Quantitative Analysis of metal alloys by AES and XPS

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The surface composition of alloys has been extensively studied using Auger Electron Spectroscopy(AES) and X-ray Photoelectron Spectroscopy(XPS) to ascertain the equilibrium concentration of constituents . In addition, our studies have included a measurement of surface after ion bombardment to establish the nature and magnitude of the driving forces which lead to surface enrichment. But a lot of experimental results of various alloys are still required to a better understanding of surface composition changes by ion bombardment.

The AuCu alloys(Au25at%-Cu75at%,Au50at%-Cu50at%,Au75at%-Cu25at%)were prepared as specimens for round robin test of Japaneses VAMAS-SCA working group. These are used to test the reliabilty of the quantification of our system and compared with published data of AES and XPS quantification. The surface concentration of AuPd alloys(Au05at%-Pd95at%,Au25at%-Pd75at%,Au50at%-Pd50at%,Au75at%-Pd25at%,Au90at%-Pd10at%) and CuPd alloys(Cu10at%-Pd90at%,Cu25at%-Pd75at%,Cu50at%-Pd50at%,Cu75at%-Pd25at%,Cu90at%-Pd10at%,Cu95at%Pd5at%) have been measured by AES and XPS. The alloys were prepared by melting the constituents in an Ar arc using high purity Ar to ensure uniform composition and remelted several times, the final melt was cooled rapidly to encourage the development of fine grain polycrystalline structure. The composition and uniformity have been checked by EDAX on electron microscope.

The AES and XPS measurement were performed on VG-ESCALAB 210 at Chungbuk Nat'l University. This is a routine analysis system comprising LEG1000 electron gun, dual(Al,Mg) and Monochromated Al X-ray source and a hemispherical electrostatic analyser placed at 45° to the electron beam and its base pressure was 5×10^{-11} mbar. The alloy samples were cleaned by 3 KeV Ar^{+} ion sputtering until any contamination such as carbon and oxygen cannot be detected by AES and XPS measurement. AES was measured with the 20 nA sample current of 5KeV e-beam with constant retard ratio mode(2 or 4). XPS measurement was done with Mono-Al X-ray source using constant pass energy mode(10 eV).

There are two methods in AES and XPS quantification. One is to use relative sensitivity factors which are provided by each manufacturer, and the other is to use pure metals as standard materials. Many studies have been reported that the latter method shows more reliable quantification than the former one. In this study, therefore, pure metals were used as standard materials. The matrix correction factor was considered including the backscattering factor (in AES, not in XPS), the inelastic mean free path(IMFP) and the density correction. In AES quantification, the backscattering factor was obtained using S.Ichimura and R.Shimizu's empirical formula which is based on their Monte Carlo calculation. IMFP was calculated using Tanuma-Powell-Penn formula in XPS and AES quantification. Figure 1 shows the surface composition of Au calculated from Auger peak of Au-MNN at 2024 eV and Cu-LMM at 920 eV as a function of bulk composition. As can be seen in the figure, the surface composition coincides with the bulk composition. The XPS quantification from Au $4f_{7/2}$ and Cu $2p_{3/2}$ peaks shows the same result as AES quantification. It means that no surface segregation is observed in AuCu alloys after ion sputtering as far as high energy Auger transition is considered in the estimation of the surface composition. It is in fairly good agreement with the reported results including the results of Japaneses VAMAS-SCA working group. Figure 2 and 3 show the surface composition of Pd obtained with Pd-MNN, Au-MNN and Cu-LMM Auger peak, respectively. In both AuPd and CuPd alloys, the surface compositions have been found to be Pd rich compared with bulk values. It is of interest to note that the surface concentration has large difference from the bulk concentration in Pd 50at% samples compared with other concentrations. The surface composition changes after sputter cleaning of alloy samples have been observed for some alloys such as CuNi, CuZr,etc, even if the high Auger transitions are used to estimate the surface concentration. Recently, these surface composition changes of alloys after ion sputtering have been understood as the results of preferential sputtering, radiation induced surface segregation and radiation enhanced diffusion. To confirm whether the AuPd and CuPd alloys have the same effects or not, Ion Scattering Spectroscopy(ISS) measurement having sensitivity of outmost atomic layer is necessary. In summary, the surface concentration of alloys can be obtained with high accuracy by AES and XPS quantification if using pure metals as standard materials and carefully considering matrix effects such as backscattering factor and IMFP.

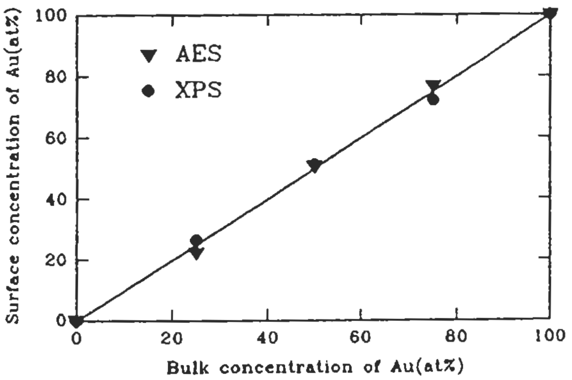

Fig.1 Determination of Au-Cu alloy surface composition by XPS and AES

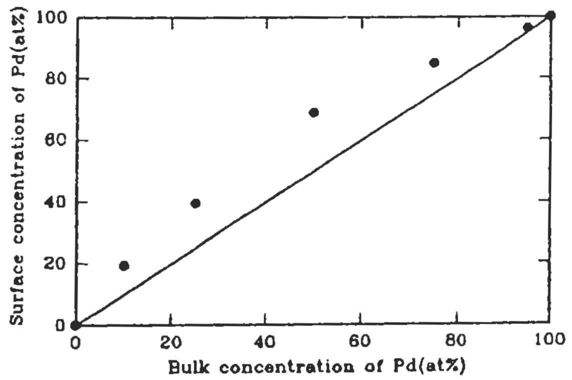

Fig.2 Determination of Pd-Au alloy composition by AES

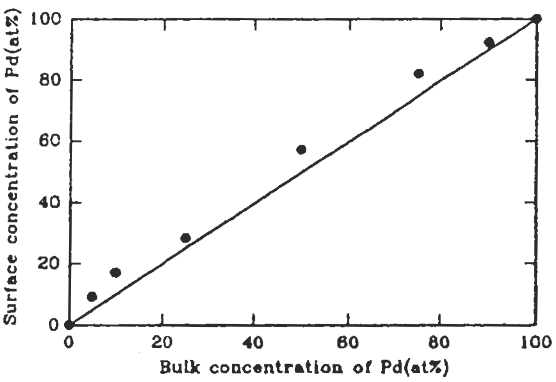

Fig.3 Determination of Pd-Cu alloy composition by AES