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On the Study of the Orientated Cracks Formed in ErD_2 Thin Film

H. H. Shen[1,2], K. Sun[2], X. Xiang[1] and X. T. Zu[1]

1. School of Physical Electronics, University of Electronic Science and Technology of China, Chengdu 610054, China
2. Department of Materials Science and Engineering, University of Michigan, Ann Arbor, MI 48109, USA

Formation of metal hydrides via hydrogen (H) interacting with metals is an effective approach for hydrogen storage [1]. Thus, these metal hydrides have attracted much attention for decades [2-3]. Cracks have been reported to form during hydrogenation that will be detrimental to the mechanical properties of the metal hydrides and thus limits their practical applications [4]. Hydrogen-induced embrittlement has thus been an important issue to be resolved. In this work, we observed the formation of intragranular cracks in the erbium deuteride (ErD_2) films, and the cracks preferentially grow along the specific directions in the ErD_2 lattice.

Pure Er thin film on molybdenum was prepared by electron beam deposition at a substrate temperature of 350 °C and a deposition rate of 10 nm/s. To obtain erbium deuteride (ErD_2) thin film, the pure Er film was firstly heated to 600 °C in a vacuum chamber, where pure D_2 gas was then flowed in. After the Er film had been exposed to D_2 gas for up to 30 minutes, the D_2 was outgassed from the chamber allowing the sample to be cooled naturally to room temperature. Cross-sectional view specimens for TEM study was prepared using an in-situ lift-out method performed in a FEI-Helios 650 focus ion beam (FIB) instrument. TEM characterizations, including bright-field (BF), dark-field (DF), and high-resolution electron microscopy (HREM) imaging and selected area electron diffraction (SAED) and energy dispersive spectroscopy (EDS), were performed on a JEOL 2010F TEM working at 200 kV.

Figure 1(a) describes that preferentially orientated cracks are visible in the ErD_2 film. The SAED pattern, taken from the A1 area circled in Fig. 1(a) with the zone axis of [011], are utilized to verify the elongation directions of the well orientated cracks. Analysis of the SAED pattern suggest that the cracks in the corresponding BF image grow along the {111} crystal planes of the ErD_2 lattice. Besides, the appearance of polycrystalline rings in SAED pattern are indexed as the (211), (222), (332) and (440) crystal planes of Er_2O_3 lattice. It indicates that many Er_2O_3 particles have been formed in the cracks, since the cracks are included in area A1.

A magnified BF image of a crack is shown in Fig. 2(a) with an inset showing HREM image of the crack. A DF image on the basis of the Er_2O_3 polycrystalline rings is also shown in Fig. 2(b), in which bright contrast features of Er_2O_3 particles distribute in the whole ErD_2 film, especially on the edges of cracks. From the results of Fig. 2, it demonstrates that the orientated cracks are composed of many Er_2O_3 particles, instead of a real gap. Element mapping performed using X-ray signals confirms the formation of Er_2O_3 particles in the cracks, as shown in Fig. 3. The improved signal (counts with background) of O element appears in the cracks support the conclusion that the Er_2O_3 particles dispersed in the whole ErD_2 film and mainly in the orientated cracks.

The macro-strain is induced during the deuteration processing, which can attribute to the formation of crack-tips in the ErD_2 film. The {111} crystal planes are the major slip system for the metal hydride with FCC crystal structure, as those planes have largest lattice distances. Therefore, it is reasonable that the cracks grow along the {111} direction of ErD_2 film duration deuteration. Erbium is a kind of highly active element and is susceptible to be oxidation even at lower temperature [5]. The interesting observation in the present study is the formation of Er_2O_3 inclusion in the cracks, which is other than a real gap. Oxygen diffusion should be significant as the deuteration processing is conducted at 600 ^{o}C [6]. So it needs further study to understand whether the oxygen diffusion towards to the crack tips is also contributed to the formation of orientated cracks.

