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X-ray Photoelectron Spectroscopy (XPS) analysis of low oxidation pressure behavior of FeAl –alloys at 600°C and 700°C

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Abstract. The low oxidation pressure behavior of binary FeAl alloys has been examined at different temperatures of 600-700°C. Polycrystalline and single-crystalline samples of Fe-10Al and Fe-25Al were oxidized for 1 h at 10⁻⁶ mbar of oxygen in an ultra-high-vacuum chamber (UHV). Prior oxidation, samples were prepared by sputter-annealing cycles in UHV. After oxidation, the samples were analyzed by X-ray Photoelectron Spectroscopy (XPS). The formed oxide layers on the surfaces consist of FeAl₂O₄ and Al₂O₃ showing that Al has a dominating role during the oxidation process. From XPS peaks, the composition of the Fe peaks of the oxide layer consists of Fe metal, FeAl-alloys, and FeO. Meanwhile, the composition of the Al peak is changed from Al metal, FeAl-alloys and Al₂O₃ at 600°C to Al metal, FeAl-alloys and FeAl₂O₄ on 700°C. From XPS sputter depth profiles, it is shown that the oxide layer was growing with increasing temperature, and the single-crystalline Fe-10Al has a thicker formed oxide layer than poly-crystalline Fe-25Al with same oxidation treatment.

1. Introduction

Iron aluminides has been applied mostly for structural applications with operational temperature between 600-800°C [1]. Oxidation on iron aluminium at 500-600°C produces oxide layers consist of α -Fe₂O₃, Fe₃O₄, and FeAl₂O₄. In this state, oxidation is dominantly controlled by diffusion of Fe ions. Above this temperature range, Fe₃O₄, and FeAl₂O₄ will disappear to become γ -Al₂O₃ until 900°C. Oxidation above 900°C will lead to formation of α -Al₂O₃ [2-4].

There are so many publications about oxidation study of iron aluminides in high pressures, but only a few were conducted in low oxidation pressures particularly with x-ray photoelectron spectroscopy (XPS) analysis [5-7]. Therefore, in this article, we report a study of x-ray photoelectron spectroscopy (XPS) analysis during oxidation of FeAl-alloys on 600-700°C with low base pressure system in order to understand oxidation growth mechanism.



2. Experimental Procedure

For this experiment, a poly crystalline and a single crystal of iron aluminides with compositions of 25 at.% Al (Fe-25Al) and 10 at.% Al (Fe-10Al) were prepared respectively. The polycrystalline Fe-25Al samples were produced by vacuum induction melting in Al_2O_3 crucibles, using elemental components with a purity higher than 99.9% and cold Cu moulds for solidification process in order to obtain 10 mm diameter of rods. The samples were cut to pieces with 8 mm diameter and 3 mm thickness using spark erosion technique. Then, the samples were mechanically grinded and polished up to 0.05 μm surface roughness with diamond paste and silica (SiO_2) suspension for the final polishing. Meanwhile, the single crystal sample Fe-10Al (111) was prepared by mechanically polished down to 0.03 μm roughness and oriented with an accuracy of $< 0.1^\circ$ (Surface Preparation Laboratory, Netherlands).

The oxidation tests were conducted on 600-700°C for 1 hour in an UHV chamber by flowing pure oxygen (O_2) gas into the chamber with base pressure 10^{-5} - 10^{-6} mbar. Before oxidation, all the samples were cleaned by several cycles of sputtering using Ar^+ ions (Energy $E = 1.5$ keV, sputter current $I = 1.85$ mA) and short annealing on 900°C.

