helium is pumped out and the bucket is positioned so that the contents can be dumped into a die, where they are compacted under high pressure to form a solid disk, typically ~10 mm in diameter and about a millimeter thick.

Unfortunately, it is almost impossible to obtain samples without porosity, although with care, densities of 98–99% of coarse-grained values have been achieved. Both the use of a heat lamp directed onto the powder particles before compaction and heating the compaction die help to drive off some of the gas adsorbed on the powder and thus decrease porosity. However, such heating causes grain growth and an undesirable dispersion in grain size.

As the individual nanoscale particles leave the vicinity of the molten pool and travel to the cold finger, they tend to socialize, joining together into clusters with one or more fellow particles separated by twin boundaries or other low-energy or near-low-energy interfaces. The compacted nanocrystalline Cu samples, for example, are seen to be heavily twinned. This condition is inherited from the clusters; it is not primarily the result of the compaction process.¹⁵ Clusters have been observed in which the individual particles have undergone elastic strain to minimize surface energy. Line broadening x-ray measurements¹⁶ show that the rms internal strains in the clusters, typically on the order of 0.1%, appear to be the source of the high internal strains in the compacted material. (Essentially, all nanocrystalline metals, regardless of how they are made, have similar rms strains. Such strains obviously affect mechanical properties.)

In the early days of nanocrystalline research, there were great expectations of marvelous properties in this new class of materials.¹⁴ Nanocrystalline metals were predicted to be very strong, and indeed, they have proved to be so. However, predictions of high ductility, formability, and even superplasticity have not been realized. The material is distressingly brittle. Gone were hopes of easily forming nanocrystalline components made from normally difficult-to-machine materials and then strengthening them by a heat treatment to cause grain growth. It was originally believed that the grain boundaries in nanostructured materials are wide and of low density, thus explaining the overall low density of the material.¹⁴ It is now known that the grain boundaries in nanocrystalline metals are similar to those in their coarse-grained counterparts. The low density of these early nanocrystalline samples resulted from extensive porosity.

New deformation mechanisms were expected to operate in nanocrystalline metals. Molecular dynamics (MD) computer simulations¹⁷ and *in situ* straining observations in the TEM^{18–20} have shown dislocation activity down to a grain size of ~ 10 nm. Although simulations give valuable insight into deformation processes at the very smallest grain sizes, uncertainty remains about actual behavior. Coble creep is frequently invoked as a deformation mechanism, as well as various forms of grain-boundary sliding.²¹

In the fall of 1989, a paper²² appeared that has more legs than just about any other in the nano field. Chokshi and his colleagues measured the hardness of nanocrystalline Pd and Cu samples as a function of average grain size. The grain-size range of these samples was fairly narrow, but the measurements clearly showed a surprising softening with decreasing grain size, a “negative Hall–Petch slope.” The authors interpreted the results as validating Gleiter’s prediction of the occurrence of Coble creep at very fine grain sizes. The strain rate associated with Coble creep is proportional to the grain-boundary diffusivity, which normally would cause the creep rate to be extremely low at room temperature, but it also varies as the inverse third power of the grain size. The great boost from this second factor was believed to cause the observed softening with increasing grain refinement.

One month later, we had a similar paper published²³ in the same journal (*Scripta Metallurgica*). It presented results in conflict with those of Chokshi et al. No decrease in hardness was seen in our samples with decreasing grain size, down through the same grain-size range studied in the paper

of Chokshi et al., nor could any creep be detected at room temperature. Needless to say, this comparatively mundane paper did not arouse the interest produced by Reference 22. The difference in mechanical behavior between the two sets of samples appeared to lie in their preparation. Both were made by IGC and compaction, but whereas ours were all in the as-prepared state, Chokshi et al.’s measurements were made on a single Cu or Pd sample that was repeatedly annealed to produce an ever-larger grain size. Ordinarily, annealing leads to softening, but in the case of nanocrystalline metals, a heat treatment evidently can cause strengthening. To test this hypothesis, we divided a nanocrystalline Pd sample into four quarters, annealed three of the pieces at increasing temperatures, and then compared their hardness values.²⁴ The measurements, repeated on a number of nanocrystalline Pd samples, clearly showed strengthening from the heat treatment. Eventually, after sufficient grain growth took place, a reversion to conventional Hall–Petch behavior was seen. Overall, about a 50% increase in hardness at comparable grain sizes could be obtained by heating. Similar results were seen by Volpp, Göring, Kuschke, and Arzt²⁵ in ball-milled NiAl. MD computer simulations²⁶ of the effect of annealing on nanocrystalline Ni shows that some of the regions of disorder in the grain boundaries that serve as possible sites for dislocation nucleation are removed by the heating, thus making plastic deformation more difficult.

