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Effect of ion-beam irradiation on power reflectivity of boron-doped CFC materials

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Abstract

Effect of ion-beam irradiation on reflectivity characteristics for the 1D CFC (MFC-1) and newly developed boron-doped CFCs (Tonen 1D CFC) has been investigated in the synchrotron radiation range from 119 to 791 μm. The B_4C-doped CFC showed higher reflectivity characteristics, such as $R > 70\%$ at 119 μm and weaker fluence-dependencies on the reflectivity at the fluences up to 8.5×10^{24} m^{-2}, compared with MFC-1. To understand the mechanism of reflectivity degradation due to ion beam irradiation, we have examined the surface property, such as roughness and electrical conductivity. Result of electrical conductivity measurement revealed that conductivity decreased as fluences similarly to that of reflectivity, which suggested that reflectivity drop was mainly caused by the change of electrical conductivity. Lastly, we presented the preliminary result of the beryllium coating on the irradiated surface to improve reflectivity characteristics, which showed that reflectivity of the beryllium-coated CFC was raised from 60% to more than 80% at 119 μm.

Keywords: Tokamak; Energy balance; Chemical erosion; Wall coating; Low Z wall material

1. Introduction

Effect of wall reflection of the synchrotron radiation emitted from a magnetically confined plasma will be important in analyzing the power balance of fusion reactor plasmas [1]. In general, synchrotron radiation will be dominant in the wavelength range from far-infrared to millimeter wave in the future magnetically confined plasmas with magnetic fields of ~ 10 T. Up to now, the wavelength- and angular-dependence [2] and temperature dependence [3,4] of reflectivity have been investigated for the candidate materials such as isotropic graphite, one- and two-dimensional carbon fiber reinforced composites (CFC), silicon carbide (SiC), and metallic materials (W, Mo, SUS, etc.). Under the practical condition, the plasma facing material will be exposed to the edge plasma and the high heat flux from hot core plasma. Thus, we have investigated the effect of electron beam irradiation of the CFC materials simulating the major disruption or high energetic electron burst, and also the effect of the low energy ion-beam irradiation simulating the edge plasma interaction [5].

Recently, boron-doped (B_4C-doped) 1D CFCs have been developed by the Japan Atomic Energy Research Institute (JAERI) group as the promising first wall material [6], where the sputtering characteristics have been investigated using the ultra-low energy hydrogen ion beam [7]. In this paper, our concern is focused on the reflectivity characteristics of the boron-doped CFC and their ion beam fluence dependence. First, we have carried out the reflectivity measurement of boron-doped CFCs and compared them with those of non-doped CFC itself and B_4C-coated isotropic graphite used in the previous measurement [2]. The effect of ion-beam irradiation on the reflectivity of boron-doped CFC will be presented and briefly discussed

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Table 1
Ion beam irradiation conditions for 1D MFC-1 samples

Sample	Flux ($10^{20}/m^2s$)	Duration (min)	Fluence ($10^{24}/m^2$)	Weight loss (mg)
No. 1	5.3	50	1.6	0.60
No. 2	6.0	100	3.7	1.46
No. 3	4.7	200	5.6	2.59
No. 4	5.1	400	12.0	4.83

about a mechanism of reflectivity degradation. Lastly, we will present the preliminary results of the beryllium coating of the ion-beam irradiated 1D CFC for aiming at the in-situ recovery of the deteriorated reflectivity.