The oxidized samples were characterized using X-ray photoelectron spectroscopy (XPS) (Physical Electronics Quantum 2000) in order to study the element composition and chemical state of resulted oxide layer on the sample surface. On each sample, survey spectra and sputter depth profiles were taken using a monochromatic Al $K\alpha$ X-ray source, source analyzer angle of 45° , step size of 0.8 eV, pass energy of 93.90 eV and a number of sweeps in the range of 8 to 25. Survey spectra were recorded within the binding energy range of 0-1350 eV, and sputter depth profiles with 0.5-1 keV Ar^+ ions were measured of Fe 2p, Al 2p and O 1s photo lines. An oxidized silicon wafer of known oxide thickness was used for sputter time calibration. Then, this calibrated sputter time with SiO_2 was used for calculating the depth of sputter profile. In order to analyse the XPS data, the Casa-XPS software was used including sensitivity factors of 2.14469 for Fe 2p, 0.301653 for Al 2p and 0.733 for O 1s. The resulted XPS spectrum of each element was calibrated first with C 1s = 285 eV before data analyses begins.

3. Results and Discussion

Figure 1 shows Auger Electron (AES) spectra of sample surface, as received from the company, after some cycles of surface cleaning using sputtering-annealing method and after oxidation test. The sputtering-annealing is usually able to remove all contaminant and oxidized parts on the surface. But, during this research, even with prolonging sputter time and increasing annealing temperature, the sample surface cannot be perfectly cleaned particularly from oxygen contamination due to CO adsorption which was still present in UHV pressures (10^{-6} - 10^{-10} mbar), and high reactivity of Fe and Al elements with oxygen gas.

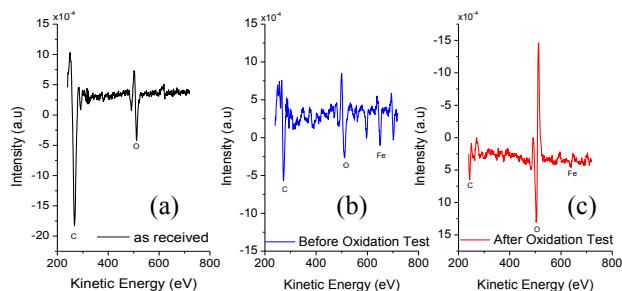


Figure 1: Auger Electron (AES) Spectra of Fe-10Al surface sample as received, after cleaning process with some cycles of prolonging sputtering-annealing, and after oxidation test.

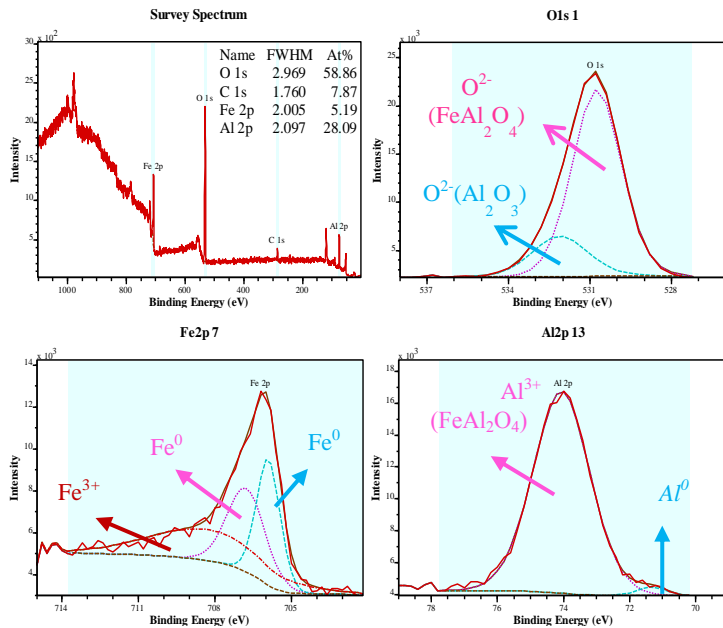


Figure 2: Surface spectrum and high resolution spectra of O, Fe and Al elements on oxidized Fe-10Al at 600°C for 1 hour.

From all survey spectrum in figure 2 and 3, with the same condition of oxidation process at 600°C and 1 hour, it shows that the Fe-10Al has the higher O content than that of Fe-25Al. With this result, the Fe-10Al has less oxidation resistance than Fe-25Al. Meanwhile, for the same composition and duration of the oxidation process, Fe-25Al produce more oxide layer after oxidation process at 600°C rather than after 700°C, as shown in figure 4.

