ION BEAM AND ITS APPLICATIONS

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ABSTRACT

Development of metal ion source, growth of high quality Cu metal film, formation of non-stoichiometric SnO_2 films on Si(100), and modification of polymer surface by low enregy ion beam have been carried out at KIST Ion Beam Lab. A new metal ion source with high ion beam flux has been developed, and effects of ion in Cu film formation have been discussed. A gas selective SnO_2 thin films sensor have been developed by a hybrid ion beam (HIB) deposition, and non-stoichiometric SnO_2 films are controlled by supplying energy. The ion assisted reaction (IAR), in which keV ion beam is irradiated in reactive gas environment, has been developed for modifying the polymers and enhancing adhesion to other materials, and advantages of the IAR have been reviewed.

1. INTRODUCTION

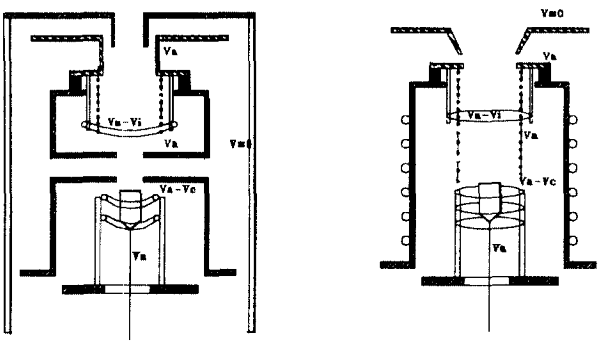
The surface modification or the thin film formation by adjusting depositing atom's or molecule's energy themselves has been considered to be one of the solution to solve many difficulties in material engineering. These methods, such as ion assisted deposition, partially ionized beam, etc., can also solve the cleaning of adsorbed impurities by sputtering. The irradiation of energetic particles onto the substrate has assisted to form the large size nuclei which be important factors in the film formation, and have changed properties of thin films such as crystallinity, non-stoichiometry, surface morphology, etc., and have modified surface properties of metal, ceramics and polymers by controlling particles energy.

In the article, we reviewed high quality Cu thin film growth on Si(100) surface by ionized cluster beam with various acceleration voltages and ionization potentials, a non-stoichiomentric SnO_{2-x} thin film growth by hybrid ion beam, and a surface modification of polymer by keV ion irradiation. We also have tried to suggest advantages and perspectives of the films and modification.

2. EXPERIMENT

A metal ion source(MIS) which has high ionization effect and uniformity was developed. Figure 1(a) shows the pristine type MIS called ionized cluster beam source and Fig. 1(b) shows the newly developed MIS. In order to confirm characteristics of metal ion source, ion beam current density of two types of MIS was measured by a Faraday cup which is located at the distance of 250-mm from the top of the MIS unit and Cu was used as a source material.

The hybrid ion beam system which have base pressure of 10^{-6} torr consists of metal ion source and one gas ion source. Sub-stoichimetric SnO_x thin films were grown on Si(100) substrate using a reactive ion-assisted deposition (RIAD). Neutral Sn metal was evaporated using a MIS and concurrently oxygen ions were irradiated by a cold hollow cathode ion gun. Average oxygen ion energy Ea (eV/atom) were controlled to obtain the films having different oxygen contents by a variation of discharge current of oxygen gas in the discharge chamber at fixed ion-beam-potential 500 V. Determination of the number fraction x and the chemical state of sub-stoichiometric SnO_x films was done by Auger electron spectroscopy (AES).



(a) (b)

Fig. 1. Schematic diagrams of ion cluster beam source. (a) previous type MIS and (b) new MIS developed at KIST

Surface modification of polytetrafluoroethylene (PTFE) was performed by an 1-keV cold hollowed type ion gun. Characteristics of modified PTFE surface was investigated by x-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM). The PTFE samples were sandwiched between the sample holders with the adhesive glue on both surfaces. The sample was cured at 60 °C in an oven for one day and then tested with an Insertion Test machine. More detail expression of experimental methods were described in elsewhere[1].

