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Scripta Materialia 69 (2013) 231–234

Scripta MATERIALIA

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Ultrahigh hardness and improved ductility for nanotwinned mercury cadmium telluride

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Received 12 March 2013; revised 2 April 2013; accepted 2 April 2013
Available online 17 April 2013

Unidirectionally coherent and boundary-free nanotwinned (nt) mercury cadmium telluride (HgCdTe or MCT) was achieved under plastic deformation of nanoindentation. The hardness of nt-MCT is up to 47.2 GPa, which is two orders magnitude higher than that of monocrystalline MCT. This increased hardness originates from the unique composite nt structure, which exhibits a repeated pattern comprising a lamellar twin >11.5 nm thick, followed by one or several lamellar twins with thicknesses <11.5 nm.

Keywords: Ultrahigh hardness; Ductility; Nanotwins; Nanoindentation; HgCdTe

Nanocrystalline (nc) metals with grain sizes <100 nm exhibit hardnesses up to five times greater than those of their coarse-grained counterparts [1,2]. This phenomenon is attributed to grain boundaries (GBs) presenting a barrier to plastic deformation [3]. However, these nc metals suffer from reduced ductility due to the presence of GBs [3]. To retain the ductility of nc metals, twin boundaries (TBs) are generated within the GBs [4,5]. Nanotwinned (nt) metals are up to 10 times harder than their coarse-grained counterparts, while retaining ductility [6]. The strength of nt metals is increased up to ~1 GPa, which is also 10 times that of coarse-grained ones, and both ductility and electrical conductivity are retained [4,5,7]. As nanotwins are produced within GBs by existing methods used to fabricate nt metals, such as electrodeposition [4,5,7], magnetron sputtering [6] and plastic deformation [8], GBs are left within nt metals. Consequently, GBs are widely dispersed within nt metals prepared by the abovementioned three methods. Nevertheless, GBs are unstable and readily coarsen under external stresses, even if these are applied only briefly [1,9]. This behavior is associated with the excess energy stored in GBs. Therefore, the existence of GBs within nt metals suppresses any further increase in both hardness and ductility. Nonetheless, little has been reported on boundary-free nt metals. On the other hand, unidirectionally coherent columnar-grained nt metals have better stability and higher hardness compared to their randomly oriented equiaxed counterparts [10]. Furthermore, unidirectionally coherent nt metals have potential for application in the semiconductor industry [11]. As a consequence, the unidirectionally coherent and boundary-free nt structure is expected to significantly increase both the hardness and ductility of nt metals.

To further increase both the hardness and ductility, a unique composite nt structure is required. This is ascribed to a critical value of lamellar twin thickness, λ, or spacing between adjacent TBs existing for the maximum strength [4,12]. The critical value of λ varies from 10 to 15 nm [4,12]. When λ is more than or equal to this critical value, strengthening dominates; however, when λ is less than this critical value, softening prevails [4,12,13]. Hence, a unique composite nt structure comprises a twin lamella with $\lambda \geqslant$ the critical value, followed by one or several twin lamellae with $\lambda <$ the

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http://dx.doi.org/10.1016/j.scriptamat.2013.04.007

critical value. Considering the potential applications of this kind of unique composite nt structure, the fabrication of a unidirectionally coherent and boundary-free composite nt structure is intriguing.

In order to fabricate the unidirectionally coherent and boundary-free composite nt structure, a metal with low stacking fault energy (SFE) is necessary [14]. Mercury cadmium telluride (HgCdTe or MCT) is a representative for third-generation soft-brittle semiconductors, which have low SFEs ranging from 10 to 14 $mJ\ m^{-2}$ [15,16]. The SFE of MCT is approximately in the middle compared to the nt metals usually used, e.g. Ni alloy (1.22 $mJ\ m^{-2}$) [8], brass (15 $mJ\ m^{-2}$) [14] and Cu (24-39 $mJ\ m^{-2}$) [17], which is typical for low SFE metals. Moreover, MCT has a soft and brittle nature [15,16], which is different from the soft-plastic nature of Ni alloy and Cu. Consequently, the improvement of unidirectionally coherent and boundary-free composite nt MCT structure in terms of both hardness and ductility is expected to be remarkable.

MCT single crystals are usually grown using a modified Bridgman method [16]. The standard methods used for nt metals, such as electrodeposition and magnetron sputtering, are not suitable for fabricating composite nt-MCT. Although dynamic plastic deformation induces polycrystalline nt metals, quasi-static plastic deformation, e.g. tension, compression and torsion, provides insights into unidirectionally nt metals [18]. Hence, quasi-static plastic deformation is a widely used solution for post treatment to fabricate nt metals grown by different methods.

In this study, we report our recent results of fabricating unidirectionally coherent and boundary-free composite nt-MCT, with ultrahigh hardness and improved ductility, by means of quasi-static plastic deformation of nanoindentation.