2. Experimental arrangements

An optically pumped far-infrared laser with wavelengths from 119 to 791 μm [2,3] was employed to study the reflectivity characteristics in synchrotron radiation range, which roughly corresponds to the 2nd to 10th harmonics of electron cyclotron frequency for the plasma with a magnetic field of 8–10 T. In the ion beam irradiation experiment, two types of 1D CFCs (one is manufactured by the Mitsubishi Chemical, referred as 'MFC-1' and the other is by the Tonen, simply referred as 'Tonen'), and 5 and 10 wt% boron-doped 1D CFC (Tonen), were tested for the ion beam irradiation experiment. Dimension of MFC-1 and Tonen CFC samples was 25 mm × 25 mm, 3 mm in thickness. Each sample was polished at the maximum roughness less than about 5 μm. The ion beam irradiation experiment was carried out using a low energy hydrogen ion beam source at the Naka Fusion Research Establishment of the JAERI [7]. A hydrogen ion beam having an energy of 50 eV and particle flux of $\sim 3 \times 10^{20}$ m^{-2} s^{-1} was irradiated onto the sample surfaces. The ion beam fluences were changed to 1.6, 3.7, 5.6 and 12.0×10^{24} m^{-2} for MFC-1, and 2.7–2.8 and $7.5–8.5 \times 10^{24}$ m^{-2} for boron-doped Tonen CFCs, respectively. For instance, particle fluence of 12.0×10^{24} m^{-2} is equivalent to the plasma discharge of roughly 30 s in the ITER class plasmas. In Tables 1 and 2, ion-beam irradiation conditions for MFC-1 and B_4C doped Tonen CFCs are tabulated, respectively. Since the dependence of incident angle on the reflectivity was previously measured [2], so that we carried out reflectivity measurement at a fixed incident angle, say, $\theta \sim 25°$.

Table 2
Ion beam irradiation conditions for boron-doped Tonen 1D CFC samples

Sample	Flux ($10^{20}/m^2s$)	Fluence ($10^{24}/m^2$)	Weight loss (mg)
Non-doped	1.9–2.2	7.0	2.39
5 wt% B_4C-doped	2.5–2.8	2.8	1.18
	2.5	8.5	2.63
10 wt% B_4C-doped	2.4–2.6	2.7	1.91
	2.3–2.5	7.5	2.94

3. Experimental results and discussion

3.1. Wavelength dependence of reflectivity

First, we measured reflectivity characteristics of the boron-doped Tonen CFCs and B_4C-coated graphite (manufactured by Hitachi Chemical, B_4C/PD-330S) in the wavelength range from 119 to 790 μm. Experimental results of reflectivity, $R_{\parallel}$ and $R_{\perp}$, of 10 wt% boron-doped Tonen CFC are shown in Fig. 1, together with reflectivities of B_4C-coated graphite, where a thickness of B_4C coated over the isotropic graphite (PD-330S) was on the order of a few 100 μm. A root mean square roughness of sample surface, δ_{rms}, was measured using a thickness meter with the stylus method and found that it was less than roughly 1 μm. According to the theoretical analysis given in Ref. [8], we can find that the effect of surface roughness on the reflectivity is negligibly small, since reflectivity drops from the ideal value only by a factor of $1 - \exp\{-(4\pi\delta_{rms}/\lambda)^2 \cos^2\theta\} \sim 0.01$ at $\lambda = 119$ μm. It is seen in Fig. 1 that boron-doped CFC has much higher reflectivities than B_4C-coated graphite; in actual, the boron-doped CFC has $R_{\perp} = 95\%$ and $R_{\parallel} = 88\%$ at $\lambda = 791$ μm and $R_{\perp} = 86\%$ and $R_{\parallel} = 72\%$ at $\lambda = 119$ μm. On the other hand, reflectivity of the B_4C-coated graphite significantly dropped from $R_{\perp} = 58\%$ and $R_{\parallel} = 38\%$ at $\lambda = 791$ μm down to $R_{\perp} = 18\%$ and $R_{\parallel} = 6\%$ at $\lambda = 119$ μm. In the previous measurement, we found that B_4C/PD-330S had a low effective electrical conductivity on the order of $\sigma_{eff} \sim 300$ Ω^{-1} m^{-1}, where σ_{eff} was

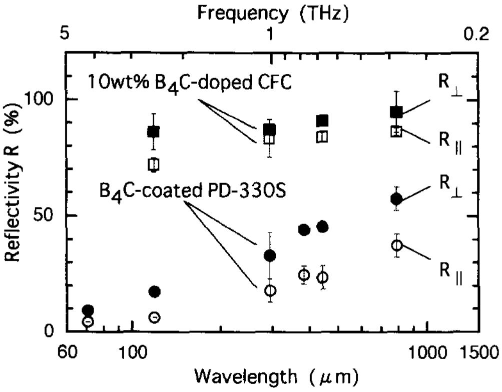

Fig. 1. Reflectivity characteristics of the 10 wt% B_4C-doped Tonen CFCs and B_4C-coated graphite (B_4C/PD-330S) in the wavelength range from 119 to 790 μm.

theoretically evaluated from the experimental results of reflectivity [2]. From an aspect of synchrotron radiation loss, boron-doped CFC is much better than B_4C-coated graphite, because a net power loss of synchrotron radiation proportional to $(1 - R)^{1/2}$ will be reduced at higher reflectivity.