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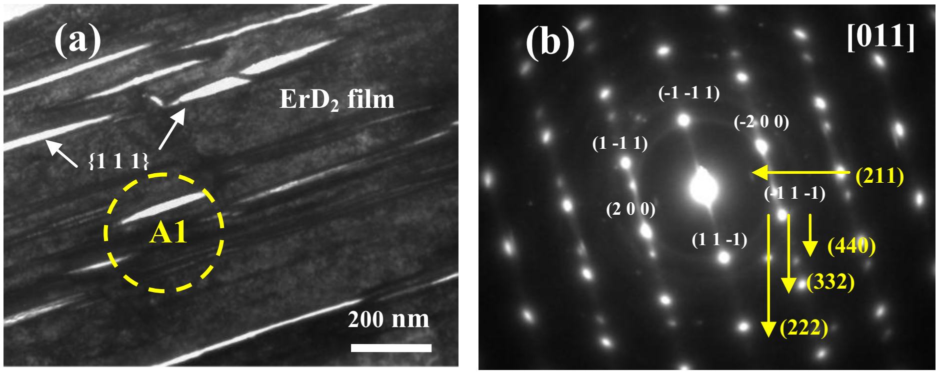

Figure 1. The cross section BF image (a) and corresponding SAED patt -ern (b) of molybdenum supported ErD_2 film. The SAED pattern (b) stems from the A1 area in (a) with the zone axis of [011].

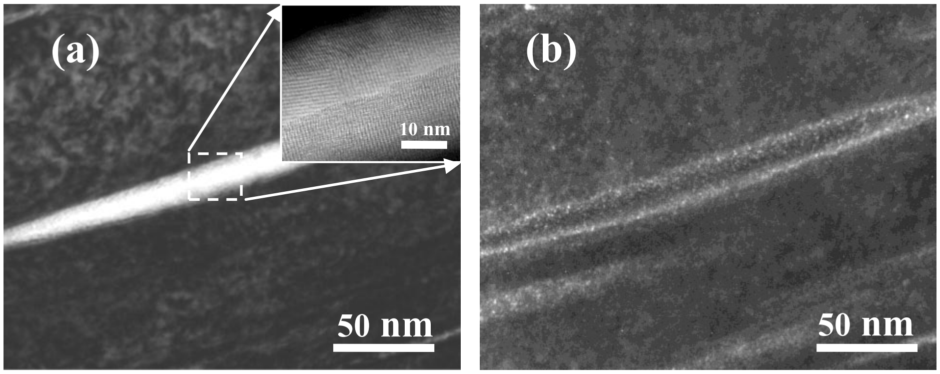

Figure 2. The BF (a) and DF (b) images of the orientated cracks. The DF image was captured on the basis of diffraction rings of Er_2O_3. The inset of (a) shows HRTEM iamge of the crack.

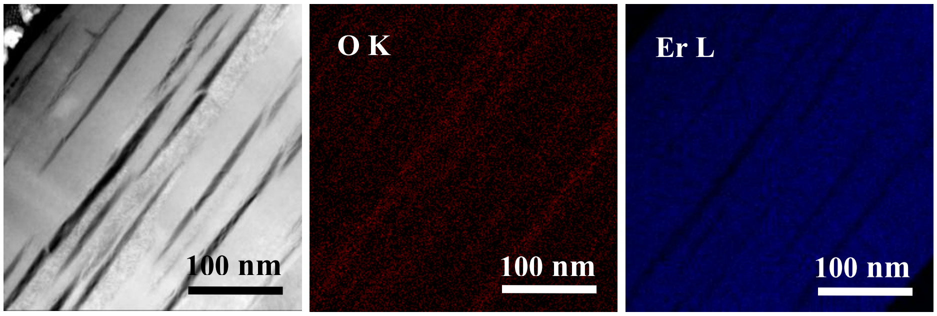

Figure 3. The element distributions of O (K), Er (L) and Mo (L) in the ErD_2 film.