The history of the observed mechanical behavior of nanocrystalline metals is closely associated with the evolution of sample preparation techniques—for example, approaching full density and decreasing porosity, flaws, impurity content, and other sources of imperfection. Improving the vacuum of our IGC chamber, using higher-purity He, and heating the compaction die all made notable improvements²⁷ in lowering porosity and increasing the density of our samples, as revealed by small-angle neutron scattering (Figure 3). This change in sample quality was reflected in a pronounced improvement in mechanical behavior.²⁹

Early experiments on the strength of nanocrystalline metals indicated an abnormally low elastic modulus.¹⁴ But when the well-studied influence of porosity on elastic modulus was taken into account, it was seen that the elastic behavior of nanocrystalline metals, at least down to ~ 10 nm grain size, is not much different from that of coarse-grained counterparts.³⁰ It should be noted that Carl Koch and his students³¹ appear to have been the first to report the close agreement in modulus

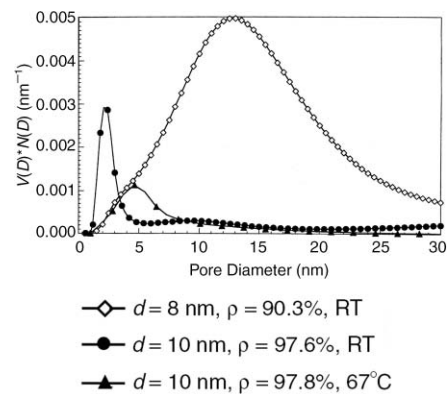


Figure 3. Volume–density pore size distributions as a function of pore diameter in nanocrystalline Cu produced under various conditions. On the y axis, $V(D)$ is the volume of a pore with a diameter D , and $N(D)$ is the number of pores in the bin of diameter D to $D + \delta D$. Open diamonds: Cu produced before synthesis upgrade, room-temperature compaction. Solid circles: Room-temperature compaction after upgrade to improved vacuum, heated die, and increased He purity. Solid triangles: After upgrade, compaction at 67°C . d = average grain size; ρ = relative density. Data obtained from small-angle neutron scattering. (From Reference 28.)

values between ball-milled nanocrystalline Cu and Ni and the conventional values.

Plan-view transmission electron microscopy (TEM) images of the internal structure of nanocrystalline samples are frequently seen in the literature. Micrographs of transverse cuts are not so common, probably because TEM sample preparation is difficult for such thin samples. The polished transverse surfaces of several of our nanocrystalline Cu samples appeared featureless, but etching revealed the presence of “roll-up” features.³² High-magnification SEM (scanning electron microscopy) and TEM pictures showed that the bonding was poor across these roll-ups, which appear to have their origin in the spiral scrapings from the cold finger (see Figure 4). Adsorbed gas on the scrapings (probably from the cold finger) prevents good bonding. Such defects obviously affect mechanical behavior, especially in tensile tests.

Our methods of tensile testing of nanocrystalline materials have evolved over the years. Working on the tiny, strong, but brittle samples is not easy. It is not surprising that most mechanical testing has relied on hardness measurements, together with the use of the general relationship that the Vickers hardness is about three times the yield strength. Our first stress–

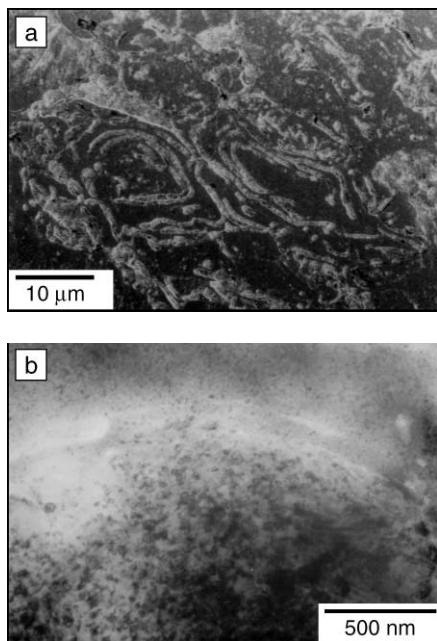


Figure 4. Electron micrographs of the face of a transverse cut through a nanocrystalline Cu sample. After etching, the apparently perfect polished surface revealed problems in bonding of the compacted nanopowder, probably across the spiral segments formed by scraping the cold finger. (a) Scanning electron micrograph showing the etched surface and “roll-up” features. (b) Transmission electron micrograph showing a poorly bonded region across a spiral interface. Note the difference in grain sizes across the interface. (From Reference 32.)

strain measurements relied on displacement of the grips to give the strain, a notably inaccurate assumption. Later, desperate but remarkably dexterous graduate students glued millimeter-sized strain gauges onto the gauge section of the samples to make “mini” samples to get a better measure of the strain. A Hall–Petch plot was constructed for nanocrystalline copper over a wide range of grain sizes.³³ (Note that the average grain size in most of our studies was determined by x-ray measurements using a Warren–Averbach analysis,³⁴ which often underestimates the actual volume-averaged grain size, as determined by extensive TEM measurements.³⁵) Data points for yield stress determined by converted hardness measurements or testing in compression—circumstances in which flaws are less likely to affect results—led to a Hall–Petch plot with the same slope as that of a plot extrapolated from coarse-grained tests (Figure 5). This slope is maintained down to a grain size of about 20 nm, at which point a flat-

