Phase transformations induced by implantation of $^{12}C^-$ ions into α-Fe and AISI 304 and 316 stainless steels studied by CEMS and SEM

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Samples of AISI 304 and 316 stainless steels, initially in austenitic (first set) and martensitic states (second set) and α-Fe (third set), were implanted with 180 keV $^{12}C^-$ to a dose of 10^{17} atoms/cm^2 at room temperature. Surfaces were examined by SEM (scanning electron microscopy) and the crystalline-phase fractions were estimated through CEMS (conversion electron Mössbauer spectroscopy). Different grades of etching were produced by sputtering during the implantations on the stainless steel samples depending on the previous crystallographic states. CEMS data reveal the $\gamma \rightarrow \alpha'$ transformation in the initially martensitic samples and no noticeable modifications as a result of the implantation on α-Fe and austenitic samples.

1. Introduction

In AISI 300 stainless steels the martensite to austenite ($\alpha' \rightarrow \gamma$) and austenite to martensite ($\gamma \rightarrow \alpha'$) phase transformations induced by ion implantation are processes not well understood yet. Several investigations have been performed in similar systems [1,2] but different explanations have been proposed regarding the type of mechanism through which the observed transformations are driven. It has been suggested [3] that ion implantation not only produces strain but also a state of stress inside the material that would enhance the phase transformations. In this work we study the surface alterations by SEM and the induced phase modifications by CEMS that take place after $^{12}C^-$ implantation.

2. Experimental

Specimens of commercial 304 SS and 316 SS, were rolled at room temperature from 8 mm down to 0.5 mm. A structure about 80% martensitic in SS 304 and about 60% in SS 316, as revealed by X-ray analysis, was achieved by this plastic deformation. Some specimens were afterwards annealed at 1050°C for 30 min and an about 100% austenitic structure resulted for both stainless steels. A $^{12}C^-$ beam from the sputtering ion source of the TANDAR accelerator was used for this work. The C^- ions were analyzed by a 90° deflector magnet and accelerated to an energy of 180 keV. The samples were placed on a massive brass target holder. The beam size was defined by a 2 cm circular aperture installed about 5 cm over the target position. Owing to a good thermal contact between the samples and the target holder the temperature rose only 20°C above room temperature during irradiation. The ionic and Ti sublimation vacuum pumps used kept the pressure near 10^{-7}–10^{-8} Torr. The implanted doses on 304 and 316 SS samples were 2.62×10^{17} atoms cm^{-2} with a beam current of 6.9 μA and on α-Fe samples was 8.9×10^{17} atoms cm^{-2} and a beam current of 3.3 μA. Mössbauer spectra were obtained as described in ref. [4]. Non-implanted samples were etched with appropriate reactives. All the implanted samples were studied by SEM without previous chemical etchings.

3. Results

Figs. 1a–5a show the optical micrographs before implantation, made by compar-

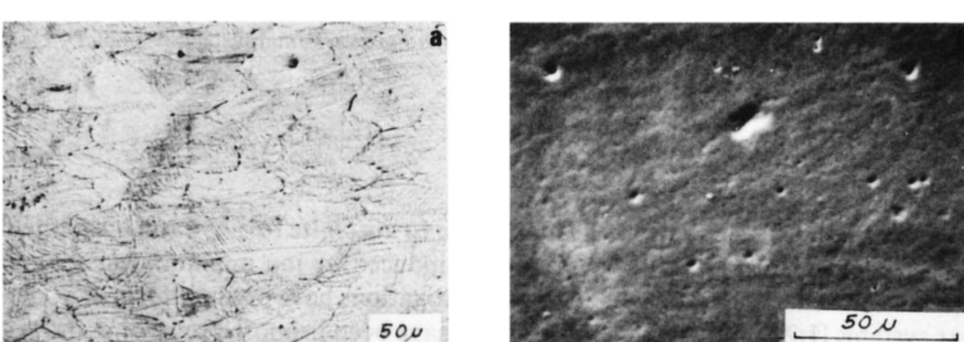

Fig. 1. (a) Martensitic 304 SS. (b) Implanted martensitic 304 SS.

