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Comparison of erosion processes of RAF and pure Fe by hydrogen and carbon mixed ion beam irradiation

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Abstract

Sputtering erosion and surface morphology of F82H and pure Fe irradiated by hydrogen and carbon mixed ion beam (1 keV H_3^+) were studied. It was found that carbon impurity (0.7–0.8%) in hydrogen ion beam caused significant reduction in the sputtering yield for Fe at elevated temperatures ($\geqslant$773 K). On the other hand, the increase in the sputtering yield at elevated temperature was observed for F82H. Surface roughening at 423 K for Fe was pronounced in the case of C: 0.1%, but not in C: 0.7%. The mechanisms for these carbon effects could be related to surface disordering and change of grain orientation by mixed ion bombardment.

1. Introduction

A reduced-activation ferritic/martensitic steel (RAF) is one of the strongest candidates for structural materials of blankets for DEMO reactors [1]. Its sputtering erosion, however, could be an important issue for the use of a RAF as a plasma-facing wall material [2]. Accurate estimation of erosion rates in actual fusion devices, however, is difficult due to the presence of impurities in edge plasmas, especially eroded plasma facing materials (PFMs).

The effects of carbon ions in a hydrogen ion beam on the erosion behavior of RAF (F82H (8Cr–2W)) [3] was studied by Ueda et al. [4]. They showed that the erosion yield at 453 K is lower than that at 773 K by 18% in the case of carbon concentration in the beam of about 0.8% (C: ~0.8%). The increase in the erosion yield with temperature is attributed to the diffusion of implanted carbon atoms into the bulk and a reduction of the carbon concentration at the surface, which protected F82H from erosion at 453 K.

To make a more basic study on the effects of carbon impurity, we have examined pure Fe in addition to F82H. Pure Fe has similar physical sputtering characteristics to RAF. Recrystallization behavior of F82H is quite different from that of pure Fe. Pure Fe shows recrystallization and significant grain growth above about 750 K, while no significant change in microstructure for F82H takes place at least up to 900 K. Grain orientations strongly affects sputtering erosion and surface roughness [5]. Therefore, recrystallized Fe or Fe at elevated temperatures (>750 K) could show different erosion characteristics from those of F82H. In this paper, we describe the temperature dependent erosion yield and surface morphology of pure Fe irradiated by

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hydrogen and carbon mixed ion beam in comparison with F82H.

2. Experimental

In this study, the ion beam irradiation device (HiFIT) was employed for hydrogen and carbon mixed ion beam irradiation. The details of this ion source were described in Refs. [6,7] and the details of hydrogen and carbon mixed ion beam irradiation experiments were shown in Ref. [4]. The ion beam used for the experiments is 1 keV H_3^+ (H_3^+ is the main component) with carbon concentrations of 0.1% and 0.7–0.8%. The ion flux to samples is about 4×10^{20} H/m^2 s.

F82H contains 7.65 wt% Cr, 2 wt% W, and Mo, Mn, V, Ta, Ti, Si and C below 1 wt% in sum total, and Fe for the balance [8]. Pure Fe samples were supplied by Nilaco Co. with the purity of 99.998%. The sample size was $10 \times 20 \times 1^t$ mm^3 with surfaces polished to a mirror finish, with a roughness of an order of 10 nm. The ion irradiation area was limited by a 5 mm diameter aperture in front of the samples. Samples were heated up to 873 K with an IR-heater and the sample temperature was measured using a thermocouple embedded in the copper holder, which was attached to the irradiation samples.

The erosion rate was estimated by weight loss during ion beam irradiation and the average erosion depth of the irradiation area. Weight loss was measured by a micro balance (Metler, MX5) with a readability of 1 μg. Erosion depth was measured by a surface profilometer (SNF, DekTak3) with a stylus radius of 12.5 μm. This profilometer can measure line profiles of erosion depth. We measured 13 lines of erosion depth profile across the ion beam irradiation area to obtain two-dimensional profiles. From these 2 D profiles and the density of F82H or Fe, sputtering yields averaged over irradiation area were calculated. The depth profile of atomic composition was measured by XPS (X-ray induced Photoelectron Spectroscopy, AXIS 165, Kratos Co.). Grain orientations for recrystallized samples were measured by EBSP (Electron Backscatter Diffraction Pattern, JEOL 680A).

