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Deformation twinning in a nanocrystalline hcp Mg alloy

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Nanocrystalline (nc) hexagonal close-packed (hcp) metals are rarely observed to deform by twinning, which is contrary to face-centered cubic metals. Here we report that, after alloying Mg with 10 at.% Ti, deformation twins are observed in an nc Mg–Ti alloy processed by mechanical attrition. The formation of deformation twins is attributed to the alloying effect, which may change the energy path for twinning. These results point to a promising approach to design nc hcp alloys for superior mechanical properties.

Keywords: Twinning; Nanocrystalline; hcp; Mg; Alloying

Nanocrystalline (nc) materials usually have high strength, but disappointingly low ductility [1]. Early attempts to increase the ductility often resulted in decrease in strength [2,3], although in some cases both high strength and good ductility are observed [4]. Twins in nc face-centered cubic (fcc) materials have been reported to increase both the strength and the ductility by accumulating dislocations at the twin boundaries [5]. Fortunately, fcc materials have been found to deform by twinning easily in their nc state [6,7]. This makes deformation twinning one of the most promising strategies for simultaneously increasing the strength and ductility of nc fcc metals and alloys [8–11]. However, this important strategy for improving the strength and ductility has so far not been available to nc hexagonal close-packed (hcp) metals and alloys because they rarely deform by twinning [12].

The twinning behavior of hcp materials is very different from that of fcc metals. For fcc metals, with decreasing grain size it becomes more difficult to deform by twinning in the coarse-grain size range [13], but twinning becomes easier once the grain size is smaller than 100 nm [6], although twinning may become difficult again when the grain size is too small (inverse grain size effect) [14]. In contrast, coarse-grained hcp metals usually need twinning to accommodate plastic deformation in addition to dislocation slip due to their lack of sufficient slip systems. However, twinning is rarely observed in nc hcp metals and alloys, with the exception of nc Zr processed by surface mechanical attrition [15]. The reason for the observed grain size effect on twinning in hcp materials is not clear. It would be scientifically and technically important to activate deformation twinning in nc hcp metals.

It is the objective of this investigation to activate deformation twinning in hcp Mg by adding Ti as the alloying element. Alloying will change the energy path for twinning, i.e. the general planar fault energy (GPFE) curves, and this will affect propensity for twinning [16]. We choose Mg as a model material in this study because it has the lowest density among structural metals except for Be. Ti is chosen as the alloying element because it has a high solubility in Mg when processed by ball milling [17,18].

Nanocrystalline Mg–10 at.% Ti samples were prepared by ball milling in a SPEX 8000 mill, using a steel vial and balls. Starting materials were elemental powders of Mg (99.9%) and Ti (99.5%). X-ray analysis reveals the Bragg peaks corresponding to the hcp Mg phase after milling for 24 h, indicating that no new phases formed and the Ti formed a solid solution with Mg (see Fig. 1a). Note the shift in the Mg alloy peaks to higher 2θ angles, indicative of a contraction of the lattice parameter. This is important because only when Ti forms a solid solution can it lower the stacking fault energy. For simplicity, the Mg–10 at.% Ti alloy is hereafter referred to as the Mg–Ti alloy. Shown in Figure 1b is the statistical distribution of grain sizes with an average grain size of 33 nm.

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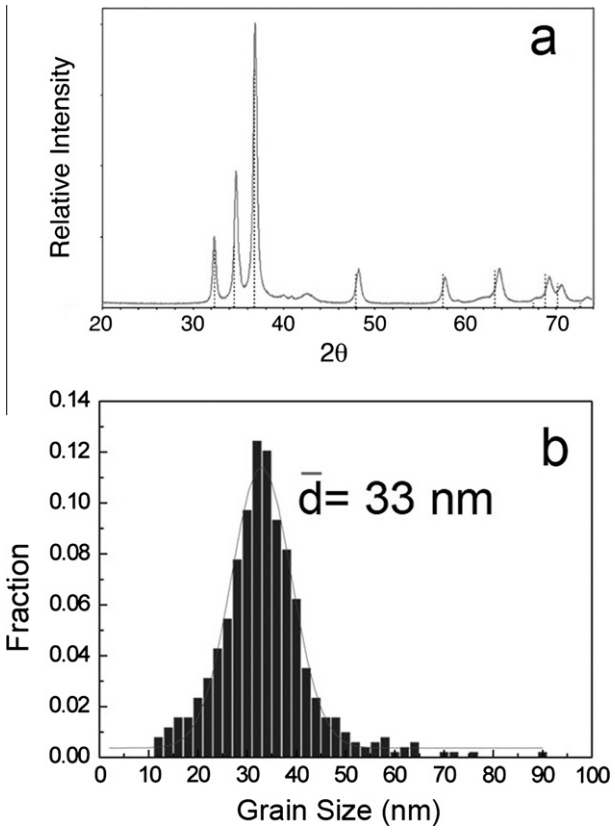