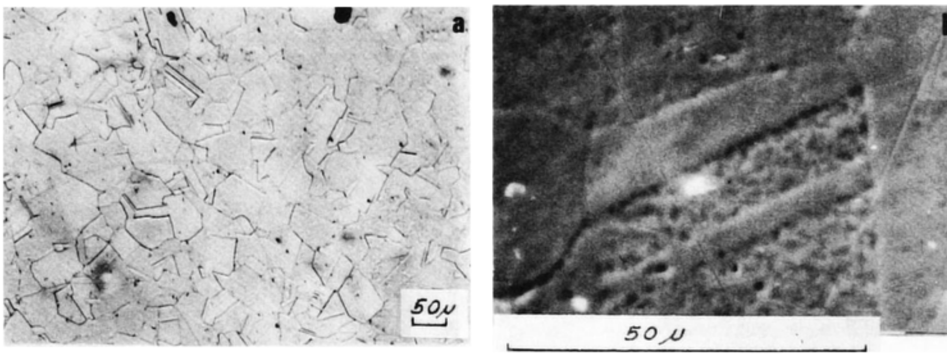

Fig. 2. (a) Austenitic 304 SS. (b) Implanted austenitic 304 SS.

ing the metallographic structure of unimplanted materials with the implanted ones; figs. 1b–5b display the SEM photographs of implanted samples. The CEMS outcomes, fig. 6 and table 1, indicate the phase modifications considering only the martensitic samples because in the other ones no relevant indications of phase transformations were observed.

4. Discussion

Hayashi et al. [3] have implanted He^+ and H^+ ions up to 10^{17} and 10^{19} atoms cm^{-2}, respectively, into a 304 SS. An austenitic (fcc) to martensitic (bct) ($\gamma \rightarrow \alpha'$) phase transformation was observed in a high proportion of the implanted volume.

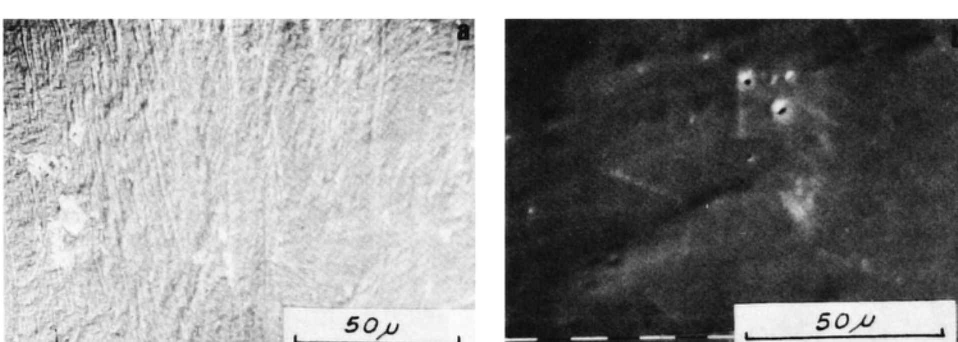

Fig. 3. (a) Martensitic 316 SS. (b) Implanted martensitic 316 SS.

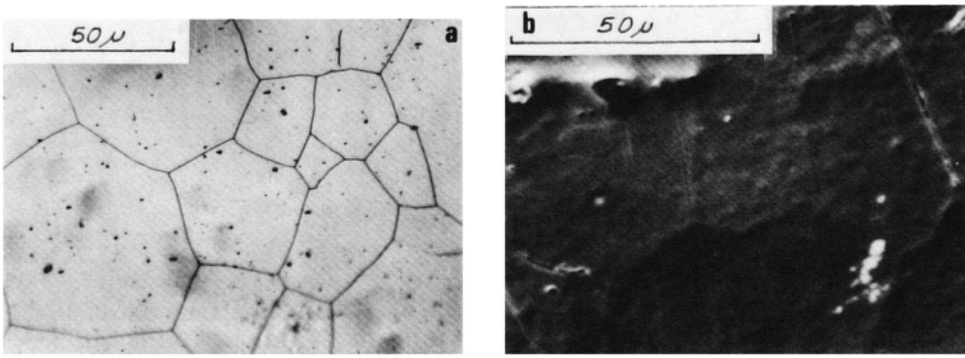

Fig. 4. (a) Austenitic 316 SS. (b) Implanted austenitic 316 SS.