3. RESULTS AND DISCUSSION

Beam uniformity and beam current density of MIS were investigated by moving a Faraday cup from the center to the edge. As shown in Fig 2, ion beam current density depended on electron beam current for ionization in both type of MIS. However, ionization efficiency of newly developed MIS is higher than old one and ion beam uniformity of new MIS is also superior to old one. Beam uniformity depends on the geometrical factor of MIS. In previous type MIS(Fig. 1(b)), ion beam current density captured at Faraday cup was slowly changed within 30-mm region but it was rapidly decreased out of this region. Newly developed MIS shows enhanced uniformity and high ionization efficiency comparing to previous type MIS. Ionization efficiency of newly developed MIS is three or four times larger than old one. And relatively large amount of ion beam current was detected within 70 mm region from the center. This beam uniformity could be controlled by changing the length of a grid. More longer the grid, more uniform the beam current density. Ionization efficiency are also affected by the length of grid. As the length of grid increased, the ionization region also increased, so enhanced ionization efficiency could be obtained[2].

Figure 3 shows SEM images of step coverage where the hole size is 0.5 μm (ϕ) × 2 μm and the samples was deposited at acceleration voltages of (a) 1 and (b) 4 kV, respectively. Some grains (~ 0.1 μm) were deposited on the wall of holes in the sample deposited at an acceleration voltage of 1 kV. These grains disappeared in the sample deposited at an acceleration voltage of 4 kV. In the case of filling hole, incident ion disturbs the grain growth on the wall. No grain was found on the wall of the sample deposited at an acceleration voltage of 4 kV (Fig. 3(b)), which means that energetic incident ion moved the adsorbed atoms on the wall to the bottom of hole by collision. This experimental result explicitly shows the role of energetic ion beam. So, use of metal ion beam in thin film formation could give a special properties to the grown film.

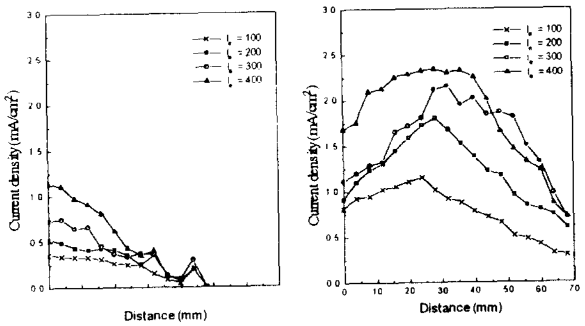


Fig. 2. Changes of ion beam current density as a function of distance from the center. (a) conventional MIS and (b) new MIS. Applied acceleration voltage was 2-kV and deposition rate was 2 Å/sec.