3. Experimental results on erosion yield for F82H and Fe

Results of the sputtering yields calculated from weight loss and depth profile measurements for F82H and pure Fe are shown in Fig. 1. The ion irradiation fluence was $(4–5) \times 10^{24}$ H/m^{-2} with carbon concentrations of 0.7–0.8% (a, c) and 0.1% (b). For F82H in the case of C:0.7–0.8% (Fig. 1(a)), sputtering yields at elevated temperatures (773 K and 873 K) are higher by about 20%, which is attributed to carbon diffusion into the bulk and a reduction of carbon concentration at the surface. This explanation was supported by surface atomic composition measurements by XPS, which showed a surface carbon concentration of about 20% at 453 K decreased to about 3% at 773 K. For pure Fe, the result is completely different. In the case of C: 0.7–0.8% (Fig. 1(c)), the yields decrease very rapidly with temperature over 700 K. The yield at 873 K is roughly half or less in comparison with the yield at 423 K,

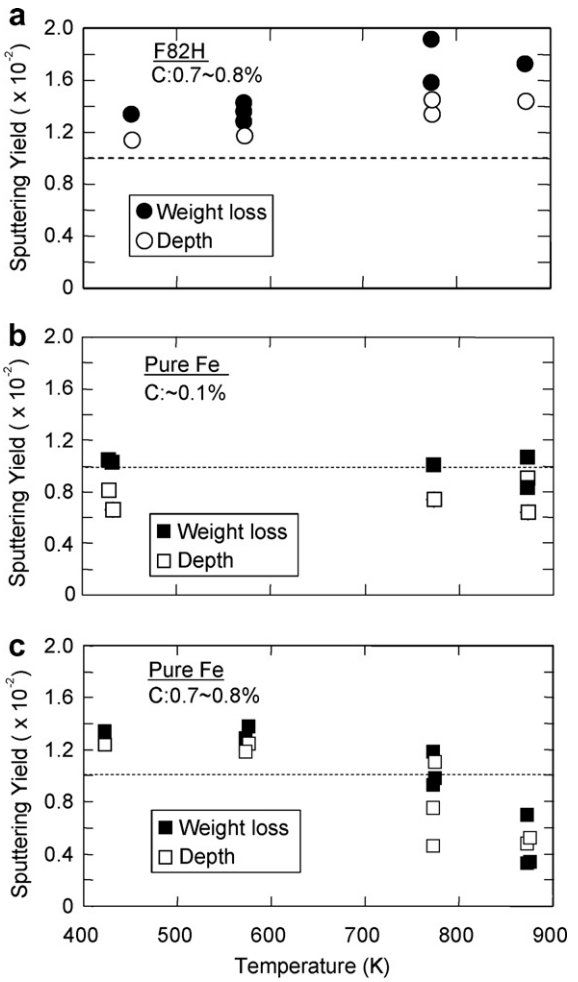

Fig. 1. Temperature dependence of sputtering yield estimated from weight loss (■) and depth profile (□) for F82H in C: 0.7–0.8% (a), pure Fe in C: ~0.1% (b), and pure Fe in C: 0.7–0.9% (c).

while the yields in the case of C: ~0.1% (Fig. 1(b)) is almost constant regardless of temperature. These results clearly indicate that the mechanism of the erosion reduction for Fe is not simply explained by the change of carbon surface concentration.

When the yield was calculated from weight loss, the effect of the carbon mass implanted by the ion beam was not considered. If all of the implanted carbon atoms remained in the samples (without resputtering), the estimated sputtering yield decreased by about 0.2. At elevated temperatures, when implanted carbon atoms diffused into the bulk and stayed there, the actual yields may be higher than those shown in Fig. 1. These increments, however, were only about 0.2 or less, which does not affect the conclusion shown above.

4. Surface morphology for ion irradiated F82H and pure Fe

Fig. 2 shows the surface morphology of F82H after irradiation with a hydrogen ion beam containing C: 0.7%. A typical lath structure (characteristic of ferritic/martensitic steel) and a grain size of about 50 μm were clearly observed both at 453 K (Fig. 2(a)) and 873 K (Fig. 2(b)). This indicates that the microstructure of F82H did not change at least up to 873 K. Surface roughness of the irradiated area slightly increased with temperature, but this change in roughness is much smaller than that of Fe as shown below.

The surface morphology for irradiated Fe samples in the case of C: 0.1% is shown in Fig. 3. Surface roughness at 423 K (Fig. 3(a)) is very prominent. Many small complicated plateaus with smooth surfaces developed at 473 K, while large grains with smooth surfaces were observed at 773 K (Fig. 3(b)). Recrystallization or grain growth does not occur at 423 K. Therefore, the surface structure stayed fine and had complicated shapes, which probably correspond to grain structure. On the other hand, at 773 K the grain sizes grew up to about 50 μm due to recrystallization and most of the grain surfaces are very smooth.

