Characterization of Lubricative Coating after Exposure Test in Orbit

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Change in such properties were characterized as tribological properties, surface chemical condition and so on for lubricative coating films of titanium nitride (TiN) that have been effected by exposure to an environment in orbit with the Russian Service Module / Space Environment Exposure Device (SM/SEED) prepared by JAXA on the International Space Station (ISS). Friction measurement with a mating pin of stainless steel showed that TiN coated stainless steel sheets after exposure test to orbit environment for a year generally decrease friction coefficient and can keep almost constant even after bakeout under a vacuum as well as at an atmospheric pressure due to some kind of modification of lubricant layer by the exposure.

1. Introduction

Orbit environment of a space shuttle and a space station around the earth is known as an ultrahigh vacuum under 10^{-5} Pa and usually suffers from atomic oxygen attack at a high speed as well as ultraviolet irradiation. Moving components materials therefore require surface modification with stable lubrication for preventing increase in friction force due to oxidation, irradiation damages and so on. We have prepared solid lubricative coating films of titanium nitride (TiN) on stainless steel sheets. TiN has advantages of combining the characteristic properties of covalent compounds, such as a high hardness and chemical and thermo-dynamical stability with metal-like electrical and thermal properties, which makes it a unique and excellent candidate in different technological areas [1]. Due to a low friction coefficient ($\mu\sim0.17$), TiN coating films are successfully utilized as surface protective coatings for tribological applications as well as sufficient diffusion barriers for metal interconnects in semiconductor devices [2-4]. TiN coated stainless steel sheets have been exposed on a space station in orbit for a year and then recovered to the Earth. We have analyzed tribological properties, surface chemical condition and so on in order to understand the change in properties of lubricant coating by exposure in orbit and also to develop advanced smooth and reliable space solid lubricant coating.

2. Experimental

Type 304 austenitic commercial stainless steel sheets (size; 14 mm × 14 mm × 1mm) have been coated with TiN using a radio frequency magnetron sputter deposition system with a film thickness about 500nm [5]. TiN coated stainless steel sheets were set onto a panel as shown in Fig.1 and installed into the Russian Service Module / Space Environment Exposure Device (SM/SEED) prepared by JAXA on the Russian service module in the International Space Station (ISS), and have been exposed in orbit then recovered to the Earth. Friction properties of TiN coated stainless steel sheets with and without exposure to orbit were observed with a system developed for measuring vacuum friction. Design of the friction measurement system comes from the Bowden-Leben type as shown in Fig.2 [6]. A sheet stage can move in x-y direction by two axes stepping motor and a manual axis in an ultrahigh vacuum. A sheet holder of an inch in diameter is easy to be fixed with plate spring. A ball pin prove above the sheet holder has a load weight holder with 0.49 N and has two strain gauges to detect frictional force. A mating pin made of stainless steel was scanned under normal load. The vacuum chamber including the friction measurement system was evacuated with a tandem turbo-molecular pumping system and an iron pump down to 10^{-5} Pa or less level. Surface chemical characterization for the sheets was carried out with a X-ray photoelectron spectros-

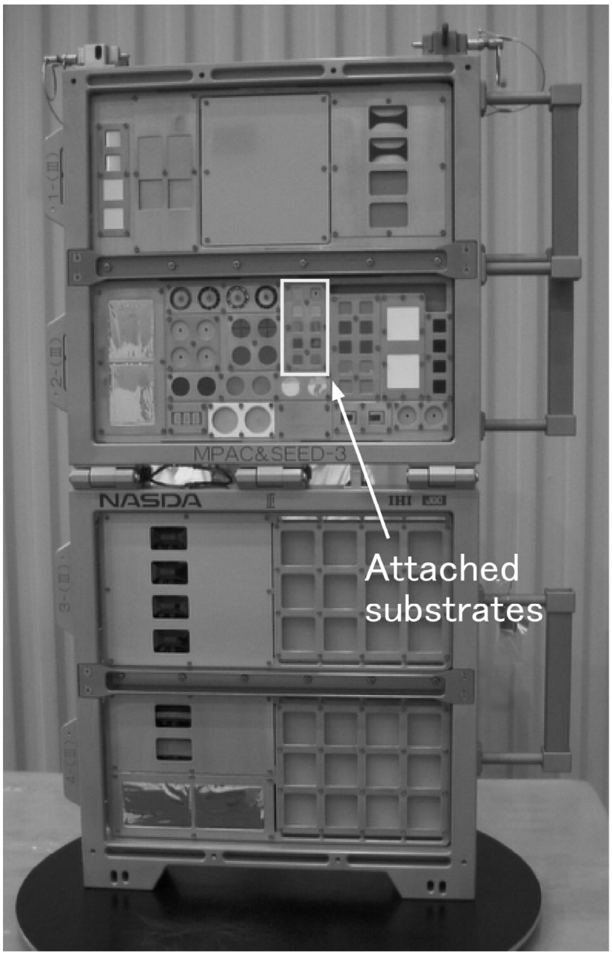

Fig.1 Photo of a panel with attached TiN coated stainless steel sheets for SM/SEED module.

