

Comparison between Soft and Hard X-ray Core-Level Photoemission of Invar Alloy

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Soft and hard X-ray core-level photoemission (SXPES and HAXPES) of the Invar alloy has been performed after *in-situ* Ar-ion bombardment. It is found that Fe 2p XPS lines have prominently broad tails on the larger binding energy side.

1. Introduction

Invar alloy shows a nearly-zero thermal expansion coefficient in a wide temperature range from room temperature up to about 500 K [1]. It has been studied for a long time, from the basic to the technological point of view. This is not only because of its importance in application, but also because it has become clear that there is very important science hidden behind its mechanism [2].

2. Experimental

Polycrystalline sample of Invar alloy with typical composition of Ni (36.2 mass%)-Fe (Bal.) was supplied by Shinhokoku Material Corp. The sample was polished in air and an area of 2×2 mm² was Ar-ion bombarded *in-situ* for 5.5 minutes with energy of 500 eV and ion current of 30 nA. Core-level photoemission was measured at SA-1 of the SR Center using a scanning soft and hard x-ray photoemission spectroscopy apparatus (ULVAC-PHI Quantes). Both surface sensitive soft x-ray photoemission (SXPES) measurements by Al K α emission (1486.6 eV) and bulk sensitive hard x-ray photoemission (HAXPES) by Cr K α emission (5414.7 eV) have been performed.

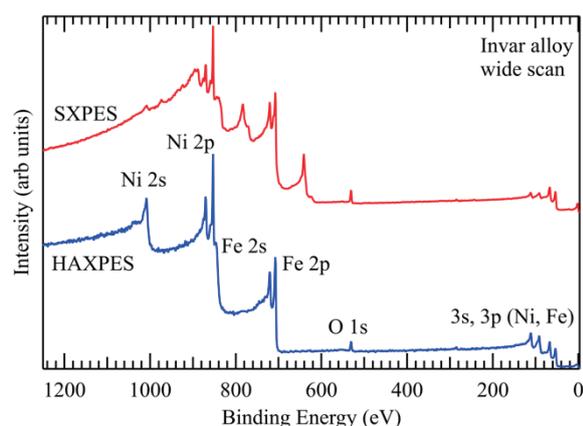


Fig. 1 Wide energy region scan results of the Invar alloy sample after *in-situ* Ar-ion bombardment.

3. Results and Discussion

The results of wide energy scans are shown in Fig. 1. Core-level XPS lines for 2p, 3s, and 3p levels of Fe and Ni are seen in both SXPES and HAXPES. On the other hand, Ni 2s level is prominent in HAXPES but is almost negligible in SXPES. One of the reasons for this is that the cross section of Ni 2s becomes relatively larger as the photon energy becomes larger. Another reason is that the Ni 2s intensity in SXPES is strongly suppressed because escape depth of photoelectron quickly decreases as the binding energy becomes larger due to the smallness of the excitation energy. In SXPES, LMM Auger lines of Fe and Ni are also seen in the binding energy region between 600 and 900 eV. The origin of the O 1s XPS peak will be discussed below in relation with Fig. 2.

Figure 2 shows the SXPES and HAXPES results for the energy region of 3s and 3p core-level XPS of Fe and Ni. The intensity of 3s lines relative to 3p is stronger in HAXPES than in SXPES. This reflects the difference in the photon-energy dependence of cross section between 3s and 3p levels; p level tends to drop faster than s level as photon energy increases. The lineshape of each peak is essentially the same between SXPES and HAXPES.

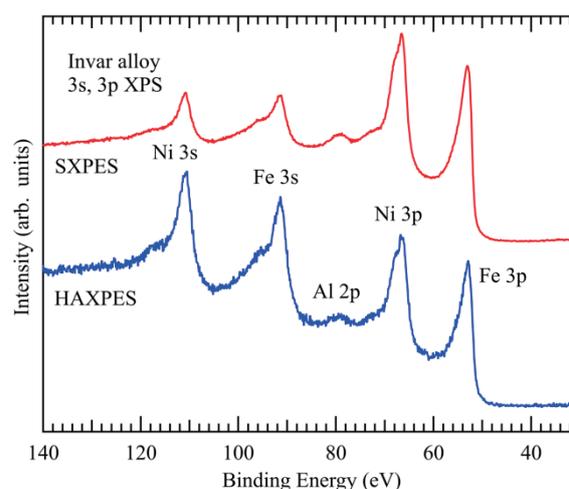


Fig. 2 3s and 3p XPS of Fe and Ni for the Invar alloy sample. Finite Al 2p XPS intensity is presumably due to contamination by Al₂O₃ powder for polishing.

