

CRYSTALLINE PLASTICITY OF NANOCRYSTALLINE MATERIALS AT ELEVATED TEMPERATURES

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Abstract. Significant differences have been observed in the deformation characteristics of nanocrystalline (nc) materials at elevated temperatures as compared to their microcrystalline (mc) counterparts. This includes significantly higher flow stresses and much enhanced strain hardening rates in nanocrystalline structure. Conventional understanding on elevated temperature crystalline plasticity cannot explain these observations. In-situ TEM experiments at elevated temperatures have demonstrated the occurrence of cooperative grain boundary sliding (CGBS) in the nanocrystalline state. The rationale behind such sliding and its role in explaining nanocrystalline plasticity, including superplasticity (using Ni₃Al as a model material), will be discussed in the context of the observed results.

1. INTRODUCTION

An important prediction for materials with decreasing grain size is their great potential to deform superplastically. It has been shown theoretically that by reducing the grain size it is possible to increase the superplastic strain rate, and/or decrease the superplastic temperature. Both aspects happen to have attractive technological significance. In a number of materials with initial grain size of about 100 nm, high strain rate superplasticity and/or low temperature superplasticity have been observed experimentally but concurrent extensive grain growth during superplastic deformation was detected in all materials [1]. Our previous results on deformation behavior of nanomaterials revealed a clear *correlation between microstructural instability and enhanced plasticity* [1,2]. In the most intensively in-

vestigated materials with grains smaller than 100 nm, high temperature deformation in the temperature range where the *microstructure is stable* results in a slight increase in ductility but is far from predicted superplastic behavior. Published results emphasize some exclusive features of deformation characteristics of nc materials [1,2].

The specific indications of superplasticity are caused by the special deformation mechanism related mainly to the intergranular character of deformation. As grain size is decreased, slip accommodation should become more difficult. An increasing role of grain boundaries in nc materials, especially as sources and sinks of mobile dislocations, was clearly demonstrated by molecular dynamics simulation [3]. Some experimental evidence of a transition from dislocation-mediated deformation to grain

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boundary-mediated deformation in nc nickel at room temperature has been recently reported by Shan *et al.* [4]. In that case, the grain size dependence of superplasticity may show a fundamental change for nc materials. Deformed nanomaterials have shown very little dislocation activity. Theoretical calculations [5,6] and molecular dynamic simulations [7] support this lack of dislocation activity. Suppression of conventional lattice dislocation glide (which dominates in coarse-grained materials) should activate alternative grain boundary deformation mechanisms in nc materials. Recently, clear evidence of grain rotation under applied strain was also detected in Al-1420 alloy produced by HPT during *in-situ* tensile test at elevated temperature [8].

In this paper, an experimental observation and analysis of deformation behavior of nanocrystalline Ni₃Al at elevated temperature is presented. This ordered intermetallic did show superplastic behavior at temperatures which do not lead to significant grain growth.

2. ELEVATED TEMPERATURE BEHAVIOR OF Ni₃Al

One of the most impressive and technologically important observations is low temperature superplasticity in nc Ni₃Al alloy. Nanocrystalline Ni₃Al alloy was obtained by high-pressure torsion (HPT) deformation under the pressure of 7 GPa that allowed us to obtain a porosity-free and contamination-free nanocrystalline matrix with grain size of approximately 50 nm. This alloy was tested both in microcrystalline [9] and nanocrystalline [10] states. A decrease in superplastic temperature for nanocrystalline state of Ni₃Al as compared to microcrystalline state with mean grain size of 6 μm is ~325 °C [10]. Similar decrease in the optimal superplastic temperature was also detected in titanium alloys [11], aluminum alloys [12] and titanium aluminides [13]. These reductions in superplastic temperatures represent a significant drop expressed in terms of homologous temperatures. For technological applications, the reduction of superplastic temperature for titanium alloys and Ni₃Al alloys are most relevant. However, a number of features observed during superplasticity in nanocrystalline Ni₃Al materials are different from the typical superplastic behavior of microcrystalline materials [1,10]. That includes high flow stresses and extensive strain hardening. One of the features of conventional superplasticity in microcrystalline material is low flow stresses (usually several tens of MPa). The observation of superplasticity with high flow stresses in

