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Field ion microscopic observations of LaB_6 on tungsten

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Abstract. The importance of field ion microscopy as a unique surface microscopic technique has been pointed out with particular reference to the lanthanum hexaboride (LaB_6)-deposited refractory metal cathodes. In the core, field ion microscopic observations of LaB_6 deposited tungsten are described. The observations are discussed in relation to the field electron emission microscopy of LaB_6/W system. The paper ends with a few comments on the scope of further study of this or a similar system using the field ion microscopy and the atom probe field ion microscope.

Keywords. Field ion microscopy; lanthanum hexaboride; tungsten field evaporation; atomistic behaviour.

1. Introduction

In the studies of atomic adsorption on a substrate lattice, it is usually difficult in practice to define the structure of the substrate on atomic scale. The field ion microscope (FIM) allows us to observe individual atoms on an atomically smooth surface on various crystallographic planes in a single experiment (Muller and Tsong 1969).

Lanthanum hexaboride (LaB_6) has been a material of interest because of its excellent electron emissive properties (Lafferty 1951). Field electron emission properties of LaB_6-deposited tungsten have been investigated earlier (Dharmadhikari *et al* 1977, 1979). The adsorbed species were observed to agglomerate on the regions around {111} and {001} planes of tungsten. The work function of the composite surface also reduced, as in any other case of electropositive adsorption in general. Studies on coadsorption of lanthanum and boron by Okuno *et al* (1978) by field emission microscopy also suggested that lanthanum is located outside the substrate surface, boron remaining intermediate. It was, therefore, important to study the adsorption of LaB_6 on tungsten using FIM in order to shed more light on the atomistic behaviour.

2. Experimental

An all-metal bakeable chamber, evacuated to 10^{-8} torr by a combination of oil diffusion pump and a sputter ion pump, was used for this purpose. The specimen was mounted on a specimen manipulator and cooled to liquid nitrogen temperature by means of a copper braid. The microscope was equipped with a microchannel plate image intensifier and a fluorescent screen. The LaB_6 source was made by cataphoretic deposition of LaB_6 powder onto a clean tungsten spiral. The source was mounted on a three pin feed-through to facilitate resistive heating. A molybdenum cylinder around the source served as a mask to avoid unwanted deposition on insulating parts. The source was degassed thoroughly in vacuum prior to deposition. Helium was used as an

imaging gas, which could be admitted through an all-metal valve. The tungsten wire was annealed at 1500°K in vacuum, before making the tip by electrochemical etching.

Initially, the tip was imaged and field evaporated to obtain the end form. The chamber was then re-evacuated to 10^{-8} torr and the specimen was manipulated to face the source. The adsorbate dose was pre-fixed by choosing the heating current and the duration of heating. The tip could be heated to a desired temperature by resistive heating in order to equilibrate the adsorbate over the surface. It was then manipulated back and the image was observed by re-admitting the imaging gas to a pressure of 10^{-5} torr. Temperatures of the tip and the source, when heated, were measured by optical pyrometer.

3. Results

3.1 *Clean tungsten*

Figure 1(a) shows the FIM of field evaporated tungsten surface cooled down to liquid nitrogen temperature, imaged at the helium pressure of 10^{-5} torr, in static mode.

3.2 *In situ deposition*

The adsorbate source was heated to 2100°K for 10 sec with the tip held at liquid nitrogen temperature. During deposition, the tip temperature was expected to rise by not more than 50°K. Figure 1(b) shows clusters of the adsorbate atoms, randomly deposited on tungsten.

3.2a *Substrate heated to* 1200°*K for* 30 *sec:* Three more doses, equivalent to the first one, were deposited onto the tip which was subsequently heated to 1200°K for 30 sec. The pattern in figure 1(c) appeared at the *best imaging voltage* (BIV) 2·3 kV. This pattern shows clusters of adsorbate on regions surrounding {121} planes of the substrate.

3.2b *Substrate heated to* 1650°*K for* 30 *sec:* Lanthanum hexaboride was again evaporated from the source and the substrate tip heated to 1650°K for 30 sec. The specimen was manipulated to bring one of the {001} planes in the field of view, as seen in figure 1(d). Clearly, growth is observed on {111} and {001} planes. Due to migration of the substrate tungsten atoms, the tip got blunted.

3.3 *Field evaporation*

As the field evaporation was carried out, the BIV rose steadily, the pattern remaining stable after the removal of every layer. However, the evaporation voltage was always only slightly higher than the imaging voltage and less when compared with the evaporation voltage for clean tungsten. Figure 1(e) shows the adsorbate covered surface with a few layers of adsorbate removed. Epitaxially grown layers on the {111}, {001} planes and the terraces joining {111} and {121} planes are clearly depicted. Figure 1(f) shows the field evaporated surface with a few interstitial atoms, particularly in the {011} planes. A few displaced atoms are also seen. The surface structure becomes more and more ordered after subsequent field evaporation.