The small peak around 80 eV in Fig. 2 is considered to be due to Al. Although very small amount of Al is added in the Invar alloy as deoxidizer, the intensity of the peak is much larger than expected from the added Al amount. The existence of both Al 2p and O 1s intensities could be due to residual Al₂O₃ powders used for polishing the surface of the sample.

Figure 3 shows the result of Ni 2p XPS, where SXPES and HAXPES lineshapes are essentially the same. A satellite is found for each of Ni 2p_{3/2} and 2p_{1/2} peaks at 6 eV larger binding energy than the main peak. This "6-eV satellite" is also found in pure Ni and other metallic Ni compounds.

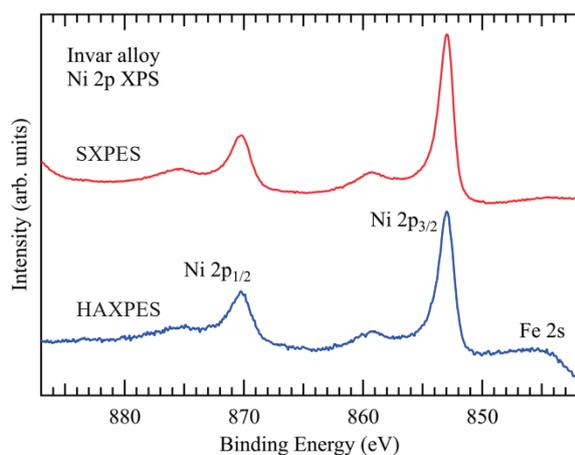


Fig. 3 Ni 2p XPS for the Invar alloy sample. Fe 2s XPS peak at 845 eV is clearly seen in HAXPES. In SXPES, a part of the L_{2,3}M_{2,3} M_{2,3} Auger line of Fe located around 900 eV is also seen.

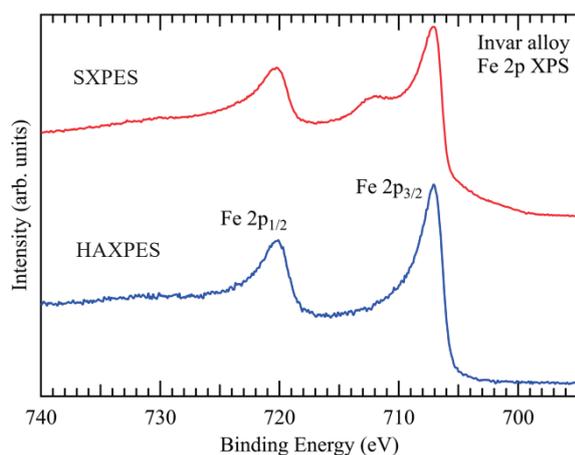


Fig. 4 Fe 2p XPS for the Invar alloy sample. Broad tail has been found in the Fe 2p_{3/2} peak for HAXPES.

Figure 4 shows the result of Fe 2p XPS. The main difference between SXPES and HAXPES results is that a peak is found around 713 eV in the SXPES spectrum. The tail between 700 and 705 eV in SXPES is expected to be the tail due to the peak at 713 eV. This peak is due to the L₃M_{2,3}M_{4,5} Auger line of Ni. We now focus on the bulk sensitive HAXPES result. The peak energy of 707 eV for the Fe 2p_{3/2} component is quite typical of metallic state of iron. However, the tail of Fe 2p_{3/2} is found to be much larger than that for pure Fe and most of metallic iron compounds [3]. It is known that multiplet structures due to local magnetic moment are found in the larger binding energy side of the Fe 2p_{3/2} peak of magnetic Fe systems [4]. Therefore, the broad tail in the present result is consistent with the fact that the size of the local Fe moments in the Invar alloys are different on Fe sites with different environment due to disordered nature of the crystal structure.

4. Conclusions

Core level photoemission spectroscopy of the Invar alloy has been performed by means of soft and hard x-ray photoemission. Finite Al and O spectral features have been detected, suggesting residual Al₂O₃ powders for polishing on the sample surface. Fe 2p XPS was accompanied by a broad tail on the larger binding energy side, suggesting that the size of the local Fe moment is different on different Fe site in the Invar alloy.

Acknowledgement

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References

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