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Surface X-ray diffraction study of boron-nitride nanomesh in air

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Abstract

The hexagonal boron-nitride 'nanomesh' surface reconstruction on Rh(1 1 1) [Corso et al., Science 303 (2004) 217–220] has been investigated using surface X-ray diffraction utilizing synchrotron radiation. This unique structure has been found to be stable under ambient atmosphere which provides an important basis for technological applications like templating and coating. The previously suggested (12×12) periodicity of this reconstruction has been unambiguously confirmed and structural features are discussed in the light of the X-ray diffraction results.
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Keywords: Surface X-ray diffraction; Boron-nitride; Rhodium; Nanomesh

1. Introduction

At elevated temperatures borazine decomposes on Rh(1 1 1) to form a self assembled surface reconstruction of thermally very stable hexagonal boron-nitride (h-BN) [1]. The unit cell of this highly regular structure is, at 32 Å, huge by surface science standards. The detailed geometry of this 'nanomesh' is still under debate but major efforts are being taken to further investigate and utilize this unique system. This is attested, for example, by the formation of a specific targeted research project 'NanoMesh' supported within the sixth framework program (FP6) of the European Commission, a recent NanoMesh workshop, and the successful use of the nanomesh as template for the ordering of fullerenes [1].

Related but different structures of hexagonal boron-nitride have been reported for other surfaces like for example Ru(0 0 0 1), Pt(1 1 1) [2,3], Cu(1 1 1), Ni(1 1 1) [4] and Pd(1 1 0) [5]. From a fundamental and an applied point of view is it important to disentangle the delicate balance in the surface free energy between h-BN and substrate contributions and the processes that lead to the formation of this fascinating structure and related structures on other substrates. For applications, it is mandatory to have detailed knowledge of the atomic and electronic structure of the surface for utilizing it as an oxygen- and carbon-free template, e.g., for the production of nanocatalysts and nanomagnets.

To solve the structure of a reconstruction involving several hundred atoms the best technique currently applicable is surface X-ray diffraction (SXRD) [6–8]. X-rays easily penetrate the surface and are therefore sensitive to the structure of an extended surface region rather than only the topmost layer. The data can be analyzed within the kinematical framework and this facilitates solving such large reconstructions. However, due to the low scattering cross-sections of boron and nitrogen, is it necessary to perform such an experiment at a 3rd-generation synchrotron source.

With applications like templating and ordering of liquids in mind, we studied the nanomesh structure on

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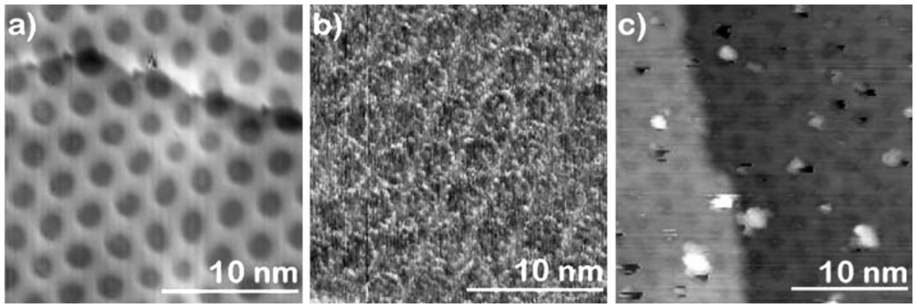

Fig. 1. Constant current STM images measured at $I_t = 1$ nA and $V_s = -1$ V. (a) STM image of the h-BN on Rh(1 1 1) nanomesh taken after preparation in UHV. The nanomesh survived 60 h air exposure as demonstrated by (b) STM and LEED images. (c) A short annealing up to 950 K is enough to remove a relevant part of the contaminants (as H_2O, O_2, CO_2, and CO) from the surface and bring the nanomesh back to its initial (pre-air exposure) configuration.

