

PS12.01.10 A NEW METHOD OF THE IDENTIFICATION OF CRYSTALLINE-AMORPHOUS INTERFACES BY QUANTITATIVE EVALUATION OF HREM IMAGES. N.I. Borgardt and B. Plikat*, Moscow Institute of Electronic Technology, 103498 Moscow, Russia, *IV. Physikal. Institut der Universität Göttingen, Bunsenstr. 13-15, 37073 Göttingen, Germany

Identification of the atomic structure of interfaces between amorphous and crystalline phases by quantitative evaluation of HREM-images of cross-sectional samples is not straight-forward. This is due to the fact that the projected potentials of the atoms in the amorphous layer overlap randomly if the distance between them exceeds the length of short range order (about 1,5 nm). As a result, the contrast of interface steps can be blurred to different extents.

Intensity fluctuations caused by the random overlap of the projected potentials of the atoms in the amorphous layer can be eliminated if the two-dimensional intensity distribution of a homogeneous region of the interface is averaged along the interface. A theoretical analysis shows that the shape of the intensity profiles obtained by this method depends on:

- i.) the distribution of steps at the interface and
- ii) the averaged distribution of the amorphous layer atoms in the vicinity of the crystal boundary atoms.

Properties of the interface structure can be obtained by comparison of theoretical and experimental intensity profiles for various defocus settings. In order to simulate stepped interfaces the EMS multislice program [1] was supplemented to take into account the short range order of the amorphous layer. For this purpose the averaged distribution of atoms in the amorphous layer as a function of the distance to the interface is calculated from the radial distribution function as obtained from X-ray or neutron scattering. Comparisons of experimental images of a c-Si/a-SiO₂ interface and simulations based on the proposed method were carried out.

1. Stadelman P.A. (1987) Ultramicroscopy 21, 131

PS12.01.11 INVESTIGATION OF STORED ENERGY IN TiN-FILMS BY THIN FILM DIFFRACTION H. Wulff, C. Eggs* E.-M.-A.-University, Institute of Physical Chemistry, 17489 Greifswald, Germany* E.-M.-A.-University, Department of Physics, 17489 Greifswald, Germany.

Thin TiN films were deposited using a Hollow Cathode Arc Evaporation Device (HCAED) for studying the influence of low energy ion bombardment on film growth and film properties. Films were deposited at various nitrogen gas flows and negative substrate voltages at a defined discharge power, and investigated by thin film x-ray diffraction and x-ray photoelectron spectroscopy.

From the broadening and shifting of x-ray lineprofiles the dislocation densities and the concentration of interstitials in the films were calculated. With the energies of individual dislocations and interstitials a calculation was made of the energy stored in the films caused by low energy ion bombardment. The results were compared with investigations of the energy transfer to the substrate during titanium respective titaniumnitride deposition in HCAED [1].

[1] H.Steffen, H. Kersten, H.Wulff, J.Vac.Sci.Technol. A 12 (1994) 2780

PS12.01.12 THE INTERFACIAL STRUCTURE AND THE PREFERRED ORIENTATION OF LAYERED PEROVSKITE SrBi₂Ta₂O₉ THIN FILMS GROWN ON THE Pt ELECTRODE. Jeong Soo Lee*, Hyun Ha Kim*, Hyun Ja Kwon*, Seshu B. Desu#, *LG Electronics Research Center, 16, Woomyeon-dong, Seocho-gu, Seoul 137-140, Korea, #Department of Materials Science and Engineering, Virginia Polytechnic Institute and State University, Blacksburg, VA 24061-0237, USA

