Hydrogen charging effects in Pd/Ti/TiO$_2$/Ti thin films deposited on Si(111) studied by ion beam analysis methods


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Motivation
Titanium hydride films have many potential applications, e.g. neutron super mirrors, hydrogen storage layers, standards for hydrogen quantitative analysis, etc. Neutron super mirrors are multilayers of nickel and titanium employed for the transport of cold neutron beams. Saturation of these systems with hydrogen improves their reflectivity, because hydrogen increases the difference between the two indices of refraction and allows one to diminish the number of layers needed. In addition, titanium deuteride films can be employed as a neutron source in ion beam technology. Titanium and its alloys have many industrial applications thanks to their excellent corrosion resistance and high specific strength. However, hydrogen absorption induces cracking of titanium layers. Titanium dioxide top layers acts as a protective layer (against corrosion), but also as a barrier to hydrogen absorption into the metal.

Samples
• Ti/TiO$_2$/Ti multilayers deposited on Si(111) substrates by magnetron sputtering
• Some of samples partially covered with palladium by MBE

Experiments and investigation techniques:
• Surface examined by HRSEM and AES
• Structure investigated by RBS
• Hydrogen charging and hydrogen profiles obtained by SIMS and NRA (N-15 method)

Structure
Prior to other experiments samples were studied by RBS. For samples with top titanium layer partially covered by palladium, no interdiffusion was observed and the thicknesses of the layers were in good agreement with the estimated values.

Surface
Before hydrogen charging – surface smooth and homogenous

Titanium layer after hydrogen charging – granulation of the surface

Palladium layer – irradiation with heavy ions (Cs) during SIMS measurements – granulation of the surface, annealing during hydrogen charging caused some cracks on the surface.

Hydrogen profiles
Pd(20nm)/Ti(50nm)/TiO$_2$(100nm)/Ti(100nm)/Si(111)
SIMS profiles before (left) and after (right) hydrogen charging

Palladium acts as a catalyst for hydrogen diffusion through TiO$_2$ layer – after charging hydrogen gathers in titanium layers, in titanium oxide its concentrarion is the same as before.

Ti(50nm)/TiO$_2$(100nm)/Ti(100nm)/Si(111)
SIMS profiles before (left) and after (right) hydrogen charging

After charging, hydrogen gathers only in titanium layers, in titanium oxide its concentrarion is lower.

Conclusions:
• Hydrogen charging causes a granulation of titanium layer. Palladium layer changed because of annealing (cracks) and irradiation during SIMS measurements (island formation).
• Hydrogen profile obtained by SIMS and N-15 measurements proved a higher hydrogen concentration in samples with partially-covered top layers, than in samples without palladium indicating that palladium acts as a catalyst for hydrogen diffusion through TiO$_2$ layer.

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