11-Surfaces, Interfaces and Thin Films

PS–11.01.08 DIRECT IMAGING OF O-LATTICE OF INTERFACES IN Ti(CN)-TiB_2-Ni CERAMICS. By J.Y. Dai, D.X. Li and H.Q. Ye, Laboratory of Atomic Imaging of Solids, Institute of Metal Research, Academia Sinica, Shenyang 110015, China.

The mixture with the compositions of 57.6 wt% TiB_2, 40.4 wt% Ti(CN) and 2 wt% Ni was hot pressed in vacuum at 1850 °C and 25 MPa. During the sintering process, precipitation of second phase and segregation of impurities have occurred. The average grain size of the ceramics is about 1 μm. Two kinds of orientation relationships (O.R.) between FCC Ti(CN) and hexagonal TiB_2 were detected by means of electron diffraction and high resolution electron microcopy, i.e. O.R. I: $[\bar{1}10]_{Ti(CN)}//[2\bar{1}\bar{1}0]_{TiB2}$, $(111)_{Ti(CN)}//(0001)_{TiB2}$ and O.R. II: $[\bar{1}10]_{Ti(CN)}//[2\bar{1}\bar{1}0]_{TiB2}$, $(001)_{Ti(CN)}//(01\bar{1}0)_{TiB2}$. In general, Ti(CN) and TiB_2 phases oriented at random. When these phases grew together immediately, they coexist with a unique orientation relationship of O.R. I. Ti(CN) particles with a size of a few nm were also precipitated in TiB_2 crystals. Fig.1 shows the typical morphology of TiB_2-Ti(CN)-Ni ceramics with TiB_2 and Ti(CN) grew together and Ti(CN) particals precipitated in TiB_2 crystals. Besides O.R. I, a different orientation relationship called O.R. II between Ti(CN) precipitates and TiB_2 can be detected occasionally.

O-lattice calculations between FCC Ti(CN) and hexagonal TiB_2 interfaces were carried out for those two orientation relationships. For O.R. I, the calculated O-lattice unit cell is hexagonal and the primary dislocation networks is also hexagonal by duality relations. However, no primary dislocation was found in the HREM image. The small mismatch (about 1%) has been accommodated by elastic strain and/or secondary dislocation networks, and the calculation of secondary O-lattice agree well with the observed secondary dislocation networks. Ti(CN) precipitates were hexagonal in shape and the ledges or facets are parallel to edges of precipitation. These can be interpreted by O-lattice theory.

For O.R. II, the calculated O-lattice unit cell is tetragonal body, and the Ti(CN) precipitate also shows a tetragonal body in shape. HREM image of Ti(CN)/TiB_2 interface shows that there are many steps with the height equal to one layer of atomic plane and average interval of seven $(0001)_{TiB2}$ interplanar spacing. O-lattice theory analysis points out that this step structure is the optimal low energy interface which passes through coherent region as many as possible.

In prensent case, intergranular phases were also detected at grain boundaries, which indicated by small black dots with white circular delineation in Fig.1. Two Ni-rich intergranular phases which containing Ti and Si, Fe impurities were found to be $Ni_{31}Si_{12}$ and $Ni_{16}Ti_6Si_7$ type structure.

PS–11.01.09 DETERMINATION OF STRAINS IN $Hg_{1-x}Cd_xTe$ THIN FILM MATERIAL. By Fuju Yu* and An Yang, Shanghai Institute of Technical Physics, Academia Sinica, Shanghai, China

$Hg_{1-x}Cd_xTe$ epitaxial layers grown by MBE, LPE are among the most important semiconductor materials widely used for making infrared focal plane devices.However those films are generally composed of highly lattice-mismatch layers and mutational composition areas due to properties of type 2-6 compounds, and lattice strain and composition mutation can sometimes not been accommodated by buffer layer and further interfere seriously epilayers.The study of lattice mismatch strain (Basson, et al. 1983) , composition mutation, and intensive strain field which created from substrate and acrossed all epilayers was nondestructively carried out by X-ray double crystal diffraction and X-ray topography.