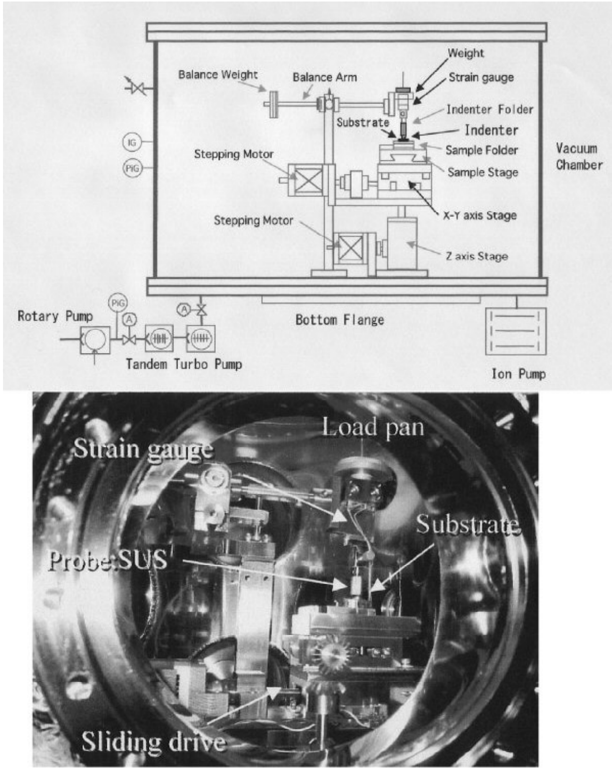

Fig.2 Schematic view of a vacuum friction measurement system and photo of a center system.

copy using a PHI´s Quantera SXM with X-ray of excited from Al Kα (1486.6 eV), constant pass energy of 69 eV, X-ray beam size of 100 μm in diameter and so on.

3. Results and Discussion

3. 1. Friction Properties

Friction properties of the TiN coated stainless steel sheets sliding against stainless steel as exposure test sheets were determined both at an atmospheric pressure and in an ultra-high vacuum. TiN coated stainless steel sheet shows a friction coefficient (μ) of 0.12 at an atmospheric pressure and shows an increased value of 0.26 in 10^{-5} Pa. TiN coated stainless steel sheet after exposure test in orbit for a year shows a low friction coefficient of 0.10 at an atmospheric pressure and shows a small increased value of 0.11 in 10^{-5} Pa. It might be considered that TiN coated stainless steel sheets with exposure to orbit environment generally decrease friction coefficient under a vacuum of 10^{-5} Pa as well as at an atmospheric pressure due to atomic oxygen attack and ultraviolet irradiation. Fig.3 shows change in a friction force of TiN coated stainless steel sheets with and without exposure as changing normal load. The exposed TiN coated stainless steel sheet shows a little bit of higher friction than no exposed TiN stainless steel sheet under low normal load. This might be due to an increase in adsorption force by change in surface roughness on a nano-scale.

We have also measured the friction coefficient of TiN coated stainless steel sheets after exposure in orbit in order to estimate the thermal stability by vacuum annealing. Fig.4 shows a change in a friction coefficient (μ) of TiN coated stainless steel sheet with exposure at an ambient temperature about 300 K to have been kept and measured in situ after annealing at 500 K in an ultrahigh vacuum about 10^{-5} Pa. Little change in friction coefficients of TiN coated stainless steel sheet exposed in orbit after vacuum annealing is shown and such thermal stability might come from formation of tough coating layer independent of adhesion layer with good effect on friction.

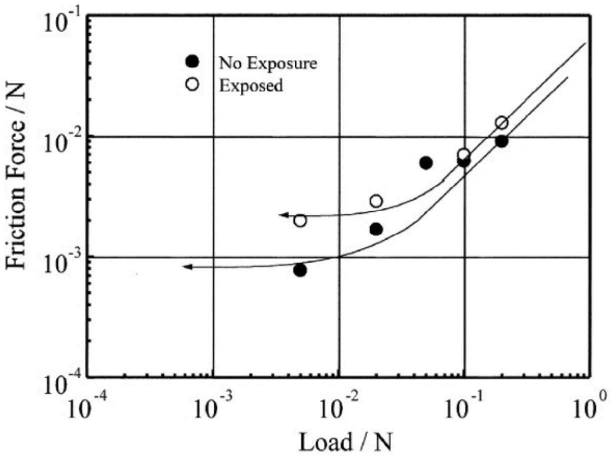

Fig.3 Change in a friction force of TiN coated stainless steel sheets with and without exposure as changing normal load.