In both temperature cases, there are very large steps between adjacent grains. In other words, the erosion depth has large variations between grains. Fig. 4 shows line profiles of erosion depth of Fe across the ion irradiation area in the case of C: 0.1%. It is clearly shown that the depth variation is very large both at 423 K (Fig. 4(a)) and at 873 K (Fig. 4(b)). At 423 K, the smallest erosion

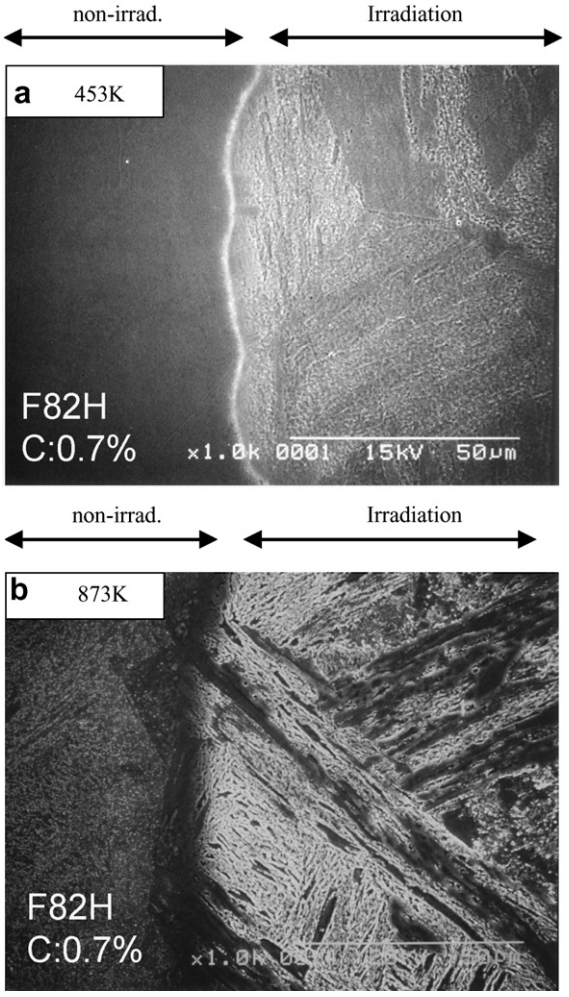

Fig. 2. Surface morphology for F82H after ion beam irradiation at 453 K (a) and 873 K (b) in the case of C: 0.7%.

depth is about 0.15 μm, while the largest is about 0.75 μm (i.e. the depth varies by a factor of about 5). On the other hand, the depth variations at 873 K reached a factor of about 9 with the minimum erosion of about 0.15 μm and the maximum erosion of about 1.3 μm. At 873 K, erosion within each grain is almost uniform, which suggests that the sputtering yield is dependent on grain characteristics. It is known that the sputtering yield depends on grain orientation [5]. Ref. [5] cited the result of single crystal Cu bombarded by Ar such that the variation of sputtering yields is a factor of 3 between Cu (1 1 1) and Cu (1 1 0). The erosion depth variation of Fe in our result is much larger than this case. The grain orientation with respect to the surface was measured by EBSP. It was found that each grain has an almost undisturbed crystalline structure with

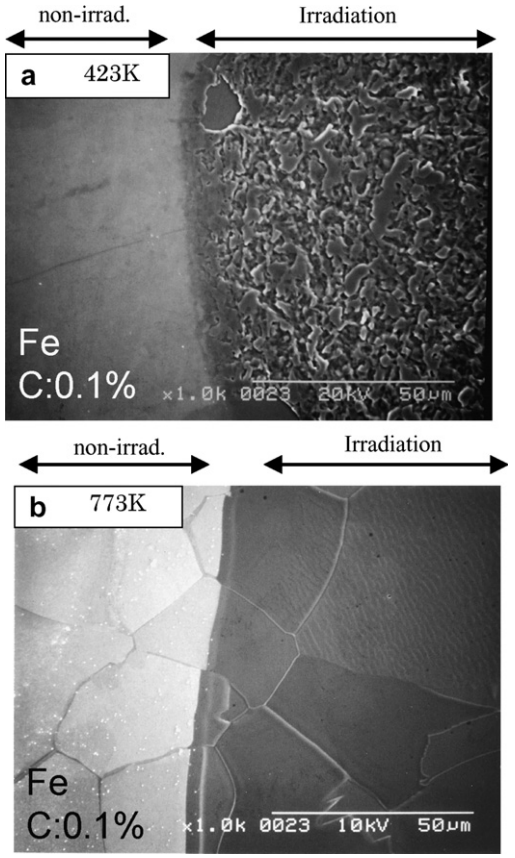

Fig. 3. Surface morphology for Fe after ion beam irradiation at 453 K (a) and 773 K (b) in the case of C: 0.1%.