Rh(1 1 1) under ambient conditions. It turned out that the nanomesh is stable even under these extreme conditions, in contrast to typical surface reconstructions, which normally require ultra-high vacuum to be preserved.

2. Experimental

The samples were prepared in an ultra-high vacuum (UHV) system equipped with low energy electron diffraction and a scanning tunneling microscope (STM) [9]. The Rh(1 1 1) surface has been cleaned by repeated sputtering and annealing cycles. The surface was held at 1070 K and exposed to borazine at a pressure of 3×10^{-7} mbar, then subsequently cooled down to room temperature.

As a pre-study of the stability of the reconstruction STM investigations have been performed before and after a 60 h air exposure, see Fig. 1.

Freshly prepared samples for the X-ray experiments were transferred to the beamline and mounted under ambient conditions inside a chamber with Kapton windows. The chamber was flushed by a constant overpressure of helium to reduce the background scattering. The X-ray photon energy was set to 15.0 keV and the glancing angle of incidence to 0.20°. A dataset consisting of 816 fractional order and 17 integer order in-plane reflections was recorded. Details about the beamline, the surface diffractometer and the data acquisition using a novel 2D pixel detector can be found elsewhere [10,11].

The integrated intensity of the recorded reflections was determined and the standard geometrical correction factors applied [11]. Averaging reflections which are equivalent due to the p3m symmetry of the substrate yielded 402 fractional and nine integer order reflections. The systematic error determined in this standard averaging procedure [7] was as high as 74%, rendering a quantitative data analysis impossible. Among other things is the use of the stationary geometry without sample rocking scans [12] and scattering from the sample holder responsible for this. Nevertheless, valuable qualitative structural information can be obtained from the SXRD data.

In the following we use the conventional surface coordinate system with $\mathbf{a} = 1/2[1 0 \bar{1}]_{cubic}$, $\mathbf{b} = 1/2[\bar{1} 1 0]_{cubic}$, and $\mathbf{c} = 1/3[1 1 1]_{cubic}$. The cubic coordinates are in units of the rhodium lattice constant, 3.80 Å at 300 K.

3. Results and discussion

One of the main results of our investigations is the stability of the nanomesh reconstruction under ambient atmosphere. As an example, the effect of a 60 h exposure to air is shown in Fig. 1. The basic structural elements can still be recognized after this prolonged exposure. After removing the adsorbates by a short annealing at 950 K, the original structure is restored. This means that this unique surface reconstruction is an ideal candidate for practical applications, such as its use as a template for the production of nanomaterials.

That the nanomesh structure is a commensurate reconstruction with a (12×12) unit cell has been confirmed by in-plane scans along directions of high symmetry. An example is shown in Fig. 2. The (13/12 0) peak of the reconstruction is of course weak but clearly observable and exactly at the expected position.

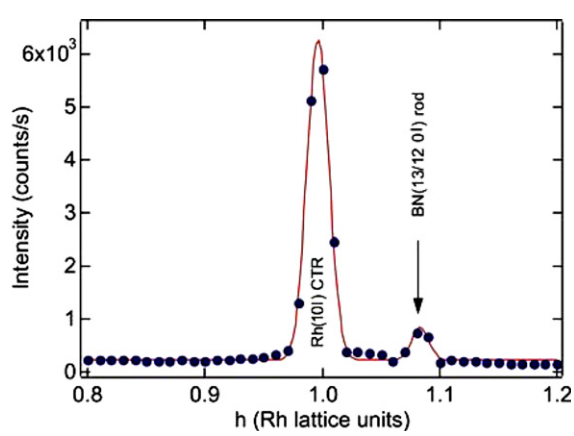

Fig. 2. SXRD intensity profile recorded in the plane of the surface ($l = 0.07$) at constant, $k = 0$. The (1 0 0.07) peak of the Rh(1 1 1) substrate and the (13/12 0 0.07) peak of the nanomesh reconstruction are observed, thereby confirming a commensurate superstructure with a (12×12) Rh(1 1 1) unit cell.

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