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Nucleation and coexistence of nanometer-scale facet domains on O/Ir(2 1 0)

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Abstract

A study of faceting phenomena exhibited by the O/Ir(2 1 0) system has been conducted using low energy electron microscopy (LEEM). The initial nucleation and growth of three-sided pyramidal facets with {3 1 1} and (1 1 0) faces, as well as the conversion of planar Ir(2 1 0) into a completely faceted surface, are found to occur uniformly over the entire sample area. Facet relaxation on the clean faceted surface to form a planar surface is also found to proceed uniformly. Individual coexisting domains of planar and faceted surface, although present, cannot be resolved in LEEM at any stage of these transformations.

Keywords: Faceting; Iridium; Oxygen; Low energy electron microscopy (LEEM); Low energy electron diffraction (LEED)

1. Introduction

A variety of atomically-rough clean metal surfaces (e.g. body-centered cubic Mo(1 1 1), W(1 1 1), and face-centered cubic Pt(2 1 0), Ir(2 1 0), etc.) can be prepared as stable orientations, but the presence of (strongly interacting) adsorbates can cause changes in surface morphology through mechanisms such as reconstruction and *facet formation* [1–10]. The faceting effects are usually explained in terms of increased surface free energy anisotropy due to the adsorbate presence [11,12]. Changes in morphology can be accompanied by changes in electronic properties and surface reactivity [11,13,14]. Structure sensitivity in the surface chemistry of acetylene and in butane hydrogenolysis has been observed over Pd/W and Pt/W faceted surfaces [15,16].

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The planar Ir(2 1 0) surface covered with $\theta > 0.5$ monolayers (ML) of oxygen becomes completely faceted upon annealing to $T > 600$ K [17,18] and is characterized by an array of three-sided pyramids exposing {3 1 1} and (1 1 0) faces. Facet orientation does not depend on annealing temperature, but facet size increases upon annealing above 600 K. The scanning tunneling microscope (STM) image Fig. 1 shows a typical oxygen-faceted Ir(2 1 0) surface. Oxygen can be removed from the surface via CO oxidation at 550 K or reaction with H_2 to form H_2O at ~400 K, while preserving (''freezing'') the faceted structure. The *clean faceted* surface remains stable for $T < 600$ K and irreversibly relaxes to the planar state for $T > 600$ K. Thermal decomposition of acetylene and ammonia occurs readily on clean planar and faceted Ir(2 1 0) and exhibits differences in reaction rates that reveal the existence of structure sensitivity and particle (facet)-size effects in these reactions [19–21].

In a previous LEEM study of Pt/W(1 1 1) faceting [22,23] it was found that, under conditions of constant temperature (sufficiently high for facet formation) and increasing coverage, clearly resolved faceted domains (250–500 nm in size) nucleate on random regions of the surface, then grow, coalesce and eventually completely cover the surface. This mode of pyramid nucleation was explained in terms of the Pt 2D island growth: facets are formed solely on microscopic areas of the surface where the local coverage is $\theta = 1$ ML. Our previous LEED experiments on the O/Ir(2 1 0) system [17] have shown that the planar and faceted structures coexist under a range of temperature and oxygen coverage conditions. Of particular interest for the purposes of studying facet formation is the high coverage ($\theta > 0.5$ ML) region at $T \sim 450$–600 K. Under these conditions, LEED spot profiles indicate that small pyramids ($\leqslant 5$ nm) nucleate and grow while coexisting with relatively large planar areas ($\geqslant 10$ nm).

The facet formation and relaxation experiments on Ir(2 1 0) described in this Letter address general questions about faceting transformations: do they proceed via nucleation and growth of large spatially separated domains, as is the case for Pt/W(1 1 1), or can they also occur via a local nucleation and coexistence of small planar and faceted areas? All present evidence points to the latter explanation for oxygen-induced faceting of Ir(2 1 0).

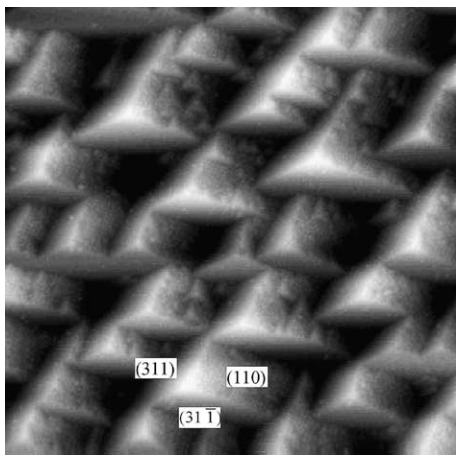

Fig. 1. A 100 nm × 100 nm STM image of the faceted surface prepared by annealing Ir(2 1 0) to ~1800 K in 1×10^{-7} Torr oxygen background. The vertical scale is ~2.8 nm. $V_g = 100$ mV, $I = 10$ pA.

2. Experimental

The LEEM experiments were performed at the Center for Microanalysis of Materials at the University of Illinois at Urbana-Champaign, using an IBM type I LEEM [24], housed in a standard stainless steel ultrahigh vacuum (UHV) system with base pressure $p_0 \approx 5 \times 10^{-11}$ Torr. Sample preparation can be performed in situ, or in a contiguous auxiliary chamber. By adjusting the sample bias, the incident electron energy on the sample can be set to a desired value (typically between 0 eV and 50 eV). LEEM and LEED images (electron signals) are enhanced by a micro-channel plate detector (MCP), viewed on a phosphor screen, and recorded with a CCD camera in the form of stills and motion pictures. The STM experiments (Fig. 1) are performed at Rutgers using a UHV variable temperature Omicron STM.

The sample is a single crystal disc ~1 mm thick and 8.75 mm in diameter, polished to a mirror finish within 0.5° of the fcc(2 1 0) surface plane. It was mounted in a Mo cup, allowing for high temperature anneals, but was separated from the cup by a