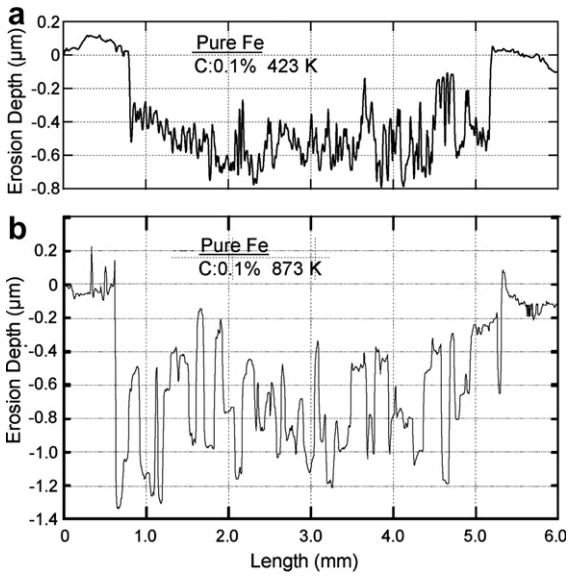

Fig. 4. Line profile of depth distribution across the ion irradiated area for Fe at 423 K (a) and 873 K (b) in the case of C: 0.1%.

reliable Kikuchi patterns, probably due to thermal annealing effects. However, the relation between erosion depth and index of each grain is not clear.

The surface morphology for pure Fe in the case of C: 0.7% is shown in Fig. 5. The entire irradiated surface seems to be flat at 423 K (Fig. 5(a)), which is quite different from the case of C: 0.1% (Fig. 3(a)). This result is also supported by surface profile measurements, see Fig. 6(a). On the other hand at 773 K, most of each grain surface is smooth but rough patterns appeared in some areas. Some of the rough areas protruded above the level of the unsputtered surface, see Fig. 6(b). These protrusions are probably impurity induced sputtered cones. Surface diffusion of adatoms nearby impurities causes these strange shapes. It was reported that a relatively high ion flux ($\sim 10^{20}/m^2\,s$) strongly enhanced the cone formation [9]. Since the hydrogen irradiation flux of our experiments was comparable

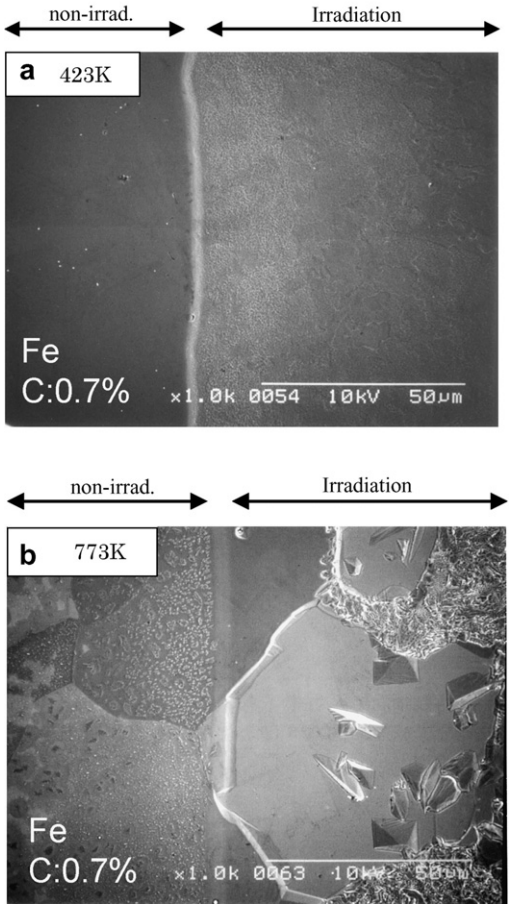

Fig. 5. Surface morphology for Fe after ion beam irradiation at 453 K (a) and 773 K (b) in the case of C: 0.7%.

