

FeO(111) formation by exposure of Fe(110) to atomic and molecular oxygen

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Abstract

The growth, structure, and morphology of ultrathin iron oxide layers formed on a Fe(110) single crystal surface are investigated by Auger electron spectroscopy, low energy electron diffraction, and grazing ion scattering. For Fe oxidation by atomic instead of molecular oxygen, the gas exposure can be reduced by almost two orders of magnitude because surface sticking and dissociation are not limiting the growth process. A well-ordered FeO(111) film with low defect density is only obtained with atomic oxygen. Compared to the bulk, the FeO lattice is laterally compressed by about 5–6% resulting in an in-plane oxygen (Fe) nearest-neighbor distance of 2.87 Å. Independent of the preparation method, long-range structural order is poor if the oxide film thickness is increased to 3–5 layers. This is attributed to the relatively large lattice mismatch between FeO(111) and Fe(110).

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1. Introduction

Due to potential applications in different areas ranging from catalysis to spin electronics there is a steadily growing interest in the research on metal oxide films and particles [1]. In order to obtain films with high quality and well-defined properties, various conditions have to be fulfilled. For the choice of substrate, crystallographic aspects and interface chemistry play an important role. In most cases, metal or oxide substrates have been used for film preparation [2]. A good correspondence between in-plane lattice constant and lattice symmetry of substrate and film is required. Additionally, migration of metal or oxygen species across the interface has to be considered. Chemically sharp interfaces may be obtained by using metal substrates which are inert against oxidation such as platinum [3].

Concerning the magnetic properties of metal oxide systems in reduced dimensions, much of the past and current research has focused on binary metal oxides (Me_xO_y) con-

taining the 3d transition elements Cr, Mn, Fe, Co, or Ni. Finite size effects on properties such as magnetic order, ordering temperature and interfacial moments strongly depend on crystalline structure and morphology. The latter, in turn, are to a great extent determined by the preparation method. Oxidation of a metal single crystal or a metal film is a simple method to obtain oxide films. Unfortunately, defect-free films with long-range structural order are hardly obtained which has been revealed by various investigations, e. g. with low energy electron diffraction (LEED) [3,4]. This deficiency is not surprising, because the lattice mismatch between the metal and the corresponding binary oxide is usually very large.

Binary metal monoxides containing Fe, Co, or Ni possess sodium chloride (NaCl) structure. Films with (111) orientation have been prepared by oxidation of Fe bcc(110), Co hex(0001), and Ni fcc(111) surfaces, respectively [4,5]. The surface of iron oxide formed on Fe(110) has been found to be ferromagnetically ordered with an orientation of the magnetization being antiparallel to that of the metal substrate [6–8]. Originally, this observation has been interpreted by an antiparallel alignment of the

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magnetizations of a FeO(111) surface and the underlying Fe(110) owing to a Ruderman–Kittel–Kasuya–Yosida (RKKY)-like interaction. A different explanation has been given in a subsequent study, where the observed magnetic behavior has been attributed to the formation of Fe₃O₄(111) instead of FeO(111) [8]. The experimental finding appears in a totally different perspective because, in the bulk, Fe₃O₄ is ferrimagnetic with a high ordering temperature, while FeO is antiferromagnetic with an ordering temperature appreciably below room temperature. The controversy concerning the properties of the Fe_xO_y(111)/Fe(110) system illustrates the importance of information on crystalline structure and stoichiometry for a correct understanding.

A sophisticated preparation method for binary compounds is oxide film growth by molecular beam epitaxy (MBE) where oxidation is performed during metal deposition [2]. For this “reactive MBE”, atomic and molecular oxygen as well as heteronuclear molecules such as NO₂ are used as oxidizing species. Supplying atomic oxygen has the advantage that no dissociation of the oxidizing gas limits the process of film growth. In the past decade, substantial progress has been made in the preparation and characterization of well-defined oxide films. Among these, the binary oxides of iron are important in a variety of contexts ranging from surface chemistry to thin film magnetism.

In this paper, we report on investigations of the chemical composition and the structural properties of ultrathin iron oxide layers prepared by oxidation with atomic and molecular oxygen as well as reactive MBE. With ion beam triangulation (IBT) [9,10], we obtain real-space information on the geometrical arrangement of atoms in the top-most surface layers.

