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"Atom-Probe Field-Ion-Microscopy Study of Fe-Ti Alloys"

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(Title of paper) Atom-probe Field Ion Microscopy Study of Fe-Ti Alloys

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A newly developed high-performance atom-probe (field ion microscope) was employed for the composition analysis of Fe-Ti alloys and their interactions with ambient gas, such as H_2 and O_2. With a mass resolution ($m/\Delta m$) better than 2,000 and a spatial resolution of a few Å, all isotopes of Fe and Ti and their hydrides and other compounds are clearly resolved during the depth profile study. Some of our findings are: (1) Titanium segregated on the surface and grain boundaries upon heating ($\gtrsim 900°C$), in the form of oxides, and (2) some Ti in the bulk forms clusters of various sizes with C, O, and/or N as nuclei.

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ATOM-PROBE FIELD ION MICROSCOPY STUDY OF Fe-Ti ALLOYS

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The original time-of-flight (ToF) atom-probe (field ion microscope, FIM) built by Müller and Panitz in 1967 has evolved into a much improved microanalytic tool, capable of achieving a mass resolution ($m/\Delta m$) of 2000 and signals free of artifacts. This was accomplished by incorporating Poschenrieder electrostatic focusing lens. Because of these improvements, this instrument can now be applied for the investigation of various problems in materials science and technology which heretofore were not amenable to study by the atom probe.

We have applied this device for the investigation of a Fe-Ti alloy. A small block of a Fe-0.15 wt% Ti alloy, after rolling to a thickness of 0.04 cm, was cut into a number of strips of 0.3 cm width, and these were annealed at 1173 K for four hours at 10^{-2} Pa (10^{-4} Torr). FIM specimens in the shape of sharp needles were prepared by electrochemical etching in a HNO_3, HCl, H_2O (1:1:2) solution.

All of the experiments reported here were carried out at 78 K tip temperature and at a pressure less than 10^{-6} Pa (10^{-8} Torr). Prior to annealing at or above 1073 K in situ, the tip was field evaporated in hydrogen until an iron image was obtained in order to remove extraneous material including oxides known to form during air or acid exposure. Then, field ion images were obtained using Ne-H_2 gas mixtures, followed by compositional analysis of the tip surface by slow, controlled pulse-evaporation of the surface atoms. The mass resolution during pulse-evaporation was $m/\Delta m \simeq 1000$, sufficient to resolve all isotopes.

The compositional analyses were carried out under three different conditions: (a) After annealing at 1073 K for 300 sec., (b) after annealing at 1273 K for 300 sec. and (c) from the grain boundary after treatment (b). We found that titanium atoms segregate to the surface under appropriate thermal conditions. The first layer is always enriched with Ti, containing approximately 13% Ti when heated at 1073 K and 18% Ti at 1273 K, while only a 2% Ti content is measured at a distance of ~20 atom layers from the surface. The most pronounced enrichment of titanium is limited to the outer-most surface layer or at most first few layers.

In the outer most atom layer(s) titanium is in the form of oxide, mainly TiO and TiO_2. However, in the subsurface region Ti is mainly in the form of elemental atoms, except for a small percentage (~1%) which is in the form of clusters. In these cases, without exception, at least one carbon, nitrogen, or oxygen compound of titanium was detected for each cluster TiO was most common, but TiC, TiN, Ti_2O, Ti_2C and Ti_3C were also detected as apparent centers for other Ti atoms in the cluster. In addition to these compound forms, there were several atomic C, N or O detected with each cluster, suggesting that a Ti cluster is the impurity trapping center. The distribution of clusters are random and their size varies up to two dozen titanium atoms per cluster. This probably is the first direct observation of clustering phenomena in metals.

Grain boundary segregation is also of great scientific and technological interest. The probe-hole, with a sampling area of approximately three atoms diameter, was positioned over the grain boundary, which could be clearly imaged, and a series of pulse evaporations were carried out. An abundance of Ti atoms was detected at the grain boundary, the average Ti content was approximately 6%. When the probe-hole was shifted to either side of the grain boundary, the titanium content dropped to the lower (2%) level reported above. The data show no dependence of grain boundary concentration on a distance from the surface in the present study. A Ti content of 6% in an alloy containing nominally 0.15% Ti clearly indicates that Ti atoms segregate to the grain boundaries. Although, segregation has been studied extensively this is probably the first atomic scale demonstration of segregation by atom-probe FIM.

All Ti atoms detected along the grain boundary were without exception, in the form of the oxide, mostly TiO and some TiO_2. No Ti atoms or compounds of carbon or nitrogen were observed, even though significant levels of O, N and C were detected at the grain boundary. It is quite possible, even likely, that these internal oxides formed already during melting of the alloy, in view of the strong gettering action of titanium as indicated by the free energy of formation TiO_2.

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