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Ion Sputtering Characteristics of Oxygen Species†

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When the ion beam (primary ion) bombards the surface of solid material, the atoms in the specimen accept some energy of the primary ion and are ejected from the lattice sites. These ejected atoms repeat impacts each other. If the atoms get larger energy than the lattice bonding force from the primary ions and the ejected atoms, they are sputtered from the the solid surface. Therefore, some of the sputtered atoms are ionized (secondary ion) and then these phenomena can be analyzed through a mass spectrometer.

The ion probe micro analyzer (IMA)[1] has two faculties—to sputter secondary ions from a surface of the solid material and to analyze them through a mass spectrometer. IMA has characteristics of high efficiency detecting for the lighter elements and an ability of analysis of sputtered ions from extremely thin solid surface ranging several angstrom. It is, therefore, expected to be suitable to analyze chemical composition of oxide films formed on metal surface. Generally, the oxide films of a metal consist from multiple layers. Each layer has a peculiar structure and the bonding force between the atoms is different. As aforesaid, the atoms of solid surface are sputtered when the atoms in the specimen accept the larger energy from the primary ion than the bonding force. It is, therefore, expected that the secondary ion yield of each element depends on the energy of the primary ion. It will be probably important to investigate the dependence of each secondary ion yield on the structure of solid materials and the primary ion energy. This note reports the relative secondary ion yield of two kinds of iron oxides using a IMA.

Analytical grade powder reagents —Fe_2O_3 and Fe_3O_4 (Wako Chem. A. R.)— were selected as samples for the measurements of secondary ion yield. These powder samples were burried in indium disks. The measurements were performed with a Hitachi IMA-SS type ion probe micro analyzer used for the elucidation of various welding defects[2]. The operating conditions of the IMA were as follows.

The measurements were made under the experimental conditions which the primary ion energy was 5, 7.5, 10 and 12.5 KeV. The drawing voltage of 1.5 KV for the secondary ions to the mass spectrometer was selected in order to get little variance of the primary ion uniformity due to the primary ion energy.

The beam diameter was hold to be constant although there occured a little change of the voltage of the objective lens.

In order to eliminate any surface contamination effect, at first time, the sample was bombarded for 10 ~15 minutes by primary argon ion beam of about 800 μm diameter. Measurement was carried out with small beam diameter of about 500 μm.

Before the measurement, the sample was sputtered for about one minute with large beam diameter of 800 μm to avoid the influence of crator shape on the result.

Each relative secondary ion yield of Fe_2O_3 and Fe_3O_4 versus to the primary argon ion energy are compared in **Figs. 1 (a)** and **(b),** respectively. The secondary ion species observed are O^+, FeO^+, Fe_2^+,

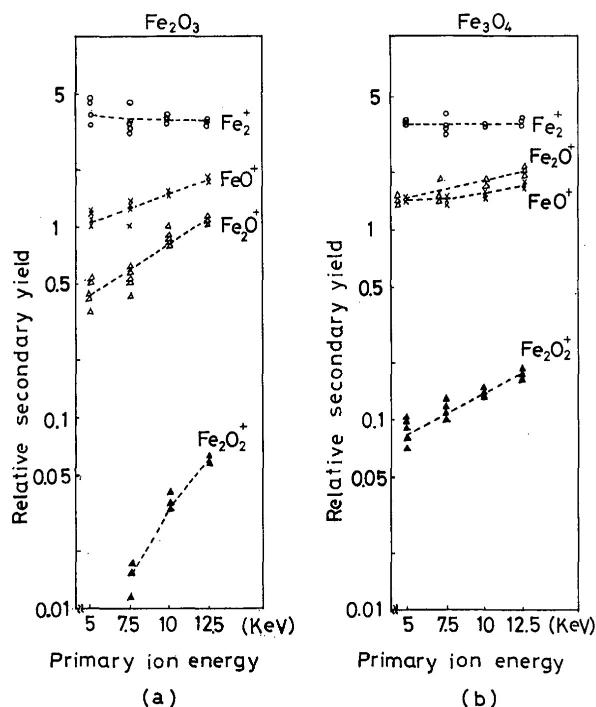

Fig. 1. Relative secondary ion yield of Fe_2O_3 and Fe_3O_4.

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Fe_2O^+ and $Fe_2O_2^+$. The vertical coordinate shows the ratio of intensities between each secondary ion and O^+ ion.

From these results, it will be anticipated that the ion yields of Fe_2^+ are almost independent upon the primary ion energy. The other ions yields increase monotonically with the energy of primary ion. The gradients in Fe_2O_3 were larger than those in Fe_3O_4. The tendency was remarkable for $Fe_2O_2^+$. The same measurements were applied to pure iron. However any dependences on the primary ion energy were hardly observed.

It is, therefore, considered that these phenomena would be probably due to the variance of bonding force between the atoms in the specimens. If it is so, it may be possible to obtain information concerning bond state of the oxide materials. However it is very difficult to investigate the details of the relations between the secondary ion yield and the bonding force because the ionization efficiencies of the every atoms and the compounds are not known for the present time and these results depend exceedingly on the degree of vacuum in the sample chamber of IMA.

Further studies using IMA on the secondary ion yield of the every elements and the compounds are in progress, especially from the viewpoint of the state analysis of the oxide films, which is important for the corrosion resistance.

References

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2) N. Iwamoto, Y. Tsunawaki and M. Fuji: Transactions JWRI, 2 (1973) 209.