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Electron Microscopy Study of Fe_3O_4(111)/MgO(111) Polar Oxide Interface

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Surface polarity is one of the open questions in surface science due to the fact that bulk terminated polar oxide surfaces have infinite surface energy. MgO(111) is model polar oxide surface that has been studied in the context of mechanisms for its stabilization [1]. This work extends the search to polar oxide interfaces, by asking how substrate surface polarity affects growth of polar oxide films. To this end we investigated Fe_3O_4(111) films grown by Molecular Beam Epitaxy (MBE) on a single crystal MgO(111) substrates.

Standard polishing-dimpling-ion milling methods were used to prepare plan view samples in [111] zone, and cross-sectional samples in <110> and <211> zones. A Hitachi H-9000 NAR microscope, operated at 300 keV, was used for bright field transmission electron microscopy (TEM), high-resolution electron microscopy (HREM), selected area diffraction (SAD), and convergent beam electron diffraction (CBED). Data was recorded digitally with a Gatan slow scan CCD camera.

TEM and HREM images (Fig.1 a and b) reveal a flat and sharp Fe_3O_4(111)/MgO(111) interface, ruling out interface faceting as a possible stabilization mechanism. The Fe_3O_4(111) film starts to grow as a well-ordered epitaxial film over most of the substrate (Fig. 1b), resulting in film surface parts that are plan parallel to the interface (Fig.1a). The film surface also has faceted parts that protrude out of the film (Fig.1a). This morphology is correlated with the presence of inclusions in the film (Fig.2). Using HREM diffractograms, combined with CBED (Fig.3) and SAD analysis, we identified two phases within the Fe_3O_4(111) film, elemental Fe inclusions growing in [110] direction, and Fe_3O_4 inclusions growing in [100] direction. The major phase is Fe, whose metallic nature offers a solution to the polarity of the oxide substrate and film. Some Fe inclusions start nucleating at the interface and others within the film. Most are constrained in their growth and enveloped by the matrix. Some grow larger and overcome the lattice mismatch most likely by twinning (not shown here). Fe_3O_4(100) inclusions are also observed at the interface and within the film, and tend to be smaller than Fe. Fe_3O_4(100) is also a polar surface and it is not apparent how this phase contributes to the stabilization of Fe_3O_4(111). The crystallographic orientation of Fe_3O_4(100) and Fe(110) is defined by their tendency to epitaxially match the substrate-matrix, Fig.3b-d. For both Fe and Fe_3O_4(100), this epitaxial direction is along the equivalent [110] type of directions; Fe(200)||Fe3O4(220)||MgO(220) and Fe_3O_4(220) || Fe_3O_4(220)||MgO(220).

In summary, using various electron imaging and diffraction techniques we found that the polarity of MgO (111) surface significantly influences the growth of polar Fe_3O_4 (111) films by inducing phase separation within the film and at parts of the interface. This observation of phase separation is in marked difference to prior reports of uniform epitaxial growth of Fe_3O_4(111) polar films on metal substrates [2] and Fe_3O_4(100) polar films on neutral MgO(100) substrates [3]. [4]

References

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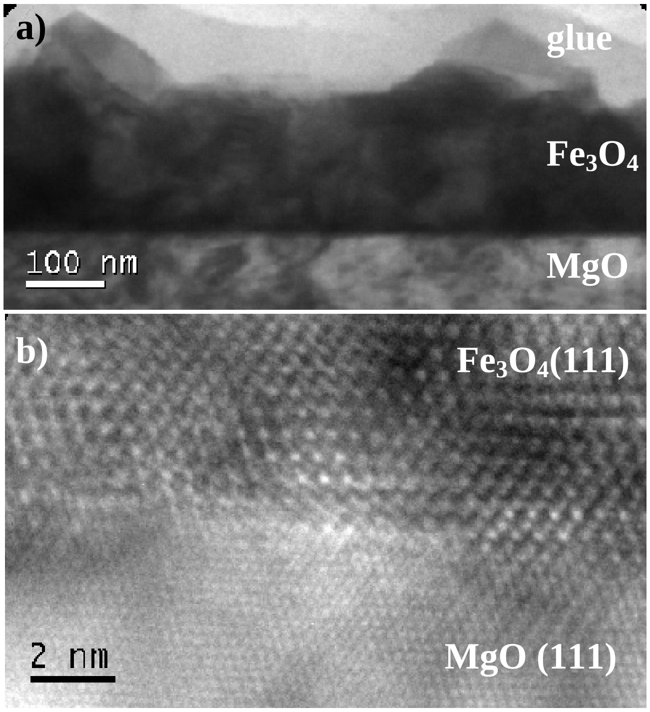

Fig1. TEM and HREM from cross sectional sample tilted to [1,-1,0] zone: (a) TEM image shows the film surface and interface morphology; (b) HREM image from the interface shows the epitaxy between $Fe_3O_4(111)$ and MgO(111).

