NANOSTRUCTURED METALS

Less is more

Annealing out dislocations in deformed metals usually leads to reduced strength and increased ductility. Exactly the opposite has been observed in bulk nanostructured aluminium.

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Metals deform under applied stresses because of the movement of dislocations — line defects in the crystal lattice. Many dislocations are produced during plastic flow. The accumulation and interaction of these crowded dislocations creates obstacles that make the propagation of dislocations difficult. Further deformation therefore requires higher stresses, leading to work hardening. Conversely, annealing, a heat treatment often given to deformed metals to rearrange dislocations and relieve stresses, reduces strength and improves ductility as the dislocation roadblocks annihilate. This well-established picture for conventional metals apparently no longer holds for nanostructured aluminium, as reported recently by Huang *et al.*[1]. In fact, what they found was just the opposite, "hardening by annealing and softening by deformation".

This result is intriguing because Huang *et al.* worked with 99.99% pure aluminium that presumably contained no alloying element. The bulk metal remains polycrystalline after processing, except that the crystallites inside are nanostructured by a severe plastic deformation process that has created a high density of grain boundaries (GBs) and dislocations. After annealing, the density of dislocations in the grain interior decreased by 60%. This alone would reduce the strength by about 37%, on the conventional assumption that the strength scales with the square root of the dislocation density. Yet the removal of dislocations actually rendered the metal 10% stronger rather than weaker[1].

Before discussing our views regarding the mechanisms that could make this happen, two questions immediately come to mind. First, does this happen in other bulk nanostructured metals? The answer is probably yes. In addition to the examples cited by Huang and colleagues, low-temperature annealing also led to a 30% increase in the yield stress in nanostructured titanium[2]. At even smaller grain sizes, electroplated nickel exhibited elevated strength without losing ductility after moderate annealing[3], and ball-milled nanocrystalline iron-based and nickel-based alloys showed no decrease in hardness after 90% of the total dislocations were removed during annealing (T.D.S. and S.H. Feng, unpublished observations). In all cases, however, there remains the possibility that the segregation of small amounts of impurities to the dislocations and GBs, much as in the well-known strain ageing scenario[4], contributed

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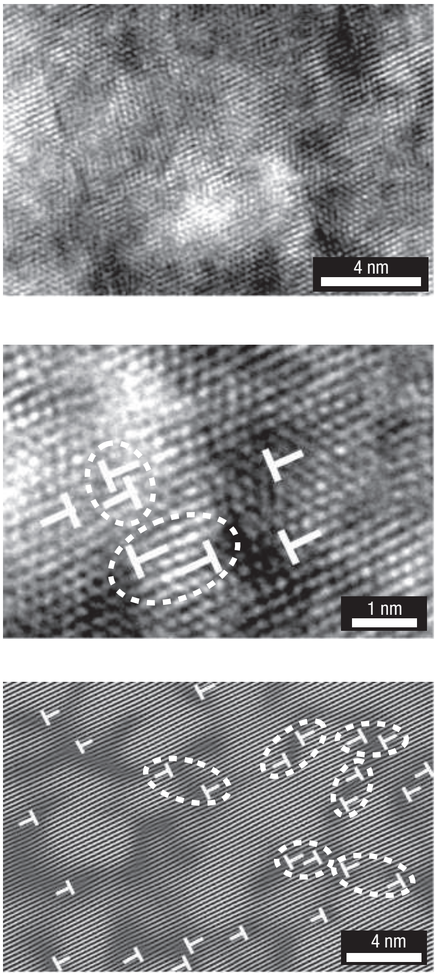

Figure 1 High-resolution transmission electron micrographs showing dislocations in a nanostructured aluminium grain (120 nm in diameter) produced by a large deformation. **a**, Contrast due to the presence of strain caused mainly by dislocations. **b**, High-magnification view, with dislocations marked (T). Note the circled dislocation dipoles. **c**, Fourier-filtered image of another grain. The ellipses show examples of dislocation dipoles.

significantly to the hardening observed, even though the impurity level might have been below the detection limit[3].

The second question is whether the removal of dislocations leading to higher strength has ever been observed in metals such as aluminium. The answer is yes, but only in carefully prepared single crystals with dimensions on the micrometre scale. Obviously, the probability of finding defects is low when the sample volume is small, such that defect-free whiskers can exhibit an extremely high yield strength approaching the theoretical limit[5]. Furthermore, dislocations existing in a small volume can all run out of the sample body on straining, causing dislocation starvation, so that continued deformation demands higher stresses to nucleate new dislocations[6,7]. These cases would be more in line with an intuitive expectation: the fewer the defects in a material, the higher its strength.

But bulk engineering metals do not behave in this way, because many dislocations and their sources are inevitably present inside the polycrystalline grains. Without the possibility to rid the material of dislocations completely, the practical strengthening strategy is rather to put in more dislocations so that they get in each other's way.

Now, what is the difference if these polycrystals have tiny grains? The lattice dislocations no longer matter as much, because the strength becomes dominated largely by how dislocations originate from and interact with GBs, as revealed by previous computer simulations[8] and experiments[9]. The stored dislocations are closely spaced, shown in Fig. 1, and often take the form of dislocation dipoles[10] — a pair of dislocations close by but with opposite signs. A dislocation dipole generates only short-range forces because of the screening of their stress fields[11]. This is likely to be true for the dislocations stored inside the tangles in the nanostructured grains in ref. 1. Thus, many of the dislocations contribute little to the long-range internal stress field and the overall strength. In fact, their movement is easier under applied stresses than the generation of new dislocations from GBs[8].

On annealing, therefore, the elimination of the dislocations renders the material less prone to plastic flow. Indeed, annealing drives the dislocations to disappear into the abundant GB sinks near by[1], and to annihilate through recombination and climb. Unlike for large grains, annealing readily sweeps the small grain volume clean. This dislocation exhaustion leaves the nucleation of dislocations at GBs as the main supply of mobile dislocations, which requires higher stresses. In fact, the annealed GBs are more relaxed and are also less likely to emit dislocations[3,12].

Thus, in nanograins the strength is sensitive to the atomic processes at the GBs, which are also where impurities tend to segregate to, especially on annealing[3]. Even when the metal incorporates only a trace amount of impurities during its processing, the segregation to the GBs could be sufficient to suppress the dislocations from emerging, or to pin them down as they propagate[3,8]. The dislocation–GB interactions are known to be thermally activated processes[8,9,13].

One can view the high strength induced by nanostructuring as a result of severe constraints on dislocation activities. In this context the moderate annealing or brief deformation[9] serves to exhaust the available dislocations further. To facilitate plastic deformation, the idea advocated by Huang *et al.* is to impose an intra-grain dislocation structure through externally forced large deformation.

Although it is clear that fewer dislocations do not necessarily mean softening and could in some cases even provide strengthening, a full understanding of hardening by annealing and softening by deformation in nanostructured metals requires more in-depth studies. The range of possible observations and mechanisms is fascinating and provides plenty of new opportunities for future research.

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