Fe signals of oxidized Fe-10Al from oxidation on 600°C (in figure 2) show the metallic state of Fe still has higher intensity comparing with the oxide signals. But, the oxide signal has already grown enough. Meanwhile the Al spectrum has very high oxide peaks and almost no metallic Al peaks. This condition is supported from the spectrum of O peaks which consist of majority aluminium oxide compounds, FeAl₂O₄ and Al₂O₃.

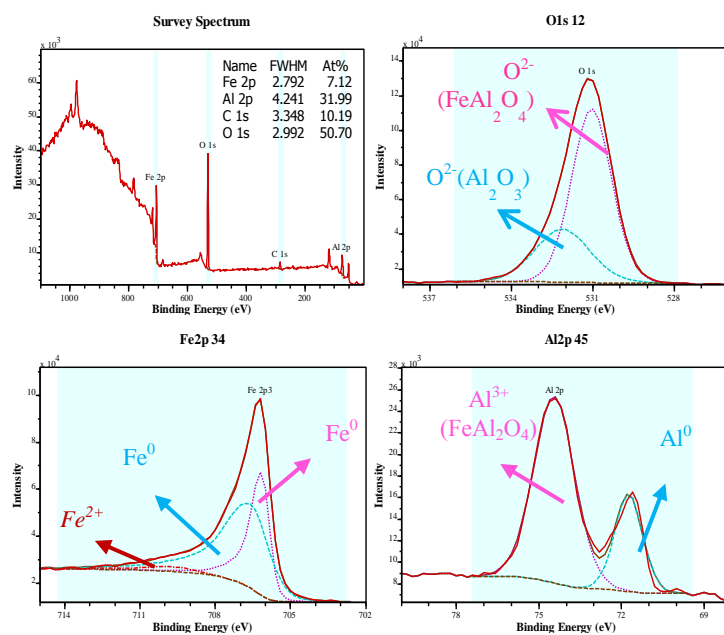


Figure 3: Surface spectrum and high resolution spectra of O, Fe and Al elements on oxidized Fe-25Al at 600°C for 1 hour.

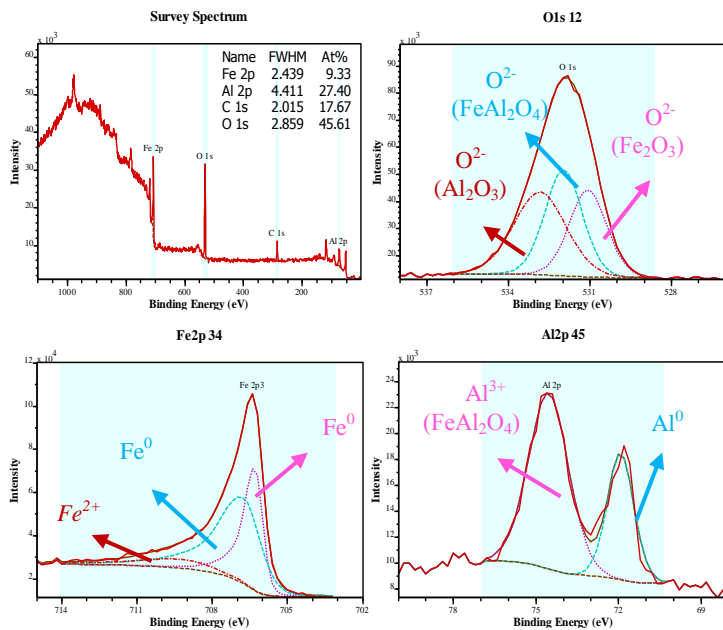


Figure 4: Surface spectrum and high resolution spectra of O, Fe and Al elements an oxidized Fe-25Al on 700°C for 1 hour.