At this stage, the {011} and the {111} and {001} planes had different BIV.

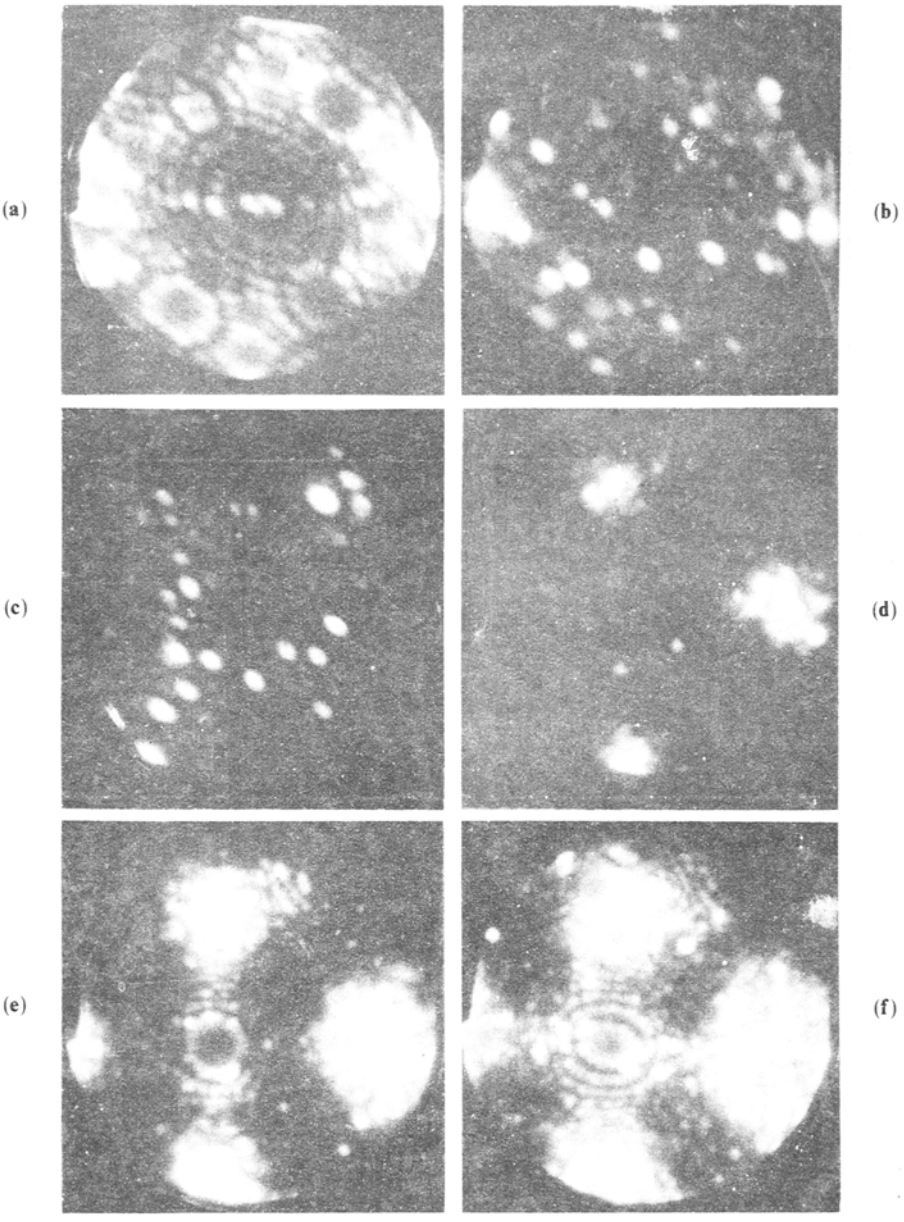
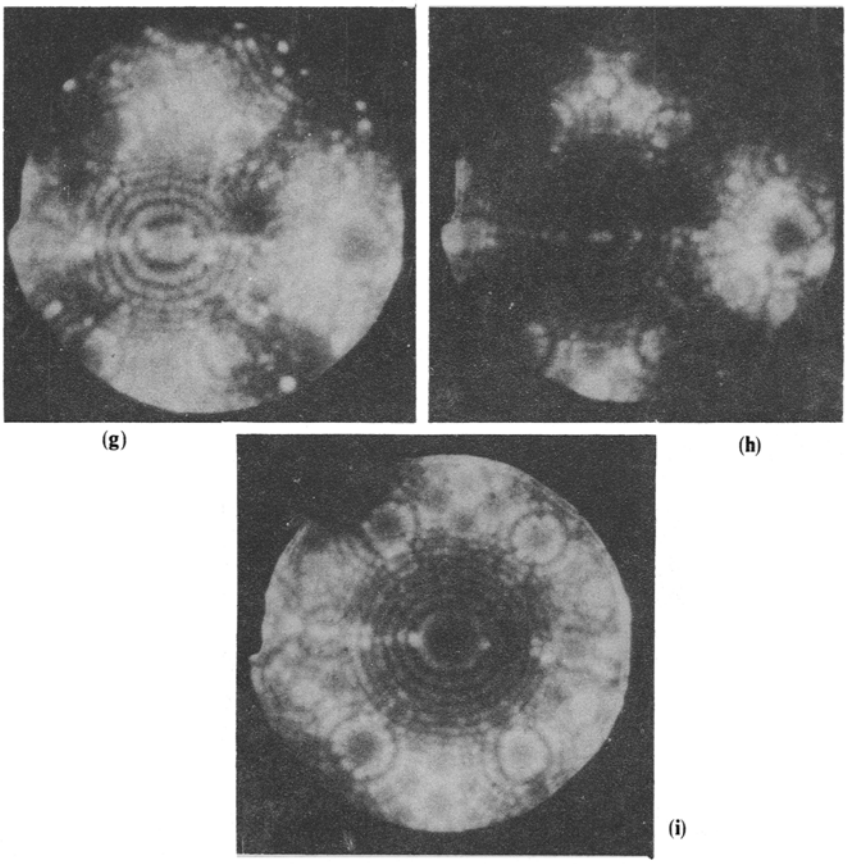