nanocrystalline material needs a new approach in order to explain the origin of such high flow stress behavior. We have observed a similar trend in other severely plastic deformed materials as well, e.g., 1420-Al alloy, Zn-Al, 2124-Al alloy [10,14-16]. Because of the grain size dependence in the constitutive relationship for superplastic flow [17], strain hardening during superplasticity has been conventionally explained in terms of grain growth. However, grain growth alone cannot account for the entire strain hardening in all these materials. At constant temperature, the stress dependence n , and grain size dependence p , have been experimentally determined for nanocrystalline Ni₃Al during the present investigation. Both of these values are equal to 2. An examination of constitutive equation [17] will reveal that at constant T and constant $\dot{\epsilon}$: $\sigma \propto (d)^{p/n}$; with $n = 2$ and $p = 2$, $\sigma \propto d$, i.e., the increase in stress should be directly proportional to increase in grain size. However, the results on Ni₃Al alloy at 650 °C and $1 \cdot 10^{-3} \text{ s}^{-1}$ strain rate shows a flow stress increase by a factor of ~5 where the grain size increased only by a factor of ~2 [18]. The transmission electron microscopy after superplastic deformation of Ni₃Al alloy also sheds light on the deformation mechanism showing the dislocation activity to be quite limited.

3. MICROSTRUCTURAL OBSERVATIONS DURING *IN-SITU* TENSILE TESTING

TEM/HREM studies including *in-situ* tensile testing had been performed at the National Center of Electron Microscopy (NCEM), Lawrence Berkeley Laboratory, Berkeley. During straining, a consistent change in grain contrast was observed in the Ni₃Al alloy. These changes were indicated by slow, slight orientation adjustments of isolated grains. An example of such orientation adjustment is presented by dark field images in Fig. 1. At the beginning of this sequence, there were no grains in a strongly diffracting condition in the area indicated by the white arrow (Fig. 1a). After 20 seconds, a bright spot emerged from a grain. Other nearby grains, which were in a strong diffraction condition, did not rotate out of contrast, confirming that there had been *no global rotation* of the specimen area and that the rotations observed at the arrowed location were internal changes of the sample structure. Local grain sliding and rotation were also observed in nanocrystalline Ni [4] and Al-1420 alloy [8] during *in-situ* straining. However, strains of hundreds or thousands of percent cannot be explained by just

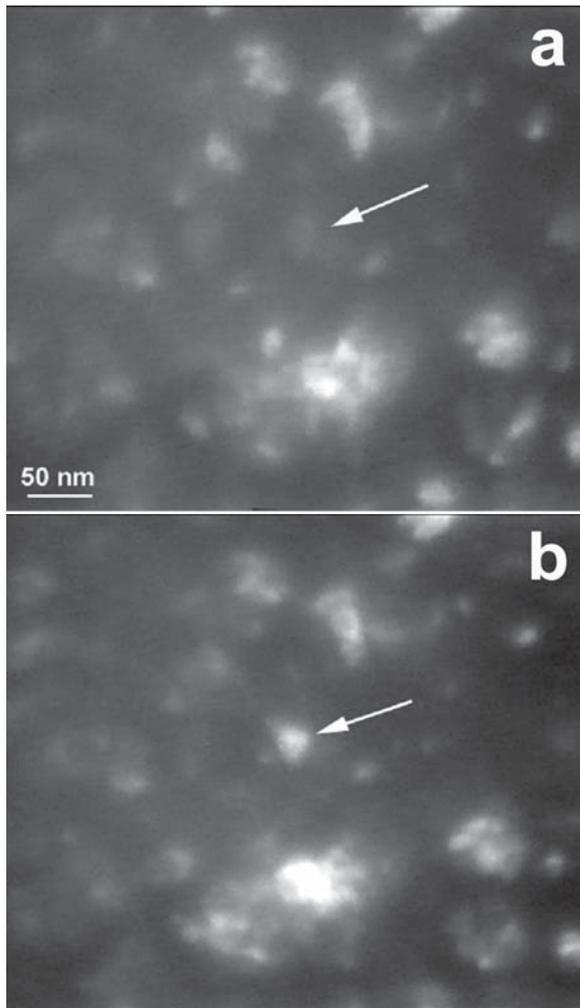


Fig. 1. Dark field images extracted from a real-time video corresponded to different times during straining.