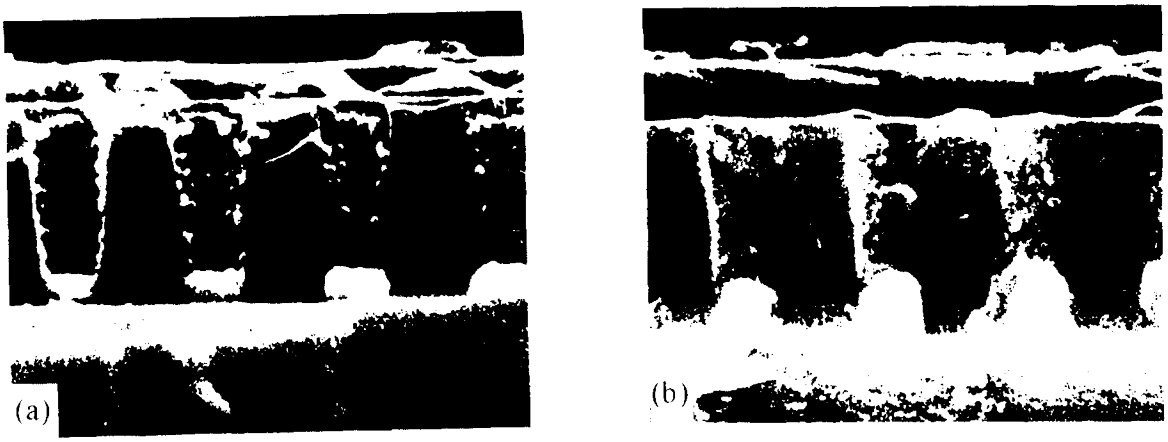


Fig. 3. SEM images of step coverage where the hole size is 0.5 μm (ϕ) × 2 μm and the samples are deposited at acceleration voltages of (a) 1 and (b) 4 kV.

The characteristics of grown SnO_x were investigated. Figure 4 shows the AES spectra of pure tin metal(a), as-deposited films (b)~(e), and stoichiometric SnO_2 powder (f). In the as-deposited films, the deposition parameter was average energy per atom and each film was deposited with (b) 25, (b) 50, (c) 75, and (d) 100 eV/atom. The major peaks detected in the AES spectra for the as-deposited films are identified as Sn *MNN* (316.8, 366.8, 429.8, and 437.4 eV) and O *KLL* (493.2 and 513.2 eV). The atomic ratio of oxygen to Sn metal uses differential peak-to-peak heights of the O $KL_{2,3}L_{2,3}$ transition and Sn $M_4N_{4,5}N_{4,5}$ transition lines. The major peaks related to all Sn *MNN* transition shifted to lower kinetic energy as oxygen contents are increased in the deposited films. These of electrons associated with cationic tin. The amount of chemical shift and N_O/N_{Sn} are listed in table 1. As shown in this result, stoichoimetry of ceramic thin film could be controlled by using ion beam[3].

Characteristics of modified PTFE surface was investigated by XPS and SEM. A SEM image of the virgin sample shows a smooth surface(Fig. 6(a)). The Ar^+ irradiated surface with the ion dose of

10^{17} ions/cm^2 in oxygen gas environment showed numerous peaks and canyon (Fig. 6(b)) which were probably formed by pitting action of the high speed ions onto the PTFE surface. The size of the peaks is about 0.1 μm in diameter and 0.5 μm in depth.

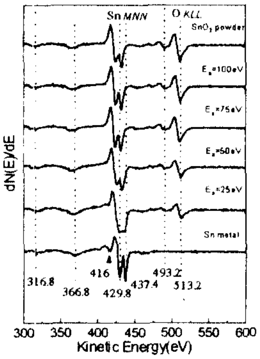


Fig. 4. AES spectra of (a) pure tin metal (b)~(e) as-deposited tin oxide films, and (f) stoichiometric SnO_2 powder.

Table 1. Observed Auger Sn transition energy(E_{obs}) and N_o/N_{sn}.

Species	E_{obs}(eV)	N_o/N_{Sn}
Sn metal	437.4	0
Ea=25eV/atom	433.6	1.14
Ea=59eV/atom	433.2	1.32
Ea=75eV/atom	432.8	1.80
Ea=100eV/atom	432.6	1.91
SnO_2 power	432.6	2.00