3.2. *Fluence dependence of reflectivity*

As described in the previous section, hydrogen ion beam was irradiated onto MFC-1, Tonen CFC, and 5 wt% and 10 wt% B_4C-doped Tonen CFCs at different fluences as listed in Tables 1 and 2. First, we plotted the fluence dependence of reflectivity $R_\perp$ and $R_\parallel$ of the MFC-1 in Fig. 2(a) and (b). Varying the ion beam fluences from zero to 12.0×10^{24} m^{-2}, $R_\perp$ and $R_\parallel$ significantly dropped at wavelengths of 119, 443 and 791 μm. For instance, at $\lambda = 119$ μm, reflectivity changed from $R_\perp \sim R_\parallel = 80$–85% to $R_\perp = 56\%$ and $R_\parallel = 50\%$, respectively. As seen in Fig. 2, it seems that the reflectivity drop will saturate at ion fluences higher than 12.0×10^{24} m^{-2}. Unfortunately, we could not observe the ion fluence level where the saturation of reflectivity degradation occurred in the present experiments.

Surface morphology of the irradiated surface was investigated by the JAERI group using the SEM [7]. At the particle fluence of 12.0×10^{24} m^{-2}, the micrograph of the surface looked more rough than that of the unirradiated surface. However, measurement of surface roughness using the thickness-meter showed the maximum surface roughness of < 5 μm, which was still less than wavelengths used for the measurement. To account for the reflectivity deterioration, we have carried out the measurement of dc electrical conductivity using the four-point method [9]. The results are presented in the next section.

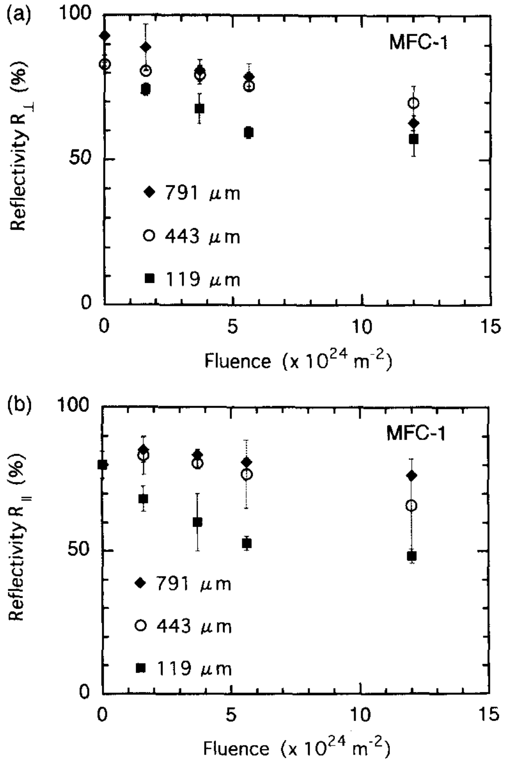

Fig. 2. Fluence dependencies of (a) $R_\perp$ and (b) $R_\parallel$ of the MFC-1 measured at $\lambda = 119$, 443 and 791 μm.