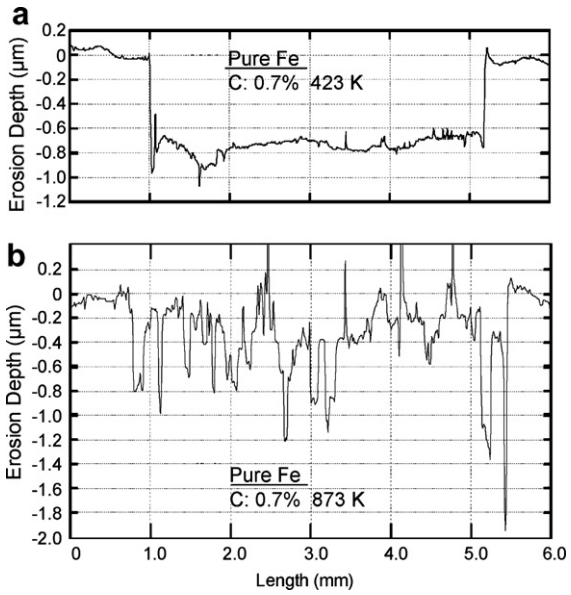

Fig. 6. Line profile of depth distribution across the ion irradiated area for Fe at 423 K (a) and 873 K (b) in the case of C: 0.7%.

to this flux, the cone formation is expected to occur on our samples.

The reason why the eroded surfaces of Fe at 423 K were relatively flat in the case of C: 0.7% could be the following. XPS measurements showed that the carbon concentration at the surface is about 20%, which was determined by the balance between influx and sputtered flux of carbon ions (no thermal diffusion at this temperature). The presence of carbon atoms at the surface (probably together with surface disorder caused by carbon ion bombardment) could suppress the effect of grain orientation on sputtering yield variation, leading to flat eroded surfaces.

The large erosion depth variations for Fe at 873 K were also observed in the case of C: 0.7%. The average erosion depth, however, was quite small compared with the case of C: 0.1%. It is noted that there have been no reports on this kind of impurity effect on sputtering. XPS measurement showed that some of the 1implanted carbon atoms diffused into the bulk and carbon concentration in the Fe was about 5% to the depth of 80 nm (the deeper layer was not measured). Although the reason for the reduction of the sputtering yield is not known yet, one interesting phenomena was found from the measurement of grain orientation by EBSP. Fig. 7 shows the irradiated surface of Fe at 873 K in the case of C: 0.7%. The area surrounded

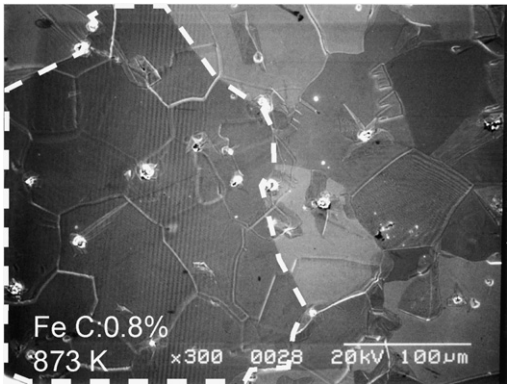

Fig. 7. A photo of irradiated surface of Fe at 873 K in the case of C: 0.8%. Inside the white broken line the grain orientation is the same.

by the white broken lines has exactly the same index, though the area contains many separate grains. These kinds of area were not observed for the case of C: 0.1%. Since very few carbon (less than 1%) remained near the surface [4], it is unlikely that the carbon inclusion in pure Fe changed crystal structure or grain orientation. Therefore, the carbon impurity injection together with hydrogen ion beam could have some effects to alter grain orientation of Fe at elevated temperatures. It is noted that since the EBSP technique only measures grain orientations near the surface to a depth of 10–20 nm, we do not know whether the grains beneath this thin surface area had the same orientation or not.

5. Conclusion

A small amount of carbon impurity in the hydrogen ion beam (1 keV H_3^+) has a considerable effect on sputtering erosion and associated surface morphology change for F82H and pure Fe. Carbon impurity (0.7–0.8%) in the hydrogen ion beam caused a significant reduction of the sputtering yield for Fe at elevated temperatures ($\geqslant$773 K), while an increase in the sputtering yield at elevated temperature was observed for F82H. Surface roughening at 423 K for Fe was pronounced in the case of C: 0.1%, but not in C: 0.7%. It was also found that carbon impurity in the beam altered some of the grain orientation near the surface (depth to about 10–20 nm) for Fe at elevated temperatures. The mechanisms for the carbon impurity effects found in this study could relate to either surface lattice disordering or change of grain orientation by carbon bombardment.

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