INDIUM EPITAXY ON AN IMPERFECT FEM/FIM TUNGSTEN EMITTER*

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Some results of a study of epitaxial growth of indium crystals, deposited from vapour under ultra high vacuum onto a tungsten tip surface, are presented. FEM/FIM techniques have been employed for this investigation. The presence of a few different degrees of perfection of the W substrate surface enabled us to find that the growth of epitaxial In crystals preferred only an atomically smooth surface with the highest degree of lattice perfection obtained by field evaporation of the W tip. Epitaxial indium layers were grown at a tip temperature of 375 K and were oriented as follows: (111) In || (110) W with [011] In || [111] W. The field emission pattern of an In single crystal and orientation relationships are briefly discussed.

1. *Introduction*

Field ion microscopy (FIM) and field emission microscopy (FEM) methods are very useful in the investigations of epitaxy phenomena. There are several papers on epitaxial growth using the FIM and FEM techniques. Systems which have been studied, so far, include Ag–W [1–8], Cu–W [9–11], Pt–Ir [12,13], Ni–W [14,15], Zn–W [17], Zr–W [18], Ir–Mo [19], Fe–Ir and Au–W [20], Pt–W [16], Cd–W [21], alkali metals on W [22–24], Al_2O_3–W [25] and WO_3–W [26].

In this paper some results of a study of the In–W system are reported. This part is devoted to the study of the influence of the W surface structure on the indium epitaxial layer. Growth of indium single crystals on a tungsten tip surface was investigated by deposition of indium from vapour in ultra high vacuum.

2. *Experimental*

A fully bakeable ultra-high vacuum FEM/FIM system was designed and built for this project. Background vacuum obtained by two PZK-20 sputtering ion pumps and a molybdenum getter was normally better than 1×10^{-9} Torr (as measured by a Bayard-

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-Alpert gauge). Helium gas for FIM image formation was introduced through hot quartz glass. The molybdenum glass FEM/FIM tube, operating at liquid nitrogen temperature, was used. The tungsten emitter, made of a 0.1 mm diameter wire, was spot welded to a molybdenum loop of a 0.2 mm diameter wire. Current and potential leads enabled one to control the temperature of the specimen tip which could be determined to within ± 20 K. The tungsten emitters were heated in situ for several hours at approximately 1000 K to eliminate contaminations from the emitter shank.

The indium vapour source was a cone shaped coil of 0.4 mm diameter tungsten wire, which could be resistively heated. The source assembly was heated at 2000 K in high vacuum for several hours before filling up with indium of 5 N purity. The pressure of In vapour above the liquid metal is 5×10^{-3} — 10^{-2} Torr at a temperature in the range of 900–950°C. Treating the source as a Knudsen-cell, an estimated value of the In beam density near the tip s rface amounted to 10^{15}–10^{16} atoms/cm^2 sec.

By counting atom rings [27], the local radius of tungsten tip curvature between (110) and (123) crystal planes was found. The radius of curvature of the In epitaxial layer was estimated using the Fowler-Nordheim plots.

3. *Results and discussion*

The tungsten emitter tip which had been applied in this work was not a single crystal as usually but a bi-crystal with a distinct grain boundary. The tip was situated in relation to the In atom source in such a way that only one of W single crystals was exposed directly to the indium atom beam.

An FIM pattern of the substrate surface is shown in Fig. 1. The FIM micrograph was made after heating the tip at 1000 K and after field desorption of several layers. The surface consisted of three parts. The first one (marked I in Fig. 1) was the area of the W crystal exposed directly to the indium atom beam. The second one (marked II) was the surface of the W crystal situated on the shaded side. The third one (marked III) was a disordered surface region of the second part of the tip surface. It was very hard to develop a perfect crystal surface in the area of this disordered region, since it had been a remainder of a deep "hole" which appeared during the process of electropolishing of the emitter tip. Only region II was perfect and smooth on an atomic scale. The local radius of this surface region was 450 Å.

The average work function of the clean W tip used, determined from the slope of F–N plots, was equal to 5.0 eV. The geometrical factor κ in the expression for field strength $F = U/\kappa r$ (where U is the applied voltage and r — tip curvature radius) was determined from the ratio of applied helium FIM best image voltage to the helium FIM best image field which was assumed to be 4.4 V/Å [27]. Such a large value of the average work function could be caused by the unusual geometry of the tip (bi-crystal) and/or possible segregation of contaminations to the grain boundary from the specimen bulk.

Single crystals of indium were grown on a tungsten emitter tip held at 375 K and with indium flux density of the order of 10^{15} atoms/cm^2 sec. The emitting area of the In crystal was comparable to that of the clean tungsten emitter. Fig. 2 shows one of the obtained

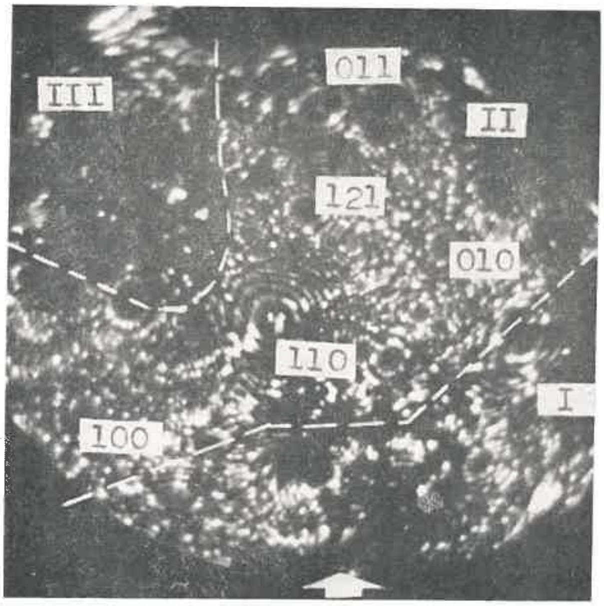

Fig. 1. Helium-ion micrograph of a clean tungsten tip at best image voltage (21.0 kV). The arrow shows the direction of evaporation