$Hg_{0.22}Cd_{0.78}Te$ (1 1 1) wafers were grown by a modified Bridgman method [16]. Each wafer was 10 mm in diameter and 0.8 mm thick. MCT wafers were lapped using waterproof abrasive paper with a mesh size of 2000. The wafers were polished using chemical mechanical polishing slurry, including silica, hydrogen peroxide and deionized water, for 15 min. After washing with deionized water and drying with compressed air, the surface roughness, R_a, of polished MCT is 0.95 nm, measured by a non-contact surface profilometer (NewView 5022, Zygo, USA). The scanning area for surface roughness measurement was $50 \times 70\ \mu m^2$. Both sides of the MCT wafer were polished. One polished side was glued to a circular stainless steel (SS) holder fixed to a nanoindenter, and another side was used for nanoindentation tests.

Nanoindentation was conducted on a nanoindenter (TriboIndenter®, Hysitron Inc., Minneapolis). A Berkovich tip was employed during nanoindentation, with a radius of ~150 nm. The included angle of the tip was 142.35°. Three kinds of nt-MCT were formed under the maximum loads of 500, 600, 700 mN, respectively. For each nt-MCT, the hardness was measured 10 times. The distance between two adjacent indentations was 500 μm. To measure the ductility of nt-MCT, four repeated tests were performed on each nt-MCT. Each test consisted of 100 repeated cycles. The distance between two adjacent ductility test sites was 1 mm. For all nanoindentations, the loading, dwelling and unloading times were 10, 10, 15 s, respectively.

Unidirectionally coherent and boundary-free composite nt-MCT was characterized using transmission electron microscopy (TEM). The TEM samples were prepared by a tripod technique [15,16]. The manually polished TEM samples were thinned in a Gatan Model 591 precision ion polishing system. During ion polishing the MCT samples were cooled by liquid nitrogen to avoid heat damage to the MCT. The tilt angles for the two ion guns varied from 2.5° to 3.0°, and power energy remained constant at 2.8 keV. TEM examinations were performed with a FEI Tecnai F20 transmission electron microscope operated at 200 kV. The surface topography prior to and after 100 repeated cycles on nt-MCT was characterized by a field emission scanning electron microscopy (SEM; Tescan Lyra 3).

Figure 1 shows cross-sectional TEM images of unidirectionally coherent and boundary-free nt-MCT induced at a maximum load of 600 mN. The selected-area electron diffraction (SAED) patterns (Fig. 1a, inset) show regular double-dot patterns, indicating the formation of twins. This is verified by the high-resolution TEM image shown in Figure 1b. SAED patterns reveal the {1 1 1} planes for the twin lamellae, which is confirmed by the perpendicular distance between adjacent lattice planes for the (1 1 1) plane of 0.373 nm. All twin lamellae are unidirectionally coherent and boundary-free. A composite nt-MCT structure is observed, which demonstrates that a thick twin lamella with $\lambda \geqslant$ 10–15 nm, followed by one or several twin lamellae with $\lambda <$ 10–15 nm. Therefore, the fabrication of unidirectionally coherent and boundary-free composite nt-MCT is achieved. Incoherent TBs are marked "ITB" in Figure 1b.

Figure 2 shows the distribution of lamellar twin thicknesses identified by TEM and high-resolution (HR) TEM images generated at a maximum load of 600 mN. Ten TEM and five HRTEM images were used to measure the distribution of lamellar twin thicknesses. Total 238 twin lamellae were employed to calculate the distribution. The frequency of lamellar twin thicknesses for 4, 5, 6, 7, 8, 9, 10, 11, 12 and 34 nm are 6.3%, 12.6%, 10.9%, 13%, 10.1%, 6.3%, 4.6%, 4.2%, 4.2% and 2.9%, respectively. The average value of all the lamellar twin thicknesses is 11.5 ± 9.6 nm. As the critical value of λ for maximum hardness of nt metals ranges from 10 to 15 nm [4,12,13], the average value of λ for 11.5 nm is considered the critical value. Accordingly, the composite nt-MCT structure

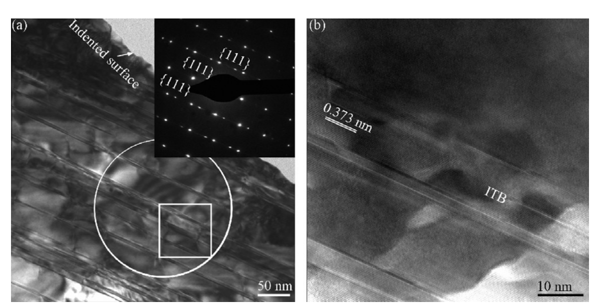

Figure 1. Cross-sectional TEM images of unidirectionally coherent and boundary-free nt-MCT at low (a) and high (b) magnifications induced at a maximum load of 600 mN. Inset shows the SAED pattern taken from the corresponding white circle. (b) is the enlarged area taken from the white square in (a).