Figure 1. (a) X-ray diffraction pattern indicating a single hcp Mg phase. (b) Statistical distribution of grain sizes in the as-processed nc Mg–Ti alloy, measured from over 500 grains in transmission electron microscopy micrographs. The average grain size is 33 nm.

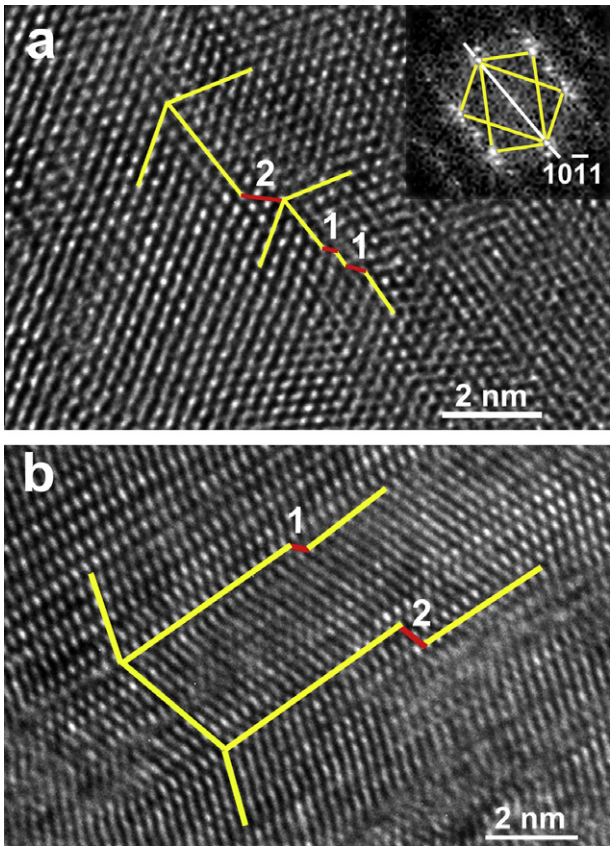

Figure 2. (a) HREM micrograph showing a deformation twin in the nc Mg–Ti alloy, viewed along a $[11\bar{2}3]$ zone axis. The twin system is $\{10\bar{1}1\}\langle 10\bar{1}\bar{2}\rangle$. A two-atomic-layer step and two one-layer steps are on the twin boundary. (b) HREM micrograph showing another deformation twin with a two-layer step on the lower twin boundary and a one-layer step on the upper twin boundary.

Figure 2 shows high-resolution electron microscopy (HREM) images of two deformation twins in the as-processed nc Mg–Ti alloy. The images are viewed from a $[11\bar{2}3]$ zone axis. Coherent twin boundaries and mirror images of atomic arrangements are clearly shown, verifying that they are indeed twins. Importantly, a two-atomic-layer step on the coherent twin boundary is shown in both Figure 2a and b (marked by 2). In addition, there also exist two one-layer steps on the coherent twin boundary in Figure 2a and one one-layer step in Figure 2b (marked by 1). The most common twinning system observed in hcp Mg is $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ [19,20]. The $\{10\bar{1}1\}\langle 10\bar{1}\bar{2}\rangle$ deformation twins observed in this study are consistent with the twinning mode observed in MD simulation of Mg under compression [19]. Interestingly, this twinning system was also observed in hcp Ti and Zr processed by severe plastic deformation under pressure, although the twinning only happened in large grains [21,22]. MD simulation predicted that $\{10\bar{1}1\}\langle 10\bar{1}\bar{2}\rangle$ deformation twins grow by the slip of a zonal twinning dislocation, ½•½ $\langle 10\bar{1}\bar{2}\rangle$, on the coherent $(10\bar{1}1)$ twin boundary, followed by atomic shuffling. This zonal dislocation involves two $(10\bar{1}1)$ planes, forming a two-layer step on the twin boundary. Such two-layer steps are indeed observed in Figure 2, which clearly verifies this twinning mechanism predicted by the MD simulation. The MD simulation also predicted that the one-layer steps are immobile and that the four-layer steps are unstable and can spontaneously dissociate into two two-layer steps [19]. The one-layer steps are experimentally observed in Figure 2, while no four-layer steps have been observed in our samples. Interestingly, Figure 2a shows two one-layer steps next to each other, which could merge to form a two-layer step, i.e. a zonal twinning dislocation. Over 50 grains in nc Mg–Ti alloy were observed from $[11\bar{2}3]$ zone axis, all of which contain deformation twins. It has been reported that ball-milled nanocrystalline Mg with grain sizes similar to the our nc Mg–Ti alloy do not deform by twinning [23,24].