It is useful that FWHM (half width of rocking curve from double crystal diffraction) value as a criterion of structure quality for epitaxial $Hg_{1-x}Cd_xTe$ films. FWHM at 153 arcsec. was measured for MCT1037 wafer including three layers grown by MBE on GaAs substrate, (N,-N) set, $CuK\alpha_1(422)$ reflection. This is a better result but still much large than theoretic one (less than 40 arcsec.), it attributes to lattice mismatch at the bounderies of heterolayers. Double crystal diffraction with high resolution is taken for measuring lattice mismatch in hetrojunction (Cohen, 1967). Measuring a heterojunction $Hg_{1-x}Cd_xTe$/CdTe LE92-5 grown by LPE on CdTe(111) substrate, two diffraction peaks appear on the rocking curve due to different lattice parameters of epilayer and substrate. The lattice mismatch at 0.27% is obtained from the difference of angles corresponding with the two peaks, then composition calculation in epilayer is carried out from Vagar's law, composition x=0.14 much approaches to the expected one. An unhomogeneously growing of $Hg_{1-x}Cd_xTe$ film on MBE was also studied by a gradual transition layer of composition Δx revealed with double crystal diffraction.

X-ray topography is also available for checking the quality of $Hg_{1-x}Cd_xTe$ epilayers (Yu Fuju, 1990). A topographic pattern of a wafer MCT410 shows that the intensive strain field on substrate propagates through the buffer layer, the layer with expected composition, the dull layer and so on, while the thin films are grown by MBE. From this point, it is very important to improve the polishing technology of substrate.

References

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PS–11.01.10 ADSORPTION OF HYDROGEN, OXYGEN AND SULFUR ON Cu(110) STUDIED BY LEED. By R. Zuschke, T. Grünberg, J. Wever, S. Pflanz, M. Burghammer, D. Baraitaru, D. Wolf and W. Moritz, Institut für Kristallographie und Mineralogie, Universität München, Fed. rep. of Germany

The different adsorbate structures and adsorbate induced reconstructions on the Cu(110) surface have been studied by LEED I/V and beam profile analysis. Hydrogen causes a (1x2) reconstruction of the missing row type, the hydrogen coverage is 0.5 monolayers (ML). It is adsorbed either in two-fold or four-fold

coordinated sites between the Cu rows. Three-fold coordinated sites like on H/Ni(110)-(2x1) can be excluded. Oxygen forms two different ordered structures, (2x1) and c(6x2) at coverages of 0.5 amd 0.66 (ML). These structures are surface compounds consisting of Cu-O chains adsorbed on the slightly relaxed but otherwise unreconstructed surface. In the case of the c(6x2) structure the Cu-O chains are kept together by additional top Cu atoms. The results of the LEED structure analysis compare well with the X-ray results [1]. The beam profile analysis shows that the mechanism of the superstructure formation is different from that after H adsorption though both structures involve mass transport of half a substrate layer. The formation of the (1x2) missing row structure involves the movement of steps and probably starts at steps. The formation of the O-Cu chains, on the other side, starts at terrace sites, as has been shown previously by STM measurements [2] and causes at higher temperatures the creation of defects on flat terraces.
Sulfur, which is slightly weaker bound to Cu than oxygen forms 5 different ordered structures. With increasing coverage c(2x10), p(2x5), p(5x2), c(8x2) and p(3x2) structures occur. LEED I/V analyses have been performed for 3 structures, p(2x5), p(5x2) and c(8x2). The results are compared with recent STM measurements [2].

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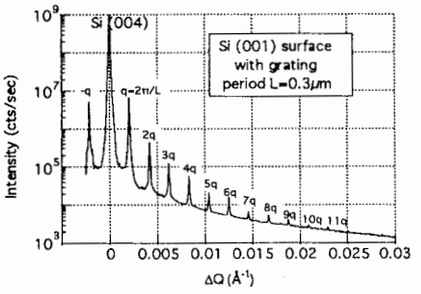
PS-11.01.11 X-RAY SCATTERING FROM COHERENT PERIODIC GRATING STRUCTURES ON SEMICONDUCTOR SURFACES. By Qun Shen*, CHESS, Cornell University, and C.C. Umbach and J.M. Blakely, Dept. of Materials Science & Engineering, Cornell University, Ithaca, New York 14853, U.S.A.