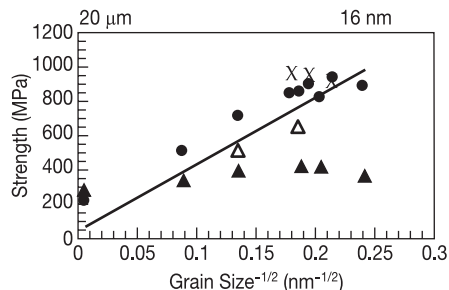


Figure 5. Hall–Petch plot of the strength, as measured by the yield stress, of copper versus the inverse square root of grain size. Data points were taken from tests in compression (x symbols), tension on the “mini” samples (solid triangles), tension on the “micro” samples (open triangles), and converted from hardness by dividing by 3 (solid circles). The solid line is extrapolation of coarse-grained Cu data. (From Reference 33 and unpublished data.)

tening of the curve may be occurring. Tensile tests, on the other hand, show little increase in yield strength below ~100 nm grain size (Figure 5, solid triangles, “mini” samples). Using special equipment³⁶ at the Johns Hopkins University, Kevin Hemker and colleagues tested even smaller samples of the same material, with a gauge volume reduced by a factor of 250, and reported a notable increase in the tensile yield strength (Figure 5, open triangles, “micro” samples). This improvement is interpreted as a consequence of lowering the chances of a large, well-oriented flaw occurring in the gauge section by reduction of the gauge volume. The considerable inherent yield strength of the nanocrystalline Cu should not be overlooked. Whereas coarse-grained pure Cu yields at less than 100 MPa, the high-purity nanocrystalline Cu of Figure 4 in the 10–30 nm grain-size range has a yield strength in compression of about 850 MPa. Coarse-grained nickel yields at a few hundred MPa, whereas nanocrystalline nickel has a yield strength of over 2 GPa in compression in the 10–30 nm range.³⁷

Modeling mechanical behavior requires assumptions about the mode of deformation of very small grains. It is not clear what the critical grain size is for cessation of dislocation contributions to deformation. MD computer simulations can give some insight into this problem,¹⁷ but a direct technique for observing activity during straining would be useful.

Several attempts have been made to carry out *in situ* straining in the TEM.^{18–20} A video camera, operating at the standard 30 frames/s, can capture some idea of

quick contrast changes, but this frequency is much too slow to spot any moving dislocations in midstream. Nonetheless, some conclusions can be drawn from the *in situ* studies. In the experiment with nanocrystalline Cu,¹⁹ brief, isolated areas of rapid contrast change—some as small as 10–20 nm—were observed. It is not absolutely certain that these changes were caused by dislocation motion. Static dislocations were observed in grains as small as 40 nm (or less). Twin boundaries were seen to act as a barrier to (presumed) dislocation motion. Occasionally, arrays of equispaced dislocations moving across a grain, then disappearing, were spotted. In Reference 18, nickel films were strained in the TEM. Dislocations were observed in grains as small as 10 nm. No obvious evidence of grain-boundary sliding was found. In a Ni film made by pulsed laser deposition, continuous thinning and high ductility suggest that significant slip transfer took place. The results of the *in situ* straining experiments appear to be generally consistent with the MD computer simulations.

Summary

Nanocrystalline metals show great potential as high-strength materials, but there are still considerable challenges. Synthesis methods must be radically improved, so that we are not making “femto-grams in a fortnight,” as Jim Williams at Ohio State has been quoted as saying. We need to have quality, quantity, and materials that are not so pricey. The knowledge base is still quite incomplete. We need to understand the deformation mechanisms at various grain sizes and in various materials, and we need a better understanding of the importance of internal structural features, high internal strains, and defects. In spite of these problems, the well-known observation is still true: The best things come in small packages.

Acknowledgments

A large number of remarkably talented and dedicated graduate students, visiting scholars, and even a few undergraduate students are responsible for the research described here. I am extremely grateful to them. I have been lucky to have had extended funding support from the U.S. Department of Energy that made it possible to explore many facets of nanocrystalline metal behavior, allowing a more nearly complete picture to be constructed. I especially want to thank my principal program managers, Drs. Stanley Wolf, Joseph Darby, and Michael Kassner. Finally, the encouragement of my family has been a bedrock of support. I would be remiss if I

did not acknowledge the invaluable help of Dr. Kai Zhang in helping with preparation of the talk and this manuscript.

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Julia R. Weertman is the Walter P. Murphy Professor Emerita of Materials Science and Engineering at Northwestern University. She obtained her BS, MS, and PhD degrees in physics from Carnegie Mellon University. Her research

currently focuses on the structure and properties of nanocrystalline metals. Weertman has been a member of the National Research Council's National Materials Advisory Board since 1999. She is a member of the National Academy of Engineering and a fellow of ASM International, TMS, and the American Academy of Arts and Sciences. Most recently, she was honored by ASM International as the Alpha Sigma Mu Lecturer (2003). She holds three patents, has published more than 150 papers and numerous book chapters, and edited three books. She is the co-author with her husband, Hans Weertman, of the textbook *Elementary Dislocation Theory*, which has been translated into three languages.

Weertman can be reached by e-mail at jrweertman@northwestern.edu.

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