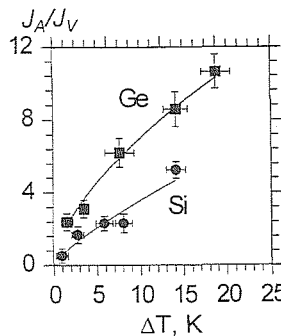
The influence of the Pt bottom electrode orientation on the growth of ferroelectric SrBi₂Ta₂O₉ (SBT) films was interpreted in terms of the atomic matching at the interfaces. The symmetry of SBT can be described as either an orthorhombic (SBT_{orth}) or a pseudo-tetragonal (SBT_{tet}) structure. The preferred orientations of the SBT layers were determined by comparing the experimentally observed x-ray $\theta-2\theta$ diffraction profiles with the simulated powder diffraction one. A highly (00l) prefer oriented SBT_{tet} film was grown when the Pt layer was mainly (001) oriented. There are three kinds of SBT_{tet} (00l) plane with different atomic configurations. All these atomic planes match pretty well with the Pt (001) plane both energetically and crystallographically if the following orientation relationship is satisfied; Pt (001) // SBT_{tet} (00l) // SBT_{orth} (00l), Pt [100] // SBT_{tet} [100] // SBT_{orth} [110], and Pt [010] // SBT_{tet} [010] // SBT_{orth} [110]. The mismatches in the [100] and [010] directions of the SBT_{tet} are quite small (-0.74%). The (110) and (105) SBT_{tet} peaks were predominantly found when the Pt electrode was (111) oriented. It was calculated that the Pt (111) plane exhibits a relatively good matching with either one of the two kinds of SBT_{tet} (110) plane or the SBT_{tet} (105) plane. The following orientation relationships are proposed; i) Pt (111) // SBT_{tet} (110) // SBT_{orth} (010), Pt [110] // SBT_{tet} [110] // SBT_{orth} [100], and Pt [112] // SBT_{tet} [001] // SBT_{orth} [001], ii) Pt (111) // SBT_{tet} (105) // SBT_{orth} (115), Pt [110] // SBT_{tet} [010] // SBT_{orth} [110], and Pt [112] // SBT_{tet} [501] // SBT_{orth} [551].

PS12.01.13 CORRELATION BETWEEN SURFACE AND VOLUME: A METHOD OF THE CRYSTAL HABITUS DESCRIPTION A. M. Ivanov and Yu. M. Smirnov Tver State University, 170000 Tver, Russia

The habitus and morphology of crystals are subject to considerable changes by the melt supercooling. In [1] we described a method enabling the analysis of the volume and surface changes during the crystal growth. The method is based on the comparison of the flux densities of extensive quantities. These quantities are presented by surface (area) A and volume V .

$$\text{The main equation is } \frac{J_A}{J_V} = \frac{\Delta A}{(\Delta V)^{2/3} (\Delta \bar{V})^{1/3}}$$

where J_A, J_V are the flux densities, $\Delta A, \Delta V$ are the changes of A and V at the interval of time, and $\Delta \bar{V}$ is the averaged volume of the crystal.



Shown in the figure is the dependence of the defined parameter on the melt supercooling ΔT for germanium (Ge) and silicon (Si). The J_A/J_V increases with supercooling and morphology in the following order: antiskeleton - face form - skeleton - dendrite.

This method was applied to the study of ceramics [2] prepared from natural raw materials. The samples were interpreted as conglomerates

composed of "skeleton" and "filler" phases. Following this approach the variation of the phase ratio was determined as a function of the firing regimes and concentration of the fusing agents. The dependences obtained appear to have a good correlation with the mechanical properties of ceramics.

The results of the present study indicate that the method employed is of advantage for both single crystals and polycrystalline materials.

[1] Yu. M. Smirnov and G. S. Blokhina. Melt supercooling and crystal morphology. In: *Physics of Crystallization* (Kalinin, KSU, 1983), p. 13.

[2] Yu. M. Smirnov and A. M. Ivanov. Classification of clay additives. In: *Physics of Crystallization* (Tver, TSU, 1994), p. 86.

PS12.01.14 PARTICULAR SINGULAR FACES IN SINGLE CRYSTALS. A. I. Kolesnikov and Yu. M. Smirnov, Tver State University, 170000 Tver, Russia

In the course of the investigation of the growth, morphology and structural defects in germanium single crystals we have defined the main morphological features of dislocation-free germanium crystals [1].

The single crystals were grown in the $\langle 111 \rangle$ crystallographic direction by the Czochralski method. The crystal-melt interfaces of the crystals grown were flat mirror singular $\{111\}$ faces (F -planes according to the Hartman's conception of PBS vectors). In addition, also observed were narrow stripes of $\{110\}$ faces on the lateral sides of the crystals (S -planes according to the above conception).

On the basis of the study of dislocation-free germanium and silicon single crystals we arrived at a principle of a particular singular face. These faces are singular, but are characterized by the least possible specific surface free energy. For germanium and silicon these are faces with the crystallographic symbol $\{111\}$. The free energy of the germanium $\{111\}$ face is $0.85-1.10 \text{ J}\cdot\text{m}^{-2}$, while the free energy of the $\{110\}$ face is $1.30-1.35 \text{ J}\cdot\text{m}^{-2}$. For other faces the free energy is considerably higher.