Figure 1a–f. **a.** Image of the field evaporated tungsten surface cooled down to liquid nitrogen temperature at the helium pressure of 10^{-5} torr (BIV 2·76 kV). **b.** Clusters of the adsorbate atoms (LaB_6), randomly deposited on tungsten (BIV 2.5 kV). **c.** Clusters of adsorbate on regions surrounding 121 planes of the substrate. Substrate heated to 1200 K for 30 sec at BIV 2·3 kV. **d.** Image of the blunted tip showing the segregation on {111} and {001} planes (substrate heated to 1650 K for 30 sec, BIV 3·66 kV). **e.** Image of the adsorbate covered surface with a few layers of adsorbate removed (BIV 4·4 kV). **f.** The field evaporated surface with a few interstitial atoms, particularly in the {011} planes.



Figures 1g–i. **g.** Dependence of field evaporation on BIV ({011} planes best imaged; BIV 5·2 kV). **h.** Dependence of field evaporation on BIV ({111} and {001} planes best images; BIV 4·4 kV). **i.** Image of the clean tungsten pattern after continued field evaporation (BIV 5 kV).

Figures 1 (g, h) depict this difference. This could be attributed to the difference in local curvatures.

Continued field evaporation led to the clean tungsten pattern seen in figure 1(i).

3.3a *Further heating of the tip:* It was observed that heating the tip in vacuum at 1200°K did not result into blunting phenomenon. Also the adsorbate deposited on the tip shank migrated to the {111} and {001} planes due to heating and the same sequence of patterns during field evaporation was observed.

4. Discussion

4.1 *Epitaxial growth*

Reproducible epitaxial layers are formed on {001} and {111} planes and terraces between {001} and {121} planes. The BIV dropped to a low value as compared to tungsten. The observed growth was compatible with our earlier investigations using field electron emission microscopy.

4.2 *Substrate-adsorbate interface*

The tungsten surface stripped off all the adlayers shows deformed atom rings. This could be caused by the lattice deformation on heating the substrate to a temperature at which migration of substrate atoms sets in. This results in the reconstruction observed in these patterns (Bassett 1965). It was observed that the removal of three to four {111} layers again resulted into well-ordered tungsten pattern.

4.3 *The imaged species*

Field evaporation from the overgrown regions occurred at voltages well below that required to remove tungsten atoms from the otherwise clean surface. Thus, the adlayers proved to be marginally stable for the helium ion microscopy at 78°K. The behaviour of the adlayers is typical of metallic adsorbates. Electronegative adsorbates usually are not imaged (Forbes 1971). Lanthanum is known to be electropositive and boron electronegative with respect to tungsten. The co-adsorption studies of lanthanum and boron on tungsten (Okuno *et al* 1978) and our earlier results using field electron emission microscopy of LaB_6/W system (Dharmadhikari *et al* 1977, 1979), indicated that lanthanum layer at the co-adsorbed surface is responsible for the observed lowering of work function of the composite surface. Also, the ionization probability is higher at the sites of enhanced positive charge. Geometrically, boron atoms being smaller in diameter as compared with tungsten, can fill the surface interstitials, while the lanthanum atoms being large in diameter as compared with the other two, remain outside the surface contributing to the observed image. All these factors tend to suggest that the imaged species are lanthanum atoms.

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