local sliding and/or rotation of separate grains. The latest data shows that this phenomenon is conditioned by the operation of a specific deformation mechanism — CGBS, which was observed and analyzed in detail for mc materials [19,20]. This mechanism is associated with sliding and rotation of entire grain groups. Recently, we have obtained experimental evidence for this process in nc materials during *in-situ* tensile testing of nanocrystalline Ni₃Al alloy [21]. The bright field micrograph in Fig. 2 is showing a formation of sliding surfaces (marked by arrows) that supports the idea that cooperative grain boundary sliding took place during deformation of nanocrystalline Ni₃Al alloy. The mechanism associated with sliding and rotation of entire grain groups, along common sliding surfaces, was ex-

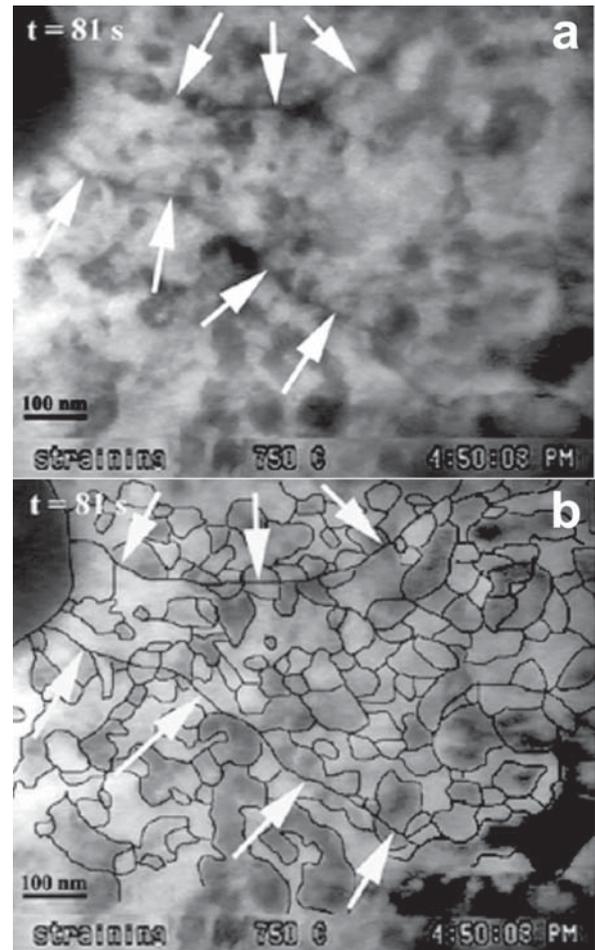


Fig. 2. (a) TEM image of nanocrystalline Ni₃Al extracted from a real-time video of *in-situ* tensile test at 750 °C. Highlighting of recognizable GB in (b) helps to see sliding surfaces that are pointed out by arrows.

perimentally observed and analyzed in detail for microcrystalline materials [19,20]. The most valuable evidence for occurrence of this mechanism in microcrystalline materials is the existence of steps along shear planes on a surface of post-deformed materials. Pre-polished surfaces of the superplastically deformed samples of Ni₃Al were also analyzed by SEM and revealed specific features that are usually attributed to cooperative grain boundary sliding [22]. These features include offset of reference marks and are indicative of shear events in two directions at 45° to straining direction. Geometrical size of the offsets and shear bands is a few orders of magnitude larger than the mean grain size of the material suggesting that large groups of grains are involved in the sliding process.