The principle was realized in the development of the growth process of paratellurite (tellurium dioxide) single crystals. As was deduced theoretically, in paratellurite single crystals the face of minimum specific free energy is face $\{110\}$, the energy of faces $\{101\}$, $\{111\}$, $\{201\}$ and others being considerably higher. We were able to obtain paratellurite single crystals as the growth pyramids of particular $\{110\}$ face. The single crystals were 80 mm in diameter and 80 mm in height. The samples obtained were free of three-dimensional structural defects (bubbles), while the dislocation density was below $3 \cdot 10^3 \text{ cm}^{-2}$ [2].

[1] Yu. M. Smirnov. The growth of dislocation-free germanium single crystals. *Nonferrous Metals (USSR)*, 1977, No.5, p.48 (in Russian).

[2] Yu. M. Smirnov and A. I. Kolesnikov. The conception of particular singular face (after the example of paratellurite). In: *Physics of Crystallization* (Tver, TSU, 1994), p. 24 (in Russian).

PS12.01.15 FIELD ION MICROSCOPIC OBSERVATION OF Si-SiO₂ INTERFACE. M. Umeno, M. Tagawa, N. Ohmae and M. Miyanaga, Department of Material & Life Science, Osaka University, Yamada-oka, Suita, 565 JAPAN.

The atomic arrangement of interfacial layer of Si-SiO₂ system was directly observed with a field ion microscope (FIM). The existence of some structural and/or compositional transition layer at the interface of a-SiO₂ and Si substrate has been pointed out by many reports, yet the direct observation of its structure has been of great concern. Si tips with $[001]$, $[011]$ and $[111]$ axes were prepared with chemical or electrolytic etching from CZ-Si wafers. After some successive treatments for obtaining atomic FIM images, tips were thermally oxidized with 300L of dry oxygen at 1000K in the FIM chamber. The image of an interfacial transition layer was easily recognized, as the oxide image which was composed of diffusely at random distributed bright spots change into the image of dense rather small spots when field evaporation was conducted, and the image of Si substrate partly appeared. The present FIM observation revealed followings:

1. The thickness of the transition layer is so thin as 2 to 3 atomic layers.
2. The transition layer has a crystalline nature.
3. The evaporation field strength has an orientation order, that is $(001) < (011) < (111)$, and this order does not correspond to either of the orientation order for oxidation velocity or of the field strength on the tip.
4. The evaporation field strength of SiO₂ was estimated to be 34V/nm which is somewhat lower than 40V/nm of Si.

A fairly perfect image of substrate Si could be obtained when all oxide layer was removed by the field evaporation.

PS12.01.16 INFLUENCE OF H₂, O₂- AND Ar-PLASMA ON THIN Al₂O₃-FILMS A. Quade, C. Eggs*, H. Wulff, M. Schmidt** E.-M.-A.-University, Institute of Physical Chemistry, 17489 Greifswald, Germany * E.-M.-A.-University, Department of Physics 17489 Greifswald, Germany **E.-M.-A.-University, Institute of Low-Temperature Plasma Physics, 17489 Greifswald, Germany

We have investigated the influence of H₂-, O₂- and Ar-plasma on the formation and stability of alumina layers on aluminium. The Al-films were deposited on smooth Si (100) wafer. The thickness of the films varies between 20 and 65 nm. An alumina layer of about 2 nm grew on the surface of the Al-films by atmospheric corrosion. The thin films were treated in a plasma of a HF-discharge (100 kHz, 100 W) with a gas pressure of 1 mbar and a flow rate of 5 sccm.

The layers were investigated with grazing incidence x-ray reflectometry (GIXR) before and after plasma treatment. XPS and AFM were used additionally to get information about the chemical composition and the structure of the surface.

As a result of the H₂-treatment we found an increase of the thickness of the whole film of about 10 nm. The O₂-treatment showed only a small increase of the thickness, while Ar-plasma had no influence of the thickness.

In order to fit the reflectometry measurements best results were obtained under the assumption of a multilayer system of γ -Al₂O₃, Al and γ -Al₂O₃.

The O₂- and Ar-plasma treatment causes a smoothing of the surface of the layers and their interfaces, while in contrast H₂-plasma causes a strong increase of the roughness of the layer. XPS-investigations show that the surface of films only consists of Al₂O₃. The AFM-measurements corroborate the influence of the different plasma on the surface roughness.