Figure 3 shows several $\langle\bar{1}\bar{1}23\rangle$ dislocations in the nc Mg–Ti alloy (marked by T), lying on the $\{10\bar{1}1\}$ planes. The $\{10\bar{1}1\}\langle\bar{1}\bar{1}23\rangle$ slip system can produce strain in both the *a*-axis and the *c*-axis directions. This is very important because the slip system of coarse-grained Mg is usually $\{0001\}\langle 11\bar{2}0\rangle$, which cannot produce strain in the *c*-axis direction. The activation of the $\{10\bar{1}1\}\langle\bar{1}\bar{1}23\rangle$ slip system should help with the plastic deformation of the nc Mg–Ti alloy. This also indicates that the nc hcp Mg–Ti alloy deforms via slip systems that are normally not active in coarse-grained Mg, a phenomenon similar to that observed in nc fcc materials.

Although X-ray analysis verified that Ti solute completely dissolved into the Mg matrix to form a hcp solid solution (Fig. 1a), there remains the questions of

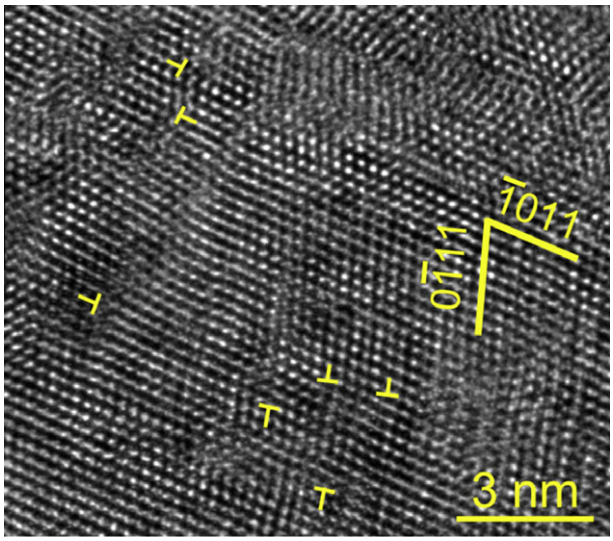

Figure 3. HREM image showing $\langle \bar{1}\,\bar{1}\,2\,3\rangle$ dislocations on $\{1\,0\,\bar{1}\,1\}$ planes. The zone axis is $[\bar{1}\,2\,\bar{1}\,3]$.

whether the Ti solute is uniformly distributed in the Mg matrix and if there is a grain size effect. To answer these questions, we performed energy-dispersive X-ray (EDX) analysis on grains of the nc Mg–Ti alloy. Shown in Figure 4a are two grains with sizes of 35 and 50 nm, respectively. Figure 4b shows that the Ti atoms are distributed uniformly in the small grain. However, in the large grain, Ti has a higher concentration in the area near the grain boundary than in the area near the grain center. This indicates that the elastic strain near the grain boundary facilitates the mixing of the Ti solute atoms into the Mg lattice. Figure 4b also shows that the small grain has a higher concentration of Ti, suggesting that the solubility of Ti increases with decreasing grain size. No intermetallic compound is observed in the EDX analysis, which is consistent with the X-ray analysis.