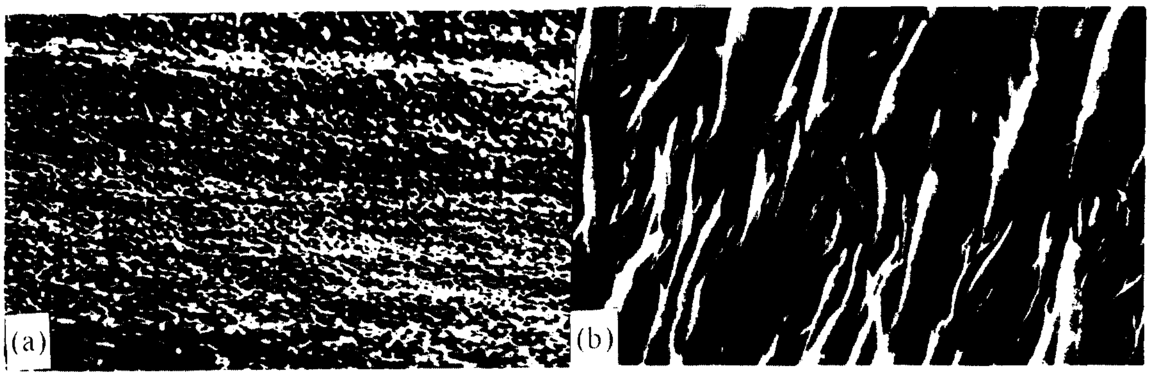


Fig. 5. SEM images of PTFE surface. (a) non-irradiated and (b) irradiated with Ar^+ ion in oxygen environment.

Figure 6 shows changes of C1s core level spectra of PTFE. As shown in Fig. 6, the XPS spectrum of a virgin surface of PTFE exhibits the strong C1s peak at 292 eV which corresponds to C-F_2 bond. When the ion dose is increased, the intensity of C-F_2 peak is reduced and broadened and a small ridge was formed at the energy position between 285 and 292 eV in C1s spectra. Peaks of C1s bonded to oxygen rise between 285 eV and 288 eV, and peaks bonded to fluorine rise between 286 eV to 294 eV. The increment of intensity level of the ridge between 285 and 292 eV means that the irradiation severed the C-C and C-F_2 bonds randomly on the $(-CF_2-)_n$ chains and produced COH compounds.

The adhesion strength of crystal bond to the PTFE surface modified in IAR was determined by tensile tests as shown in Fig. 7. A low adhesion strength of 200 kg/cm^2 was obtained when the unmodified PTFE samples were used. The maximum value obtained was 750 kg/cm^2 at the ion dose of 10^{16} ions/cm^2 which is 3.7 times the values obtained with unmodified PTFE. When the failure surfaces modified at the 10^{16} ions/cm^2 with oxygen environment were investigated, however, it was found that the failure was not occurred at the interface but at the crystal bond. It seems that the adhesion strength between the crystal cement and the PTFE modified at the ion beam condition is stronger than the mechanical tensile strength of the crystal cement. This improvement might be due to a mechanical interlock by the rough surfaces and a chemical interaction between hydrophilic groups and crystal bonds by the hydrogen bonds or Van der Waals bonds.

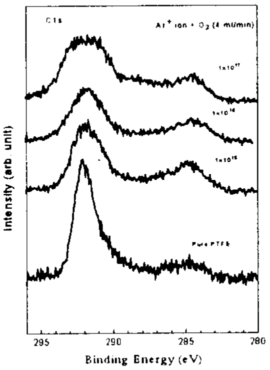


Fig. 6. C1s XPS spectra of PTFE after IAR treatment

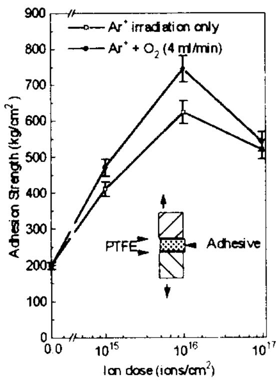


Fig. 7. Changes of adhesion strength of PTFE as a function of ion dose.

4. CONCLUSIONS

The Cu thin film and the SnO_{2-x} thin film were grown by ion beam techniques, and keV ion irradiation for PTFE surface modification have been discussed. In the case of filling hole by MIS, small grains were observed on the wall of hole with acceleration of 1-kV but no grain was observed on the wall with acceleration voltage of 4-kV. In the case of tin oxides, stoichiometry could be changed by controlling the average energy of incident oxygen gas. In polymer modification, we confirm that hydrophilic groups are formed on the surface of polymer and surface morphology could be changed by ion irradiation. These surface modification remarkably improve the adhesion strength.

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