Effect of Decontamination on MgO by Heating in the Air

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The Organic Materials Section of the Surface Analysis Society of Japan is planning to collect standard spectra for the construction of the network database. A lot of discussion is taking place regarding this method.

Water and hydrocarbons accumulate thickly on the specimen surface exposed to the air. Generally ion sputtering and heating in a vacuum is recognised as a means of removing this contamination. In fact ion sputtering can be used for the metal oxide. However, since selected sputtering may easily cause a change in the chemical state, care should be taken in using standard spectra measurements. It is necessary to attach the heating equipment to the analysis device for vacuum heating to occur. Therefore only institutions possessing such equipment can undertake the collection of standard spectra. In practice, examination of heating in the air was therefore possible.

MgO single crystal substrate was employed as a test sample. The effect of surface decontamination at respective temperatures ranging from 200 °C to 900 °C in steps of 100 °C was studied by XPS. The surface temperature of heated MgO decreased rapidly. Therefore it was preferable to transfer the sample into a vacuum chamber within two minutes of heating in the air.

The results of the experiment were:

- (1) from a peak of 300 °C the C-H (C 1s at 285eV) showed a decline.
- (2) the C-O (C 1s at 287eV and 290eV) increased to 300 °C.
- (3) the O 1s (532eV) showed an initial increase to 300 °C.
- (4) Subsequently O 1s (532eV) showed a decrease at the 700 °C point.

In the case of (2) and (3) the origin of the C-O species was assumed to be the contaminant on the wall of the electric furnace or the crucible used in the pretreatment. The above case (4) suggests the decrease of O-H.

We were able to confirm that all but a small quantity of the contamination was removed at temperatures above $500\,^{\circ}\text{C}$. Furthermore, in the case of MgO we were able to ascertain that stoichiometry was achieved by heating to over $900\,^{\circ}\text{C}$.

Additionally, a repeat experiment on the original surface was conducted by placing a single drop of distilled water on the now cleaned MgO surface and was exposed to the air for one hour. Also this was compared to the ion sputtering on MgO.

We hope that the method of heating in the air will be adopted by many institutions in the form of pretreatment of standard spectra measurements in organic materials.