Figure 3, Fe-25Al after oxidation on 600°C for 1 hour, it has the same characteristic of Fe, Al and O spectrum from the Fe-10Al results with the same oxidation condition, as shown in figure 2, but with lower Fe oxide peak on Fe spectrum and higher Al metallic peak on the Al spectrum. These high resolution spectra validate the survey spectra results (in figure 2 and 3) that Fe-25Al has a higher oxidation resistant than that of the Fe-10Al.

Meanwhile the high resolution spectra of the oxidized Fe-25Al on 700°C for 1 hour (in figure 4) show fewer Fe oxide signals on the Fe spectrum and higher Al metallic than oxidation results of Fe-25Al at 600°C for 1 hour. But, the interesting result comes from the O spectrum that shows trivalent oxide peaks with more iron oxide content.

The depth sputter profiles of all oxidized Fe-10Al and Fe-25Al on 600-700°C for 1 hour are shown in figure 5. The profile shown Fe-10Al with 600°C for 1 hour has the thickest oxide layers. Meanwhile, the Fe-25Al from 700°C for 1 hour has slight thicker oxide layer than from 600°C for 1 hour. This might be due to formed trivalent oxide peaks on the oxide layer.

Overall, from all XPS spectrum and depth sputter profiles of Fe-10Al and Fe25Al, the low oxidation pressures have limited the growth of iron oxide, thus aluminium become more dominant than the iron during oxidation process on higher elevated temperatures [3, 7, 8]. But, in the more oxygen pressure, such as in atmospheric pressure, the iron is more dominant during oxidation process [2-4]. These phenomena can be explained through Ellingham diagram [9]. On the same temperature, the formation of iron oxide, FeO/Fe₂O₃/ Fe₃O₄ requires more oxygen pressure (p_{O_2}) than the formation of aluminum oxide. This experiment results also show no any difference of oxidation growth between the polycrystalline and single crystal samples during this experiment.

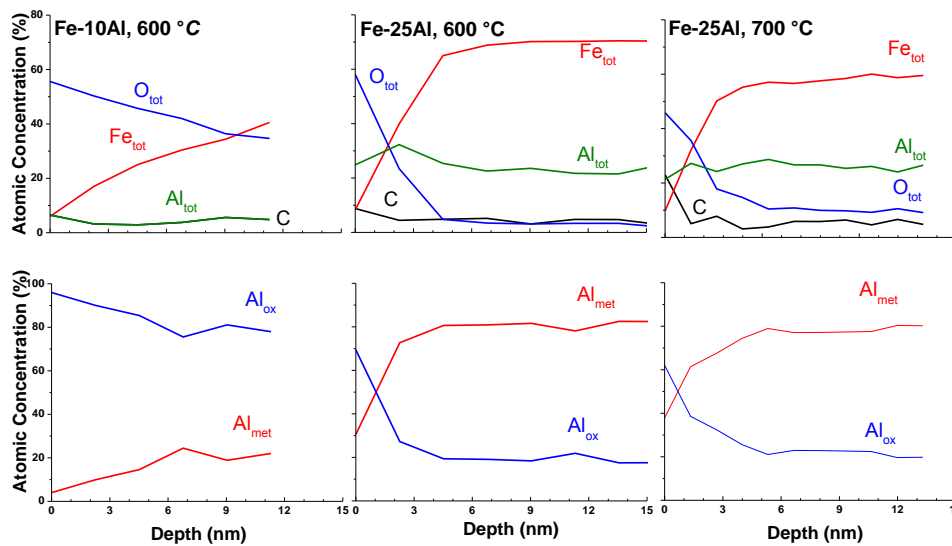


Figure 5. Sputter depth profile of oxidized Fe-25Al on 600-700°C for 1 hour respectively.

4. Conclusion

Fe-10Al has a lower oxidation resistance than Fe-25Al composition. The depth sputter profile shows that Fe-10Al produces the thickest oxide layer. Meanwhile, for the same composition, Fe-25Al from 700°C for 1 hour has thinner formed oxide layer than from 600°C for 1 hour, but with trivalent oxide formation. The most important thing from this study, the aluminium has the majority role during oxidation at 600-700°C for 1 hour with low pressure system.

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