A comparison of the optical microscopy photographs before implantation with the SEM photographs after implantation shows that implantation has affected via sputtering the surfaces of the materials in different grades according to the phase present before the implantation. In the present study only the martensitic samples suffered the $\gamma \rightarrow \alpha'$ transformation and within the sensitivity limits of our techniques this transformation was neither found in the initially austenitic stainless steels nor in α-Fe. Although, some transformation could be found beyond our CEMS range of 1000 Å, especially at the range of 2100 Å for 180 keV C into Fe as calculated by a simulation program. Folstaedt, Knap and Pope [5] studied the microstructure and composition of Ti and C implanted 304 SS. All surfaces implanted with He^+ studied by Fukahori et al. [6] displayed surface deformation. The authors concluded that ion implantation not only produced strain on the material but also induced a stress state inside it. The $\gamma \rightarrow \alpha'$ transformation could be explained con-

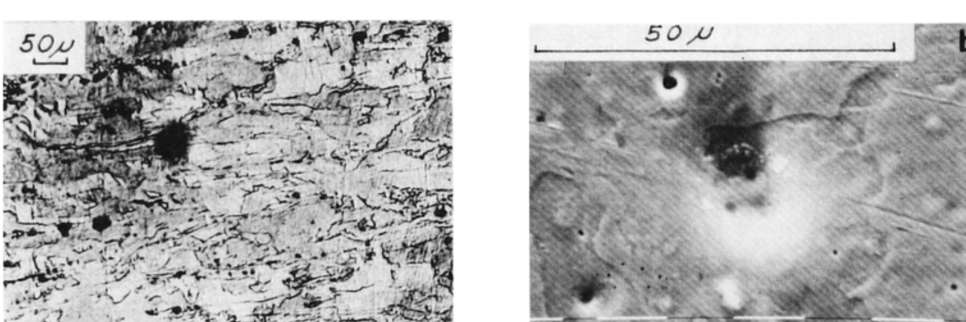

Fig. 5. (a) α-Fe. (b) Implanted α-Fe.

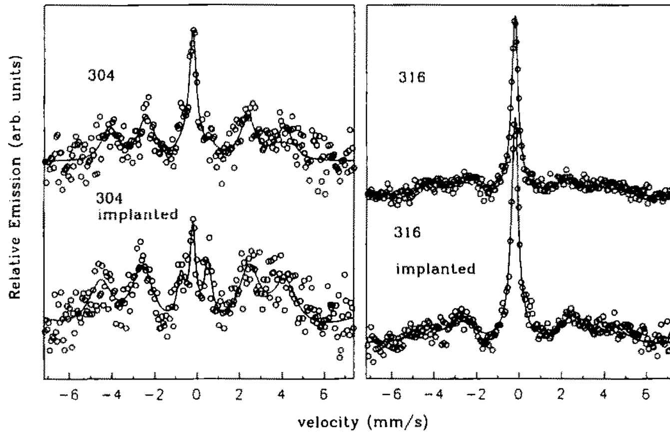

Fig. 6. CEMS spectra of martensitic samples.

sidering that the remanent austenite in the martensitic samples is stressed in a state of higher elastic energy than the austenite in the austenitic samples. In this way, the oncoming ions would increase the elastic energy so inciting the transformation. At the beginning, close to the surface, the interaction ions-matrix at these energies is not sensitive to the crystalline structure in which the ions are travelling. This would become important only in the region immediate to the penetration depth about 500 Å ahead. The ion implantation on austenitic AISI 304 and 316 would not induce austenite–martensite transformations because the increase of elastic energy provided by the incoming ions would not be enough to induce the transformation through a matrix with low elastic energy. However, in martensitic samples the ions passing throughout small austenitic crystals would increase the residual tensions and surpass the transformation threshold level. Kato and Pak [7] have observed that the increase of the principal component of the stress tensor produces a decrement of the temperature of martensification Ms. In our case, with an average dose of 5×10^{17} atoms/cm^2, we believe that the ion implantation also may have produced a reduced Ms.

Table 1
Percentage of austenite and martensite phases, determined by CEMS, before and after implantation into samples initially in mainly martensitic phase before implantation.

Sample	Martensite (%)	Austenite (%)
304 mart. unimpl.	76 ± 11	24 ± 4
304 mart. implant.	87 ± 11	13 ± 3
316 mart. unimpl.	62 ± 10	36 ± 4
316 mart. implant.	64 ± 11	36 ± 4

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