A Study of Temper Brittleness of Tin Doped Steels by Auger Electron-Spectroscopic Method*

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The phenomenon of temper brittleness in alloy steels is known to be caused by segregations of phosphorus, antimony, arsenic, and tin to prior austenite grain boundaries by a reversible process.¹⁻³⁾ This segregation has recently been clarified for the case of antimony and phosphorus by Auger electron microscopy.^{4,5)}

In the present study, the degree of segregation of tin to prior austenite grain boundaries was investigated by Auger electron spectroscopy.

Vacuum-melted 0.1%C-3%Mn-1%Sn alloys were prepared. After austenitized for 2 hr at 1300°C, materials were water quenched, then tempered for 1 hr at 675°C followed by water quenching. Embrittlement treatment was done for 5 hr at 550°C. By this treatment, brittle fracture occurred almost in intergranular mode. Auger electron spectroscopy was used to detect the segregating elements at the fracture surface. A display type LEED apparatus with four spherical grids was used as an energy analyzer. The details of the apparatus had been shown elsewhere.⁶⁾ Standard V-notch Charpy specimens were broken both in the air at liquid nitrogen temperature and in a high vacuum chamber at -160°C. Specially designed low temperature cleavage manipulator was used in order to fracture a V-notch Charpy specimen in a high vacuum chamber, which is shown in Photo. 1. The details will be described in another report.⁷⁾ After analyzing a given surface layer, the layer was removed by sputtering with Ar⁺ ion and the next layer was then analyzed.

Figure 1(a) shows the derivative of energy distribution function $dN(E)/dE$ of electrons emitted from the

intercrystalline surface of the specimen which was broken in the air at -196°C. The change of $dN(E)/dE$ due to the sputtering by Ar⁺ ion bombardment is also shown in Fig. 1(b) to (d). No peaks corresponding to tin could be observed in the case of as fractured-surface (Fig. 1(a)). It is noted, however, that tin peaks were observable after about 20 atomic layers being removed by sputtering (Fig. 1 (b)), and decreased by further sputtering (Fig. 1(c) and (d)).

From these results, it is concluded that the qualitative analysis of tin segregated to the grain boundary is successfully performed even when the specimen is fractured in the air but the surface adsorption and oxidation prevent an accurate examination.

Figure 2 shows $dN(E)/dE$ of electrons emitted from the fresh intercrystalline surface of the specimen fractured in the vacuum chamber of 2×10^{-8} Torr at the temperature of -160°C. All of the specimens were fractured in intergranular mode, which was confirmed

Fig. 1. Auger spectra from the fracture surface of 0.1%C-3%Mn-1%Sn steel (embrittled and broken in the air at -196°C) before and after sputtering of surface

- (a) As-fractured
- (b) After 20 atomic layers removed
- (c) After 40 atomic layers removed
- (d) After 60 atomic layers removed

Photo. 1. Low temperature cleavage manipulator

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Fig. 2. Auger spectra from as-fractured surface of 0.1%C-3%Mn-1%Sn steel (embrittled and broken in the vacuum chamber of 2×10^{-8} Torr at -160°C)

by scanning electron microscopy. The amount of segregated tin was obtained by comparing the peak height of tin at the fracture surface with those obtained from the interior of the same specimen. This figure also shows the absence of sulfur and oxygen peaks. The peak height ratio of Sn to Fe is shown in Fig. 3 as a function of distance from the fracture surface. If the peak height of the $dN(E)/dE$ peak for each element is assumed to be proportional to the amount of the element present,^{8,9)} it is concluded that tin is segregated to prior austenite grain boundaries at a concentration level about 7 times as high as that of the

Fig. 3. Sn/Fe peak height ratio of the $dN(E)/dE$ at various depths from the intergranular fracture surface of embrittled 0.1%C-3%Mn-1%Sn steel

bulk in this experiment.

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