DOI: 10.1017/S1431927605505154

Microsc Microanal 11(Suppl 2), 2005

Radiation Damage by Ar^+ Ion-Milling in Ferroelectric Oxides

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Ferroelectric oxides have a variety of applications as infrared sensors, actuators and non-volatile memory devices. A subgroup of these oxides fall into the category of complex perovskite relaxors, in which there is dispersion in the dielectric constant versus temperature as a function of frequency [1]. The relaxor oxides subjected to significant studies include $Pb_2(Sc,Ta)O_6$, $Pb(Mg_{1/3},Nb_{2/3})O_3$, $Pb(Sc_{0.5},Nb_{0.5})O_3$ and $Pb(Zr,Ti)O_3$ [2-3]. The ceramics, films and crystals of these materials have interesting structural, microstructural, and domain characteristics that can be studied by transmission electron microscopy, TEM. In most cases where TEM study is required, Ar^+ ion-milling of the samples must be carried out.

Pb-based oxides undergo some degree of milling radiation damage [4]. The severity of damage depends on the sample thickness immediately before milling and the time used to obtain electron transparent regions. Radiation damage is often visible as mottled or spotty background contrast, which originates from fully amorphous to partially amorphous transformations. A thin amorphous layer may also be found at the outer edge of most specimens subjected to about 15 minutes of irradiation. At the microstructural level of investigations, the adverse influence of radiation damage on image formation can be minimal. With the exclusion of the specimen outer edge, antiphase boundaries, ferroelectric domains, structurally ordered domains can be imaged in regions where radiation damage is present. However, in high-resolution TEM (HRTEM) studies, evidence of structural distortion or change originating from radiation damage can be easily observed. The result of such damage is poor quality images, lack of sufficient contrast and uncertainty about the true structure or interplanar spacing. In radiation damaged oxides, fine incommensurate structures can be difficult to identify or distinguish from altered clusters. Here, various examples of the appearance of radiation damage in $Pb_2(Sc,Ta)O_6$ are illustrated. Images recorded from milled samples and those recorded from crushed samples, Figures 1a-d, illustrate the high level of structural damage in milled samples. We have noted that thick samples, which require longer period of milling, undergo greater level of damage. We have also found that reducing sample thickness to about 20 microns, prior to thinning, as well as careful polishing by various grades of diamond paste, minimize radiation damage and improve image quality by a factor of about two. It is evident that grinding and polishing a sample to such a thickness is generally time-consuming and rather difficult. However, the gains are significant in that the milling time is substantially reduced to about 15 minutes in some cases and the TEM results recorded are certainly more representative of the true structure.

References

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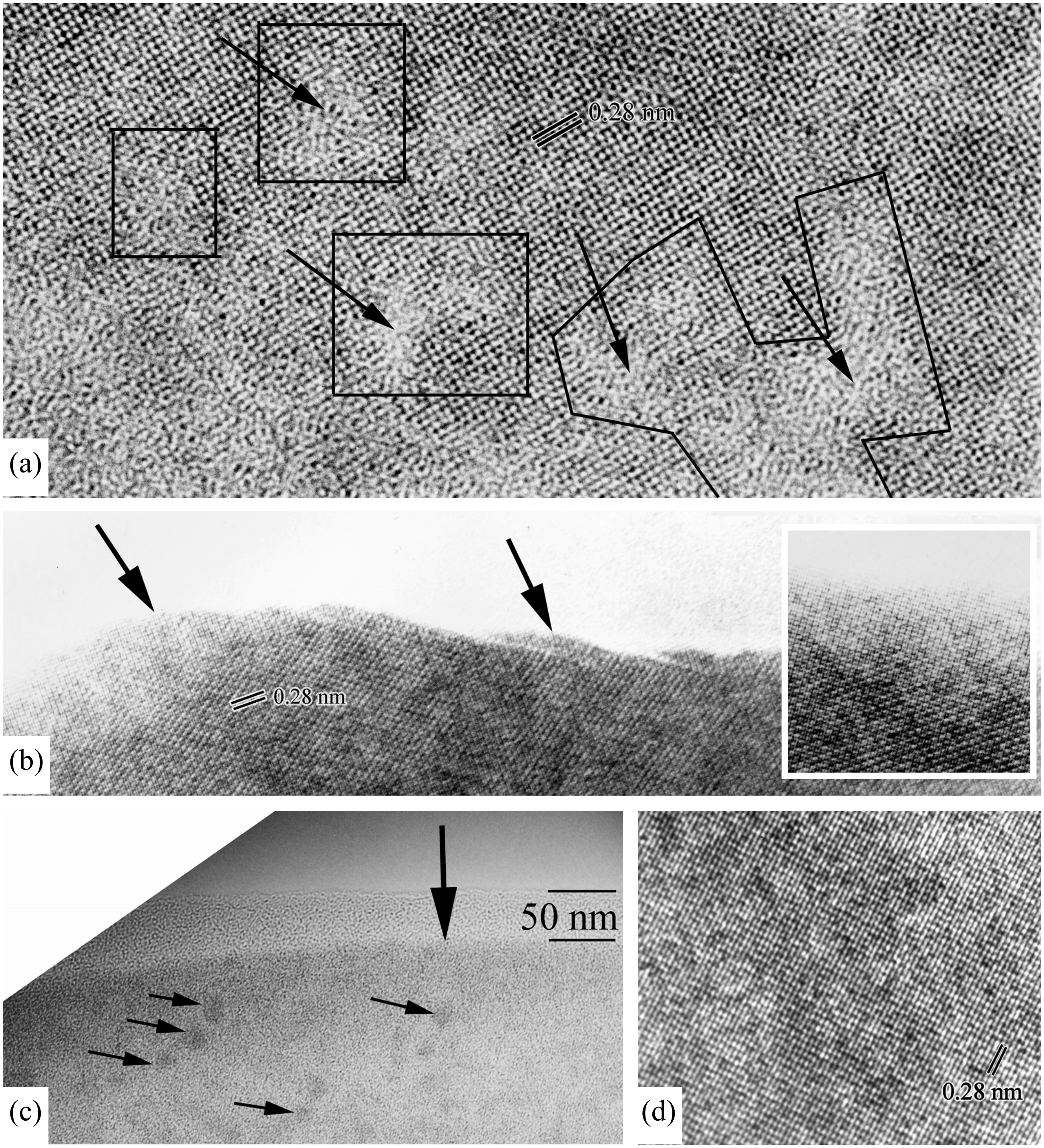
[5] Tai C.W. is supported by a grant from the HKPolyU., No.G-U054. Figure 1c was recorded at the Arizona State University.



Figures 1a-d. a) HRTEM image showing amorphous clusters resulting from ion-milling. b) HRTEM image of a crushed sample, showing evidence of micro-fracture at the sample edge, inset: enlarged image of a crushed sample showing no amorphous clusters. HRTEM images illustrating c) a 50nm amorphous layer at the sample edge and d) a defect induced by ion-milling.

https://doi.org/10.1017/S1431927605505154 Published online by Cambridge University Press