We have performed a high resolution x-ray diffraction experiment on a Si (001) sample with surface gratings in both the [110] and the [1,-1,0] directions, using synchrotron radiation at CHESS. More than ten orders of grating interference reflections arising from a 0.3 μm grating period have been observed (see Figure above). The widths of these grating peaks are as narrow as that of a crystal reflection, despite of only ~20 gratings probed within the coherent width of the x-rays. This demonstrates that every atom at lattice site within a single crystal domain scatters coherently and contributes to the superlattice reflections. The intensity distribution among these grating superlattice reflections provide valuable information on the shape and the structure of the surface gratings as well as on possible crystal lattice distortions. With a transverse coherent length of 5-10 μm and a wavelength of ~1Å, x-rays are perfectly suited both for analyzing submicron-sized grating structures and for determining crystallographic qualities of the grating material.

PS-11.01.12 PREPARATION OF THIN TRANSITION METAL ALLOY FILMS BY SUCCESSIVE VACUUM DEPOSITION AND THEIR STRUCTURE INVESTIGATION BY HREM. By K. Yoshida, R. Yamashita, T. Kawai and K. Shimonishi, Faculty of Engineering, Kobe University, Rokkodai, Nada, Kobe 657 Japan.

Intermetallic compounds with lattice dimensions larger than 10 Å are frequently found in many binary transition metal alloys. Some of them are industrially important as in powdering phenomenon during plastic deformation and pin-hole formation in protective surface coatings. The larger are the lattice constants, the more are they suited to the high resolution electron microscopy because of its limited resolving power. Thin, enough for the observations, alloy films can be prepared by a low temperature heating of double layer thin films which are prepared by successive vacuum deposition. Results at the present for the alloy films of Mn-Bi and of Fe-Zn system will be shown.

Bi was deposited about 300 Å thick on Carbon supporting films of 100 Å thickness. Mn was then deposited onto top surface of the Bi layer 200 Å in thickness. Degree of the vacuum during these depositions was on the order of 10^{-6} Torr. The double layer films were then heated at 265°C, just below the melting point of Bi, 271°C, in the same vacuum for 50 to 200 hr. Thickness of thus prepared alloy films is less than 500 Å and they can directly be observed under an electron microscope. More than four different kinds of new crystals were found in the specimens (K. Yoshida et al, Suppl. to Trans. JIM, 1988, 29, 135-138), whereas only one intermetallic compound, MnBi phase, is described to exist in the published phase diagram. The most remarkable one of the new alloys is the so-called long-period tetragonal phase, whose lattice constants are as long as a=17.26 Å, c=10.21 Å. Symmetry and the lattice constants were determined only from its single crystalline net electron diffraction patterns (K. Yoshida et al Acta Cryst., 1989, B45, 40-45). At the first sight, its high resolution images are very similar to those of Sigma-phase of Ni-Cr alloy system. However, there is a clear discrepancy between this new Mn-Bi alloy phase and the Sigma-phase structure in the extinction of the diffraction spots. Structure of this new phase must then be a deviated one from the regular Sigma-phase structure. Correct positions of almost 60 atoms in the large unit cell are now being searched for referring both to its high resolution images and to its diffraction spot intensities. Some quasicrystalline regions of twelve-fold symmetry, preferably interpreted as an aggregate of atomic clusters,16 Å in diameter, were occationally found in the specimens (K. Yoshida et al, Phil. Mag. Lett.1991, 63, 127-132). This region will be a preliminary stage to the full establishement of the long-period tetragonal lattice.

In the case of Fe-Zn alloy films, Zn is very easy to evaporate. Therefore, Zn was deposited much thicker, 800 Å, on top of the Fe layer whose thickness was about 200 Å. The double layer films were then heated at 150°C in the vacuum for 150 hr. The melting point of Zn is 419.5°C. Small Fe grains seemed to extend somewhat by this heating and selected area diffraction patterns from different grains showed that Capital Gamma-phase (Γ-phase) was formed in the specimen. This alloy has a cubic lattice with a=8.98 Å and atomic positions in the lattice were postulated by a few precedent investigators. Their results are now being re-examined by comparing their computer simulated images with the obseved high resolution ones as well as the diffraction spot intensities. Many high resolution images showing various types of lattice defects were also taken, which may have some correlation to the powdering phenomenon of industrical galvanized steels.

Finally, it can be said that the preparation procedure described above will be best suited to structure investigations using high resolution electron micrographs together with the electron diffraction patterns.