4. MODELING

Collective shuffling of a few tiny grains on a shear plane has also been predicted in computer simulations [23] and was experimentally observed in nc metals [24-29]. Molecular dynamic simulation also lends support to the above observations [30] showing a formation of common shear planes during deformation. With the formation of local shear planes, some grains can move collectively relative to some others. The authors [30] also showed that due to presence of grain boundaries that are resistant to sliding (i.e., special low angle grain boundaries or misorientation close to twin boundary), local shear planes can concentrate around these grain boundaries creating a cluster of grain embedded in a sliding environment. Hahn *et al.* [31,32] have pointed out that in order to overcome grain-boundary obstacles, two or more grain boundaries must cooperate to form a plane interface, which then by further interconnection with other plane interfaces, will lead to long-range sliding. To accommodate these plane interfaces, the local migration of grain boundaries is necessary. Such migration involves the triple junction migration and a change in the triple junction geometry. Gutkin *et al.* [33] have attributed a change in the triple junction geometry to the local grain boundary sliding via grain boundary dislocation glide through the triple junction (Fig. 3). These authors have also developed a theoretical model where stress-induced grain boundary migration is described as a new mode of rotational plastic deformation in nc materials [34]. Thus, a combination of grain boundary migration via grain boundary sliding and grain boundary migration via intragranular slip and grain rotation are believed to be dominant processes by which shear planes form [22].

5. CONCLUDING REMARKS

Superplasticity is a much more complex physical process than just the ability of fine-grained polycrystalline materials to exhibit high elongations prior to failure. A direct observation of CGBS in the nanocrystalline material is a confirmation that this deformation mechanism is common for mc and nc materials during superplasticity. Under conditions of microstructural stability, sliding surface formation with subsequent CGBS is believed to be responsible for such elongations. Such a deformation mechanism is common in mc and nc materials to the point where dislocation or diffusion accommodation can support it, and is considered as superplastic flow. When considerable grain growth is ob-

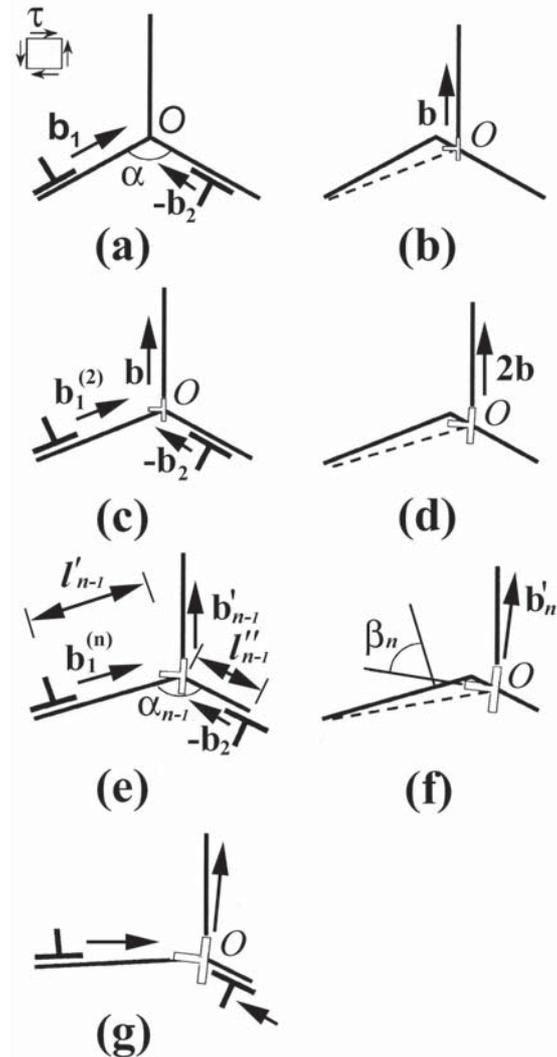


Fig. 3. Numerous acts of transfer of GB dislocations across a triple junction and accompanying local migration of GBs make GB planes (adjacent to the triple junction) temporarily parallel to each other [See Ref. 33].

served during deformation, the formation of sliding surface and CGBS is restrained. Despite the fact that these nc materials show enhanced plasticity, their deformation is more likely to occur by dislocation or diffusion creep than by grain boundary sliding responsible for superplastic flow in nc materials with stable microstructure.

Thus, limited ductility of nanocrystalline materials and some specific features of their elevated temperature behavior should be attributed to microstructural stability as well as to the probability of forma-

tion of sliding surfaces, sliding distance, and related accommodation mechanisms.

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