2. Experiment

The experiments were performed in an ultrahigh vacuum (UHV) chamber at a base pressure of 5×10^{-11} mbar, attached via differential pumping stages to beam lines of two different electrostatic ion accelerators with energies up to 30 kV, or, alternatively, up to 350 kV. The Fe(110) single crystal disk was prepared by cycles of grazing sputtering with 24 keV Ar⁺ ions and subsequent annealing to 900 K, until the surface was clean and flat as checked by low energy electron diffraction (SPA-LEED, Omicron), Auger electron spectroscopy (AES), and polar angular distributions of scattered ions [11]. Auger electrons are induced either by 3 keV electrons at oblique incidence or by 200 keV protons scattered under a grazing angle of incidence $\Phi_{\text{in}} = 0.8^\circ$ from the surface.

Exposure of the well-prepared atomically clean Fe substrate to atomic or molecular oxygen was performed at partial pressures of several 10^{-8} mbar at a constant temperature of 420 K. As a source of atomic oxygen, a commercial thermal cracker (Model TC 50, Oxford Applied Research) was used which exploits thermal dissociation

on a ThO₂-coated filament. At relatively low temperature (<1000 °C), substantial dissociation of about 60% for oxygen is achieved. The gas exposure is given in Langmuir (L), where 1 L corresponds to 1×10^{-6} Torr s ($=1.33 \times 10^{-6}$ mbar s).

For reactive MBE, iron was deposited from a high-purity rod by electron beam heating in a commercial UHV evaporator (Focus EFM3) equipped with a current monitor to control the flux rate. The growth process was monitored in situ and in real time by recording the beam intensity of specularly reflected 20 keV He⁺ ions [12,13].

For ion beam triangulation, well collimated beams of 20 keV He⁺ ions were directed onto the target surface at a grazing incident angle $\Phi_{\text{in}} = 1.2^\circ$. The ion-induced emission of electrons as a function of the azimuthal angle θ is investigated by the uncompensated target current [9].

3. Results and discussion

3.1. Oxidation by molecular oxygen

Fig. 1 shows the intensity of specularly reflected 20 keV He⁺ ions as a function of exposure of the clean Fe(110) surface to molecular oxygen. We also give relative intensities of the oxygen KLL and the Fe LMM Auger signals, $I(\text{O}_{\text{KLL}} \text{ at } 510 \text{ eV})/I(\text{Fe}_{\text{LMM}} \text{ at } 651 \text{ eV})$, at certain stages of exposure. According to Auger electron cross sections, the $I(\text{O}_{\text{KLL}})/I(\text{Fe}_{\text{LMM}})$ Auger ratio of about 2.4 corresponds to iron monoxide (FeO) at a primary electron energy of 3 keV [14,15]. The probing depth of the O_{KLL}- and Fe_{LMM}-Auger electrons amounts to 10–15 Å [16] which corresponds to approximately 4–6 layers of iron oxide. A ratio of $I(\text{O}_{\text{KLL}})/I(\text{Fe}_{\text{LMM}}) = 1.5$ therefore indicates that a maximum of 3–4 metal oxide layers are formed on top of the metal Fe(110) substrate for exposures up to 145 L O₂. At the given substrate temperature of $T = 420$ K and oxygen pressure, the formation of Fe₃O₄ or Fe₂O₃ is unlikely which has been shown, e.g., by X-ray photoemission spectroscopy [4]. For thin oxide layers, the absence

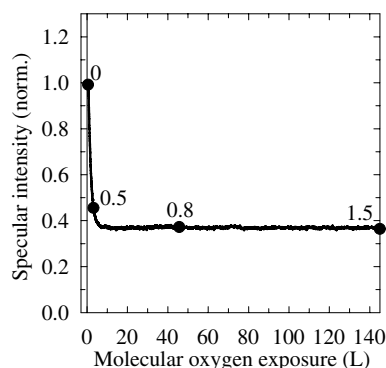


Fig. 1. Normalized intensity of 20 keV grazingly scattered He⁺ ions ($\Phi_{\text{in}} \approx 1.2^\circ$) with increasing exposure of clean Fe(110) substrate surface to molecular oxygen at $T = 420$ K. Numbers next to curve give oxygen-to-iron Auger ratio of $I(\text{O}_{\text{KLL}})/I(\text{Fe}_{\text{LMM}})$ at corresponding exposure.

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