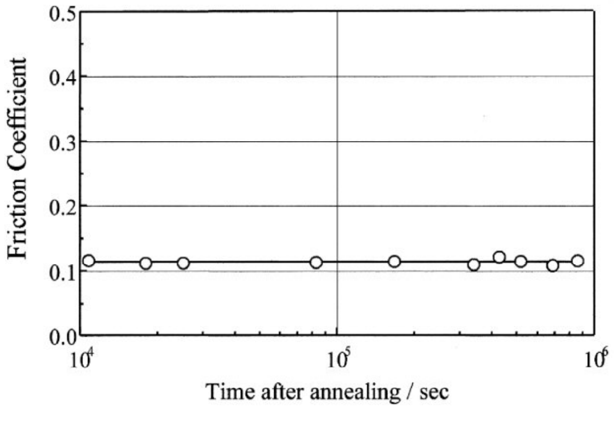

Fig.4 Change in a friction coefficient (μ) of TiN coated stainless steel sheet with exposure after annealing at 500K in a vacuum.

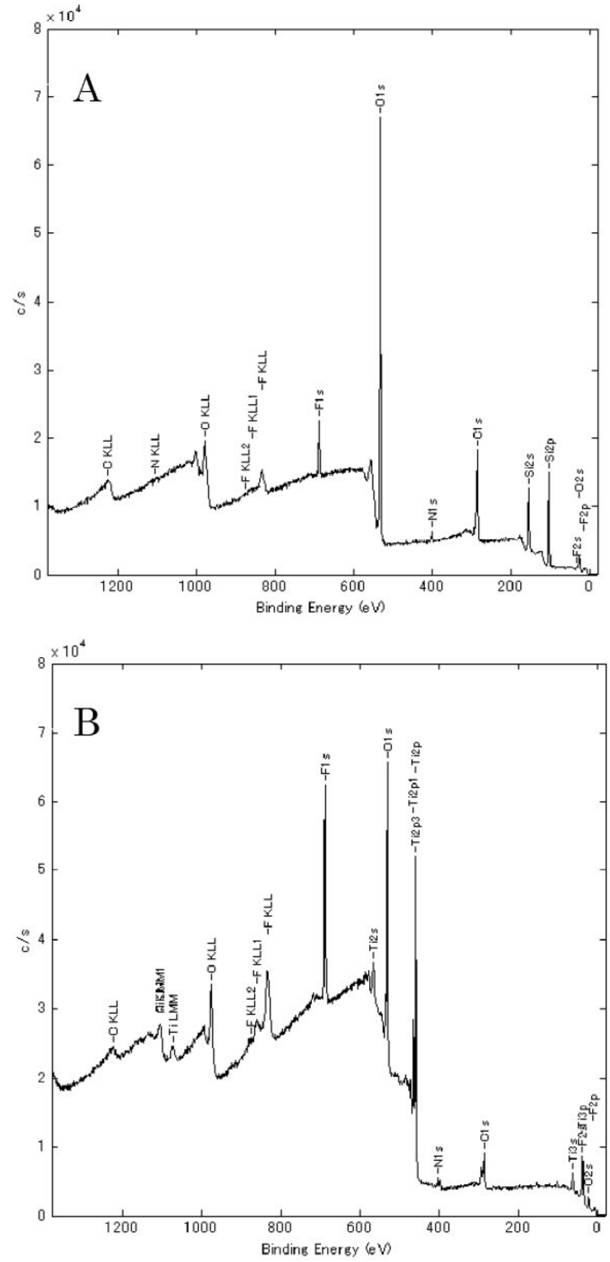

Fig.5 Survey spectra of exposed (A) and screened area (B) of TiN coated stainless steel sheets.

3. 2. Surface Analyses

Surface chemical condition and depth profile were obtained with a X-ray photoelectron spectroscopy for two areas of TiN coated stainless steel sheets, one for exposure and the other for screening against exposure in orbit. Fig.5 shows survey spectra of exposed and screened stainless steel sheets coated with TiN. Silicon (Si) peaks and large oxygen (O) peaks are observed on the exposed area while large fluorine (F) peaks are observed on screened area of the TiN coated stainless steel sheet. Fig.6 shows depth profiles of exposed and screened area of the stainless steel sheet coated with TiN. Silicon and oxygen concentrated layer is observed at the surface of TiN coating layer at the exposed area in orbit.

