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Structural study of epitaxial tungsten oxide nanoclusters

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Abstract

Tungsten oxide nanoclusters were prepared by evaporation of tungsten oxide powder from a specially designed evaporation cell under ultra high vacuum (UHV) conditions. The (0 0 0 1), ($\bar{2}$ 1 1 0) and ($\bar{1}$ 1 0 2) α-Al_2O_3 single-crystal substrate surface orientations were used as substrates. The structure and epitaxial parameters of tungsten oxide nanoclusters were determined by reflection high-energy electron diffraction (RHEED). Structural properties of tungsten oxide deposits were found to be dependent on the substrate surface orientation and deposition conditions. Well-oriented populations of WO_2 nanoclusters were observed. They exhibited preferentially (1 0 $\bar{1}$) WO_2 epitaxial planes.
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1. Introduction

Tungsten oxide is widely used in a variety of electrochromic devices [1], catalysts [2] and chemical sensors [2–5]. One of the characteristic properties of tungsten oxide is a strong dependence of its electrical conductivity on the oxidation state, which can vary from the wide-gap semiconductor state (for WO_3) to the conductor one (for WO_2). A number of other phases of WO_x exist between these two principal states. They differ not only in their composition and oxidation state but also in the crystallographic structure. The structure and morphology have a large influence on the sensitivity, selectivity and stability of the gas sensors and catalysts. Tungsten oxide thin films have been prepared by a variety of deposition techniques including thermal evaporation [3,6], chemical vapour deposition, sputtering [7–9] and sol–gel method [10].

The goal of this work was to prepare epitaxial well-defined tungsten oxide nanoclusters on sapphire single-crystal surfaces. The structure and epitaxial parameters of tungsten oxide thin films were investigated by reflection high-energy electron diffraction (RHEED).

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2. Experiment

Experiments were performed in an ultra-high vacuum (UHV) system equipped with a RHEED facility, permitting simultaneous observations of the sample surface and the overlayer formation during deposition. The RHEED instrument was operated at the acceleration voltage of 25 kV. Diffraction pattern was recorded by the RHEED–Vision Computer system (STAIB INSTRUMENTS). A movable sample holder made it possible to change both polar azimuthal angles of incidence of the primary electron beam onto the sample surface and heating up to the temperature of 1200 °C. Heating of the sample was performed by electron bombardment of a molybdenum sample holder. Before the experiment, the heating system was calibrated using a thermocouple. The sample transfer system permitted the rapid sample exchange without breaking vacuum in the UHV chamber.

The (0 0 0 1), ($\bar{2}$ 1 1 0) and ($\bar{1}$ 1 0 2) α-Al_2O_3 single-crystal substrate surfaces were prepared by cleaning in alcohol and, subsequently, heating at the temperature of 1200 °C in the air for 2 h. The substrate was introduced into the UHV chamber and annealed at the temperature of 300 °C for 1 h, at the background pressure of 1×10^{-7} Pa. The substrate surface quality was checked by RHEED.

Tungsten oxide thin films were grown in situ by deposition from a specially designed evaporation source. It operated on the principle of thermal evaporation of tungsten trioxide powder from a graphite crucible heated by electron bombardment. Tungsten oxide was deposited with a low evaporation rate of about 0.1 nm/min at a background pressure below 1×10^{-5} Pa. During deposition the substrate was maintained at different temperatures between room temperature and 600 °C. Only a small amount of material (from 1.5 to 2.0 nm) was deposited in order to obtain non-continuous nanostructured thin films. The tungsten oxide deposits were treated at different temperatures under UHV conditions.

3. Results and discussion

Before the deposition of tungsten oxide, the α-Al_2O_3 substrate surface was examined by the RHEED method. An example of the observed diffraction patterns is presented in Fig. 1a. An analysis of the diffraction pattern shows that it corresponds to the ($\bar{1}$ 1 0 2) α-Al_2O_3 surface with the [$\bar{1}$ 1 0 1] crystallographic direction parallel to the primary electron beam. The elongated streaks indicate a (1 × 1) structure of the substrate surface having very low roughness.

Tungsten oxide was deposited at different substrate temperatures from room temperature (RT) to 600 °C. As the next step, the samples were annealed under UHV conditions at different temperatures. Results for all samples are summarised in Table 1. The table shows that after the

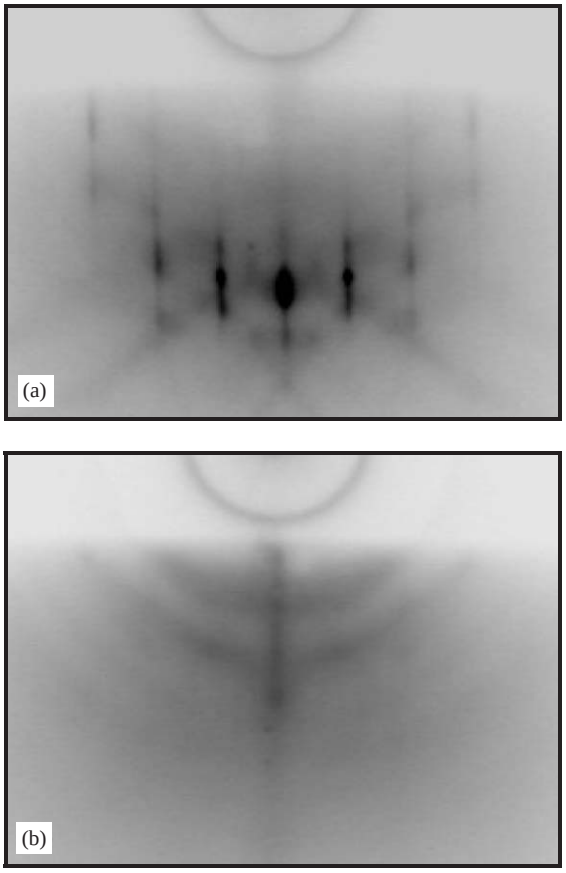

Fig. 1. RHEED diffraction pattern from (a) the clean α-Al_2O_3 ($\bar{1}$ 1 0 2) substrate surface and (b) after deposition of tungsten oxide. The primary electron beam was parallel to the [$\bar{1}$ 1 0 1] crystallographic direction. The diffraction semicircles correspond to the tungsten oxide polycrystalline phase.

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