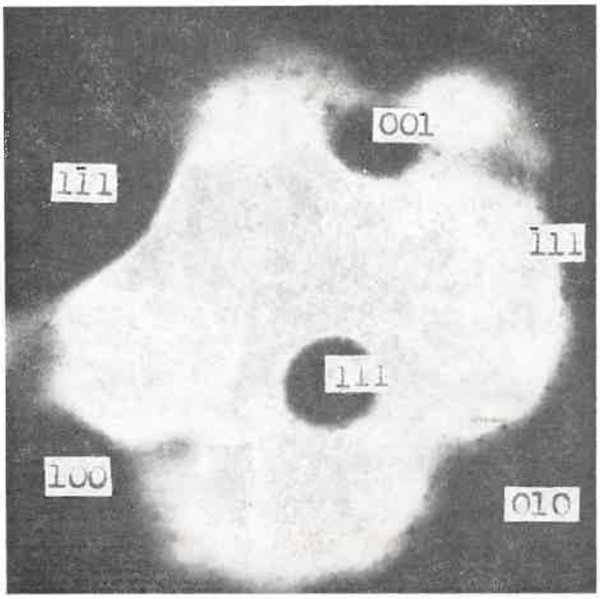

Fig. 2. Emission pattern of In epitaxial layer at 78 K; tip temperature during deposition 375 K; time of deposition 600 sec

indium single crystals. The radius of curvature of the indium layer of about 400 Å was calculated using the slope of the Fowler-Nordheim plot. The geometrical factor for indium crystal was treated as equal to that for the clean tungsten tip. Generally, such an assumption is not reasonable. In this case, however, the authors believe that the supposition does hold because of the observed shape of the discussed indium layer.

Basing on a comparison of the obtained emission pattern (Fig. 2) to the stereographical projection of (111) oriented In crystal lattice (Fig. 3) the investigated indium layer was identified to be an In single crystal. Indium crystallizes with fcc crystal lattice tetragonal

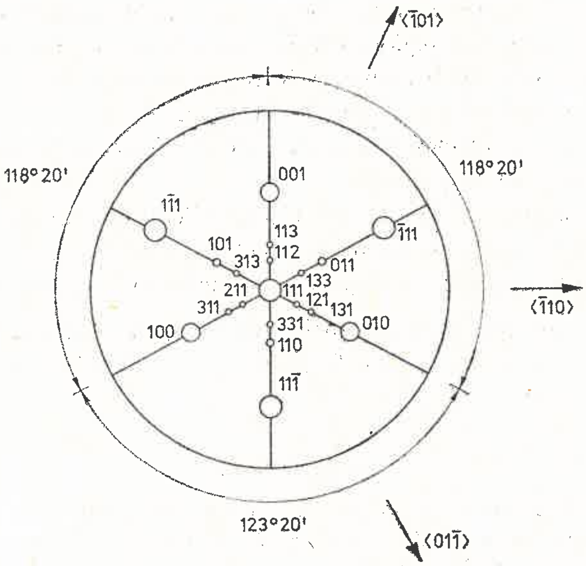

Fig. 3. (111) oriented stereographical projection of the indium lattice (tetragonal deformed fcc lattice with $c/a = 1.0758$)

deformed with the ratio of $c/a = 1.0758$ [29]. The closest packed crystal planes in this structure are the {111} and {001} ones. In Fig. 2 they are seen as dark spots of the emission pattern. It should be noted that in the case of the indium lattice the {111} plane types have two-fold symmetry rather than three-fold one since the angles between ⟨110⟩ zones are not identical (see Fig. 3). In the stereographical projection only one of the {100} plane types has a two-fold symmetry. This is a result of the above mentioned deformation of fcc crystal lattice and it has been observed in the obtained FEM patterns of In layers.

Comparing the FEM pattern of the tungsten substrate surface (Fig. 1) to the FEM one of the indium layer one can see (Fig. 2) that the investigated In crystal was oriented relative to that W surface region which had been regular and smooth (region II in Fig. 1). The In layer orientation is: (111) In|| (110) W with [110] In|| [111] W.

Such an orientation relationship was observed usually. Since this perfect region had not been exposed directly to the In atom beam one can believe that the surface diffusion of indium species played an important role during formation of the indium crystal. No indium layer was oriented relative to the I and III surface regions of the tungsten substrate. The In epitaxial layer covered the grain boundary (separating regions I and II) and no distinct influence of the latter on the emission pattern of the indium crystal structure was found.

The nucleation of indium took place on the ledges of (110) tungsten crystal plane. This was found by the FEM observations of initial stages of In crystal growth.

4. *Conclusions*

(*i*) The growth of indium epitaxial layer prefers an atomically smooth W substrate surface rather than the surface regions which have a lower degree of lattice perfection.

(*ii*) The presence of a grain boundary in the substrate surface does not seem to influence the final structure of the obtained In crystal.

(*iii*) Surface diffusion of indium species (with the substrate held at 375 K) is an important factor in the growth process.

(*iv*) The obtained relationship of the crystal orientation differs from those [1, 11, 14, 19] which could be expected for fcc/bcc metal systems in spite of the fact that the indium crystal lattice deviates only slightly from the regular fcc lattice.

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