PHASE TRANSFORMATION IN He⁺ AND H⁺ ION IRRADIATED TYPE 304 STAINLESS STEEL

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\(\gamma\) (fcc) \(\rightarrow\) \(\alpha\) (bcc) phase transformation in type 304 stainless steel has been observed after irradiation of He⁺ and H⁺ ions up to fluence levels of \(10^{17}\) and \(10^{18}\) ions/cm², respectively. Depth selective conversion Mössbauer spectroscopy and surface-sensitive X-ray diffractometry were employed to study the effect of irradiation. It is shown that the amount of the ion induced phase is highly sensitive to the fluence, the ion species and depth from the surface. It is worth noting that H⁺ ion irradiation is rather ineffective in inducing the transformation.

1. Introduction

Austenitic stainless steel is considered prospective materials for the first wall of thermonuclear reactors, and a number of works has been done to study the changes in physical properties of the alloys under He⁺ and H⁺ irradiation /1/. Conversion electron Mössbauer spectroscopy (CEMS) is a particularly suitable technique to study the irradiation effect in such alloys, providing micro-structural information on atomic scale. Moreover, depth profiling of the spectra (DCEMS) could give further insights on property changes in the implanted surface zone.

We have shown that \(\gamma\) (fcc) \(\rightarrow\) \(\alpha\) (bcc) phase transformation is induced in He⁺ implanted 304 and 316 stainless steels and Fe⁺, Ni⁺, Kr⁺ and Ar⁺ implanted 17/7 stainless steel and that the transformation has a martensitic nature /2, 3/. In this report DCEMS has been measured in He⁺ and H⁺ implanted 304 stainless steels. The results make clear that the amount of the transformation strongly depends on the depth from the surface and ion species.

2. Experimental

10 \(\mu\)m thick foils of commercial grade 304 stainless steel (annealed) were used in this study. The ion irradiation was carried out, normal to the specimen's surface, with mass analyzed He⁺ and H⁺ ions from a low energy DC accelerator. The beam energy was kept at 8 keV. The projected ranges of He⁺ and H⁺ ions in stainless steel were estimated to be about 40 nm and 50 nm, respectively. The specimen temperature was about 200°C during irradiation.

CEMS spectra were taken at room temperature in a standard constant acceleration spectrometer, using a 5 mCi \(^{57}\)Co source in Rh matrix. Backscattered conversion and Auger electrons were detected by a gas flow proportional counter, employing a mixture of 90% He – 10% CH₄. We have demonstrated that depth profiling of CEMS (DCEMS) can be effectively measured by the gas proportional counter and DCEMS can provide useful information on physical properties at the
damaged surface zone /4/. The surface conditions are also
determined by a surface-sensitive X-ray diffractometer with grazing
incidence (~2°) of Cu Kα-ray (Rigaku Corp., RAD-B).

3. Results and Analysis

Fig. 1 shows the changes in X-ray diffraction patterns of the
stainless steel specimens after He⁺ and H⁺ irradiation to high
fluences such as 10¹⁷ ions/cm². While (110) peak from bcc α phase
can be clearly seen after He⁺ irradiation of 10¹⁷ ions/cm² and
increases with increasing He⁺ fluences, the diffraction peak in H⁺
ion-irradiated specimens is detected only after fluence levels of
10¹⁹ ions/cm². The result indicates that the observation by X-ray
diffraction, which can be done in a few hours, is effective and
useful in estimating the phase condition in the vicinity of
irradiated alloy surface.

Fig. 2 shows CEM spectra taken from the He⁺ ion irradiated
stainless steel up to two fluences. 2(B) - 2(D) spectra are DCEMS
from the specimens irradiated to a fluence of 4.7 x 10¹⁷ ions/cm².
The spectra are characterized by a superposition of the paramagnetic
peak from the austenite (fcc γ phase) and the six peaks from the
ferromagnetic bcc (α) phase. The DCEMS clearly demonstrates that
the amount of the induced α phase decreases distinctly with
increasing the depth, i.e. from the top surface, 2(B) spectrum, to
the inside, 2(D) spectrum. The relative amount (area ratio) of the
phase to total ones are estimated as 68%, 79%, 63% and 38% for 2(A),
2(B), 2(C) and 2(D) spectra, respectively. It is noted that the
hyperfine parameters for the paramagnetic peaks remain unchanged
through Fig. 2 spectra, with I.S. of -0.11 mm/s (relative to α iron)
and line width of 0.35 mm/s.

Fig. 3 shows efficiency curves of the transformation, defined as
the relative area of α phase in CEM spectra, plotted against He⁺ and
H⁺ ions fluences. The figure shows dependences of the efficiency