Changes in chemical condition were obtained for elements composing TiN coated stainless steel sheets after exposure in orbit as shown in Fig.7 with main binding energies [7]. Titanium forms TiN with nitrogen mainly and also forms TiO_2 with oxygen in part. Silicon forms SiO_2 with oxygen at the surface of TiN coating layer.

Comparison between these surface analytical results of exposed and screened area of TiN coated stainless steel sheet indicates as follows. TiOx layer formed during TiN film preparation. Silicon evaporated from silicone adhesives used for space station structure might react the atomic oxygen to form SiO_2 during exposure in orbit. The mixed structure of SiO_2 in coating film is considered to form a good lubricant layer that can offer smooth and stable sliding against annealing in a vacuum.

4. Conclusions

TiN coated stainless steel sheets were prepared with a radio frequency sputtering deposition and then installed into SM/SEED in orbit for a year. Results obtained with the friction measurement system using a mating pin of stainless steel and X-ray photoelectron spectroscopy showed that TiN coated stainless steel sheets with exposure to orbit environment for a year decreased friction coefficient and could keep almost constant even after annealing in a vacuum as well as at an atmospheric pressure due to some kind of oxidation of lubricant layer especially consisting of silicon oxide during the exposure. TiN coating will be therefore one of good candidates for space lubricative modification of moving materials and oxidization of TiN with mixed silicon oxide might lead to new lubricant material for tough environments.

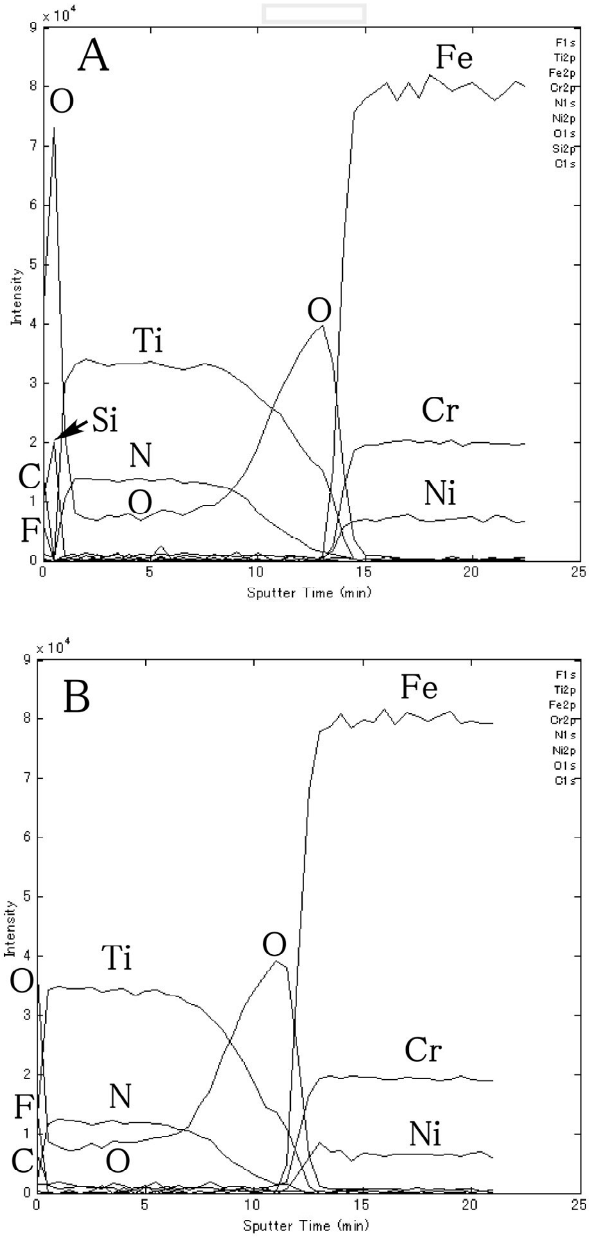

Fig.6 Depth profiles of exposed (A) and screened area (B) of TiN coated stainless steel sheets.