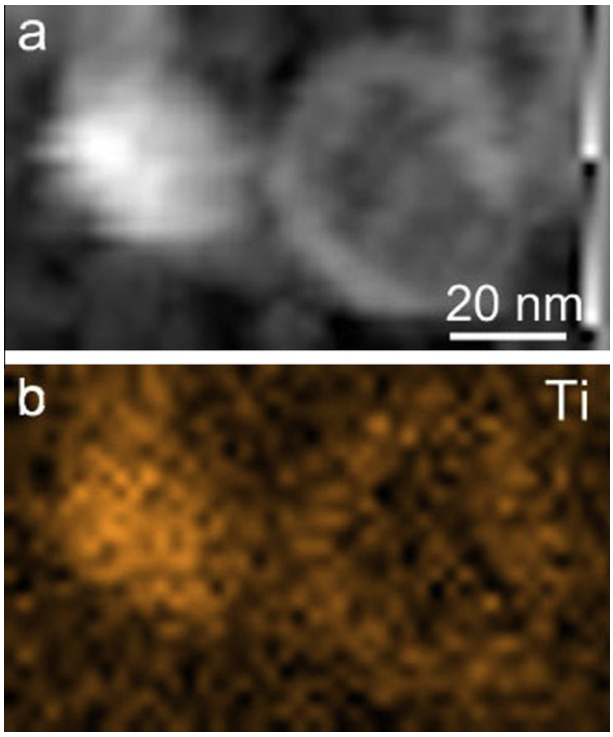

Figure 4. (a) EDX images of a small grain (left) and a large grain (right). (b) EDX analysis reveals uniform Ti distribution in the small grain, but in the large grain Ti has higher concentration in the region near the grain boundary than in the region near the center.

The segregation of Ti atoms to the region near the grain boundaries could have helped with the nucleation and growth of deformation twins observed in the nc hcp Mg–Ti alloy. It is reported that dislocation nucleation and emission from the grain boundaries became a major deformation mode in nc fcc metals because of the lack of dislocation source in the grain interior [6,25–27]. The dislocation nucleation and emission likely also play a major role in nc hcp metals. Since the GPFE energies primarily affect the dislocation nucleation [28], the segregation of Ti atoms to the grain boundary region should strengthen the alloying effect on the dislocation nucleation, and might consequently help with the formation of deformation twins observed in the nc hcp Mg–Ti alloy.

The twinning system in the nc Mg–Ti alloy is $\{1\,0\,\bar{1}\,1\}\langle 1\,0\,\bar{1}\,\bar{2}\rangle$, which can provide strain in the *c*-axis direction. In addition, a new slip system, $\{1\,0\,\bar{1}\,1\}\langle \bar{1}\,\bar{1}\,2\,3\rangle$, is also activated, which can also provide strain in the *c*-axis direction. In other words, both the deformation twinning and the newly activated slip system can facilitate plastic strain in the *c*-axis direction. This is significant for the nc hcp Mg–Ti alloy because it makes the total number of independent deformation systems larger than five, as required by the von Mises criterion for compatibility [29].

The activation of deformation twinning in the nc hcp Mg–Ti alloy is most likely caused by the change in the GPFE curves caused by Ti alloying. This statement is supported by the following observations. First, nc hcp metals and alloys have rarely been observed to deform by twinning [12], although twinning is a major deformation mechanism in their coarse-grained counterparts. Ball-milled nanocrystalline Mg with grain sizes similar to our nc Mg–Ti alloy has been reported not to deform by twinning [23,24]. Second, twins were observed in all nc Mg–Ti grains with an $\langle 1\,1\,\bar{2}\,3\rangle$ zone axis, which indicates the prevalence of deformation twins in the nc Mg–Ti alloy. It should also be noted that the high strain rate used in the ball milling should also have promoted twinning in the nc Mg–Ti alloy [14]. This is because a higher strain rate usually leads to a higher flow stress.

Three important energies act on the GPFE curves: the stacking fault energy, the unstable stacking fault energy and the unstable twin fault energy [16]. These are energy barriers associated with the nucleation and gliding of partial and twinning dislocations. They have been reported to significantly affect the twinning tendency in fcc metals and alloys [16,30,31], and a similar effect should also apply to hcp metals such as Mg. For example, it is known that lowering the stacking fault energy promotes the deformation twinning in nc fcc metals and alloys [9–11,32–34]. Also, a lower unstable twin fault energy should make twinning easier. It would be of interest to calculate the GPFE curves of the hcp Mg–Ti alloy as a function of Ti concentration in the future.

In conclusion, deformation twinning has been activated in an nc hcp Mg–Ti alloy, which is most likely caused by the modification of GPFEs through Ti alloying. The segregation of the Ti atoms near the grain boundaries makes the alloying effect stronger. These results suggest that it might be possible to design nc hcp Mg alloys to activate deformation twinning for improving the mechanical properties. In addition, new slip systems that are not available to their coarse-grained counterparts could be activated in nc hcp Mg alloys. Further studies are needed to investigate which elements are most effective in promoting deformation twinning in nc Mg alloys. In addition, the physics of the grain size effect on deformation mechanisms of nc hcp metals also needs further investigation.

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