I. 13. Thermal Desorption Spectroscopy (TDS) Study of Rare Gases Implanted to Pure Fe

Hanada R. and Saito S.

Institute for Materials Research, Tohoku University,

Introduction

Rare gases are practically insoluble to Fe and yet can be implanted if accelerated to several 10keV by ISOL. This paper reports the result of TDS study of Ar and Kr implanted to pure Fe with the energy between 20 and 60keV. The purpose of the experiment is to examine the effect of the phase transformation on TDS of rare gases from Fe. Such an example has been reported for Ar from Co. (1)

Fe has three phase transformations. Namely,1) Ferromagnetic to paramagnetic transition at 768C(1041K). (2) bcc (body centered cubic) phase (α-phase) to fcc (face centered cubic) phase(γ-phase) at 906C(1179K) and (3) fcc-phase(γ-phase) to bcc phase(δ-phase) at 1401C(1674K). In the present study, TDS was measured between RT and 1000C to examine the effect of the transformations (1) and (2).

Experimental

Fe specimens are high purity (99.995%) polycrystalline foils(10×15×0.3 mm³) from Johnson Matthey Inc. They were annealed at 800C for 2 days in an UHV for the recrystallization. Because of the long annealing time, the grains grow to 2-5mm in diameter. Ar and Kr were implanted to them at RT using ISOL at CYRIC. The acceleration energies were 20-60keV and the doses were between 10^{16}-10^{17}/cm². The method of the TDS measurement is exactly the same with those reported in ref. (2).

Result

Figure 1 shows the TDS for Kr implanted to Fe at 40keV with three different doses. One prominent gas release is observed at 1000K for the specimens implanted above 4×10^{16}/cm² and the other at 1200K for all specimens. Similar results are obtained for Ar implanted Fe and especially the TDS peak at 1200K has been observed all specimens investigated.

These results clearly show a prominent rare gas release takes place at or right after the phase transformation (2), namely, at the α-γ phase transformation.
Discussion

First let us discuss the site of the implanted rare gases in Fe lattice. Since rare gases have much larger atomic sizes (Ne:3.20, Ar:3.82, Kr:4.0 and Xe:4.40A)\(^3\) than the interstitial sites of bcc Fe lattice (the o-(octahedral) site:0.19 and the t- (tetrahedral) site:0.36A) or fcc Fe lattice (the o-site:0.28 and the t-site:0.52A)\(^4\), it is very unlikely that they can stay at these sites as interstitial impurities. So even when they happen to land on these interstitial sites after the implantation, they should change the site to the substitutional site either by pushing out the Fe atom or by finding a lattice vacancy and sitting in it. Although the energetics of these processes will be a subject of further considerations, it is usually found in the hyperfine interactions studies that the implanted heavy ions are found at the substitutional site. So let us assume that the implanted Ar or Kr are at the substitutional site.

The first possibility to explain the observed gas release at the phase transition will be the different diffusion coefficients in the different phases. Namely, the diffusion coefficient in the fcc phase (γ-phase) is higher than that of the bcc phase (α-phase) so as the immobile rare gases in the bcc phase start to migrate when the host bcc lattice changes the structure to the fcc structure. However this model does not apply to the substitutional impurities in Fe. Namely, the inspection of the compiled data in Fe reveals that the diffusion coefficients in the fcc phase is 2 order of magnitude smaller than that of the bcc phase for all substitutional impurities investigated\(^5\). So if this rule applies to the rare gas atoms in Fe, we cannot ascribe the observed gas release upon the phase transition to the difference in the diffusion coefficient.

The second possibility is to ascribe it to a short circuit diffusion (The faster diffusion that takes place in the paths other than the bulk, for instance, along the grain boundaries or the dislocations)\(^6\). Namely, it is usually found that a high density of dislocations are formed during the phase transformation, for instance, in the martensitic phase transformations in the alloyed steels. The cause of the dislocation production has been known as to relieve the internal stress caused by the different unit cell volume in the different phase, that can be imagined easily by the Kurdjumov-Sachs relation for bcc to fcc transition\(^7\). If this applies to the present case, a high density of dislocations is formed at the phase transition temperature and the rare gas atoms diffuse along the dislocation lines (a pipe diffusion, one mechanism of a short circuit diffusion) to be released from the surface. One difficulty of this model is that whether a high density of dislocations is formed during the slow warming up rate (20K/min) as in the present and also if formed they may be annealed out instantaneously because of the high temperature (1200K) where the release takes place.

The other mechanism of the short circuit diffusion is the grain boundary diffusion. If the α-γ phase transition is caused by the nucleation and growth mechanism (the production of the small γ nuclei in α matrix and the subsequent growth that results in the γ bulk), new grain boundaries are formed in the γ phase. So rare gas atoms diffuse along the new grain boundaries. As has been known from the diffusion experiments, grain boundaries as well as
dislocations have been known as a path for a short circuit diffusion. One difficulty of this model is then why no grain boundary diffusion takes place in the α phase up to 1200K.

Several possibilities above are based on the assumption that the implanted rare gases are present in the Fe lattice more or less as an isolated solute. However as has been discussed in ref.2), the implantation dose is high in the present and the local rare gases atom concentration becomes quite high as to form gas bubbles. Also a channeling study of Kr implanted to Al has revealed that an ordered compound of Kr is formed in the Al lattice8). If these are the case for the rare gases in Fe, the interaction of the gas bubble or the compound with the stress or the structure change due to the phase transformation must be examined that is the beyond the scope of the present paper.

Technologically, the sudden gas release from the structure materials at the phase transformation temperature might be hazardous in a fusion or fission reactor operation and the methods of the protection must be found.

Further experimental works are now in progress.

Acknowledgements

The discussion with Prof.Shigeru Suzuki (Institute for Advanced Materials Processing, Tohoku University) is deeply appreciated.

This work is supported by a Grant-in Aid for Scientific Research in a Priority Area from the Ministry of Education, Science and Culture, Japan.

References

Fig. 1. TDS spectrum for Kr implanted to Fe to various doses Warming up rate 20K/min.