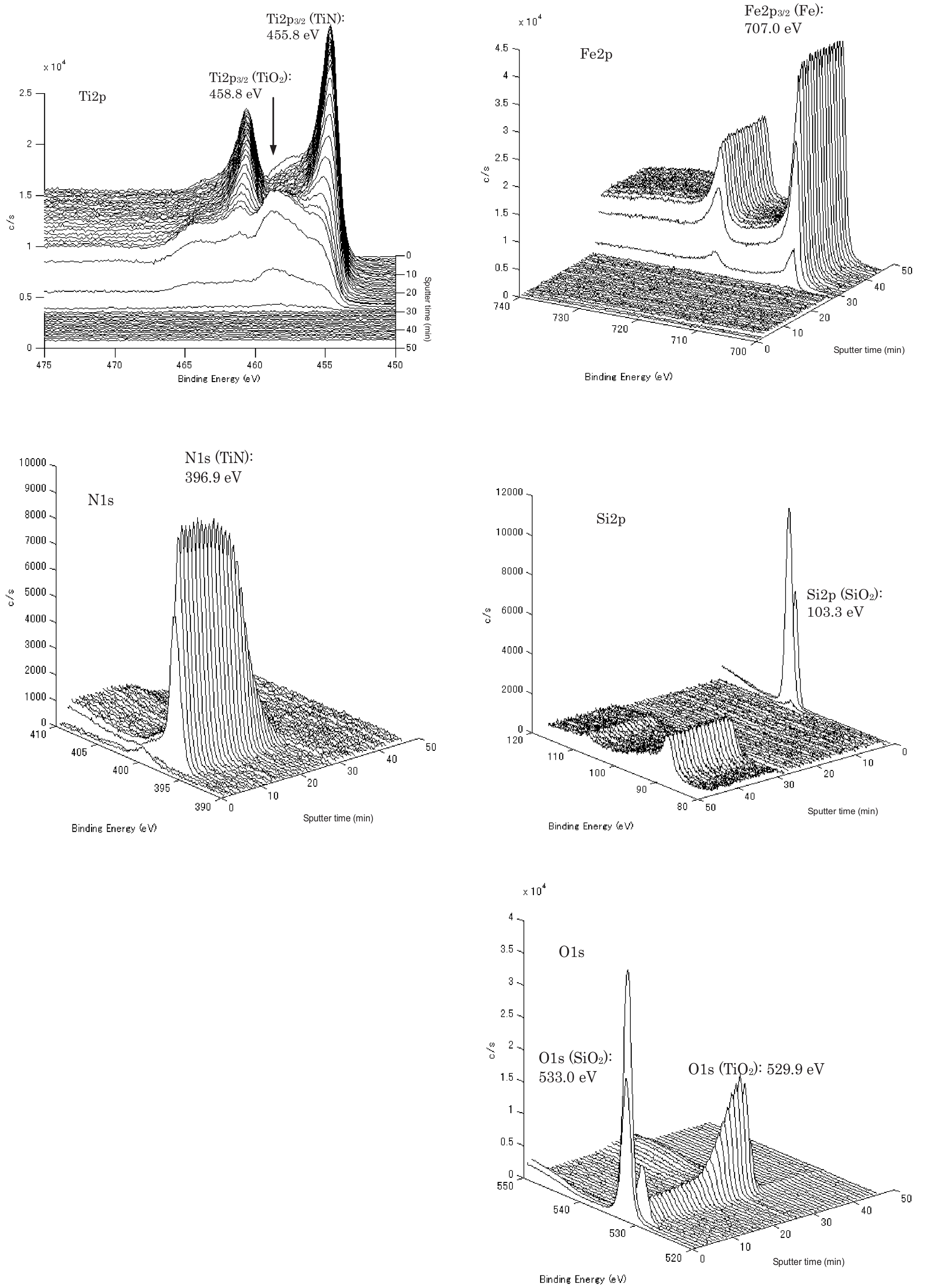

Fig.7 Changes in XPS spectra for main elements with sputter time for obtaining depth profile.

References

[1] J. E. Sundgren, *Thin Solid Films* **128**, 21 (1985) .

[2] J. S. Chun, I. Petrov, and J. E. Greene, *J. Appl. Phys*. **86**, 3633(1999).

[3] M. Stoiber, E. Badisch, C. Lugmair, C. Mitterer, *Surf. Coat. Technol*. **163**, 451 (2003).

[4] S. Ikeda, J. Palleau, J. Torres, B. Chenevier, N. Bourhila, and R. Madar, *J. Appl. Phys*. **86**, 2300 (1999).

[5] M. Goto, A. Kasahara, M. Tosa, J. Hobley, M. Kishimoto, K. Yoshihara and H. Fukumura, *J. Vac. Sci. and Technol*. A**20**, 1458 (2002).

[6] A. Kasahara, M. Goto, M. Tosa and K. Yoshihara, *J. Electroanalytical Chemistry* **45**, 559(2003).

[7] J. Chastain and R. C. King, Jr., Handbook of X-ray Photoelectron Spectroscopy, Physical Electronics Inc., Minnesota (1995).