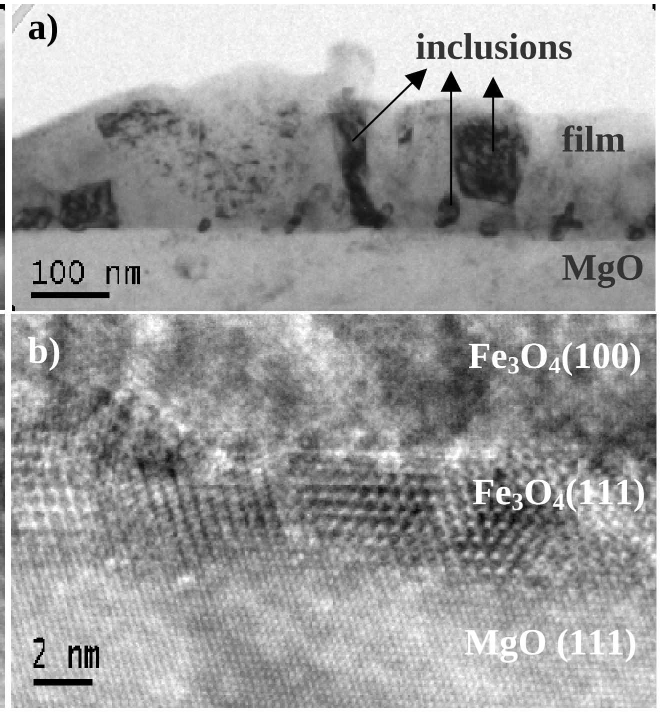

Fig2. (a) TEM image from cross sectional sample, tilted away from [1,-1,0] zone, shows presence of inclusions in the film.(b) HREM image shows an $Fe_3O_4(100)$ inclusion nucleated within the $Fe_3O_4(111)$ film close to interface.

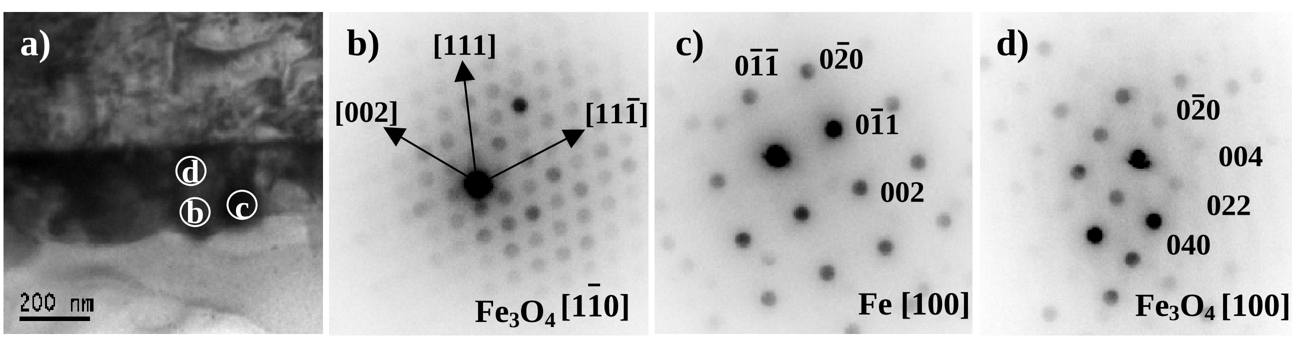

Fig3. TEM image and CBED patterns from film and inclusions: (a) TEM image in [1,-1,0] zone with labeled areas for CBED; (b) CBED pattern from $Fe_3O_4(111)$ film in [1,-1,0] zone shown as reference for orientation of inclusions; (c) CBED pattern from Fe(110) inclusion in [100] zone;. (d) CBED pattern from $Fe_3O_4(100)$ inclusion in [100] zone.

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