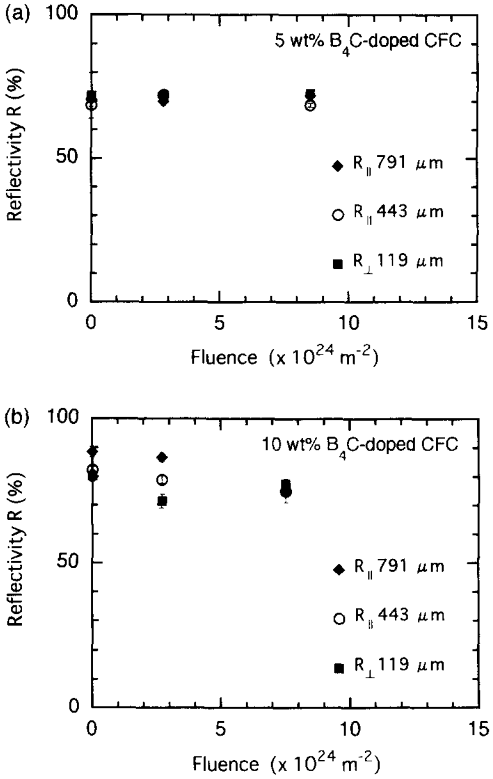

Fig. 3. Fluence dependencies of reflectivity $R_\perp$ and $R_\parallel$ at $\lambda = 119$, 443 and 791 μm of (a) 5 wt% B_4C-doped CFC and (b) 10 wt% B_4C-doped CFC.

Next, we plotted the reflectivities $R_\perp$ and $R_\parallel$ for B_4C-doped CFC measured at $\lambda = 119$, 443 and 791 μm in Fig. 3. It is noted that, at $\lambda = 119$ μm, a high reflectivity of $R > 70\%$ is observed for the samples irradiated at fluences of 7.5–8.5×10^{24} m^{-2}, which is apparently different from that of MFC-1 shown in Fig. 2. Furthermore, it is also noted that reflectivities of 5 wt% boron-doped CFC are almost independent of the fluences up to 7.5–8.5×10^{24} m^{-2}. Reflectivity characteristics of Tonen CFC itself are not shown here, but they are very similar to that of 10 wt% boron-doped CFC.

3.3. *Electrical conductivity of irradiated surface*

Here, we will briefly discuss a mechanism of reflectivity degradation of MFC-1 due to ion beam irradiation. One

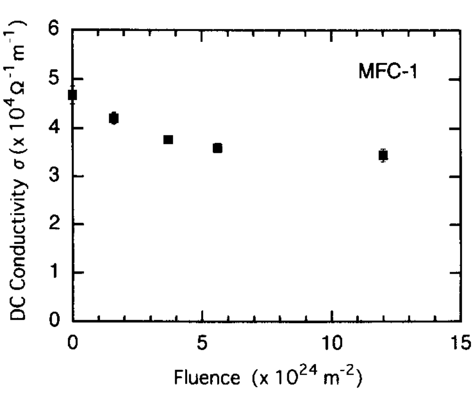

Fig. 4. Experimental results of dc electrical conductivity measurement of the irradiated MFC-1 sample using the four point method.

of possible explanations is a change of surface characteristics, such as surface roughness or electrical conductivity. As for the surface morphology of graphite and CFC, we measured surface roughness using the thickness meter with stylus method and using the SEM, as described before. The results showed no significant difference between unirradiated and irradiated surface. Thus, we considered that the reflectivity degradation of CFCs might be mainly caused not by the change of surface roughness but that of electrical conductivity due to the ion beam irradiation. Reflectivities of conductive material are approximately given by Ref. [10], $R_{\parallel} \approx 1 - 2\xi/\cos\theta$, and $R_{\perp} \approx 1 - 2\xi\cos\theta$ for $\xi \ll 1$, where $\xi = ((2\varepsilon_0\omega)/(\sigma_{\mathrm{eff}}))^{1/2}$, θ is an incident angle, ω is the angular frequency of incident wave and σ_{eff} is the effective electrical conductivity, defined by $\sigma_{\mathrm{eff}} = \sigma + \omega\varepsilon_{\mathrm{i}}$ in terms of dc conductivity σ and an imaginary pan of the dielectric constant, $\varepsilon = \varepsilon_{\mathrm{r}} + j\varepsilon_{\mathrm{i}}$. Therefore, both the reflectivities $R_{\perp}$ and $R_{\parallel}$, decrease as σ decreases. Although we could not directly observe the change of effective conductivity, we can deduce that the reflectivity deterioration might be caused by a change in surface electrical conductivity through the production of volatile hydrocarbon composite or the soot of carbon cluster. As described in the latest section, we have carried out the measurement of the dc electrical conductivity of the irradiated surface using the four point method. Experimental results show that the dc electrical conductivity of CFC decreases by 27% at a ion beam fluence of 12.0×10^{24} m^{-2}, as shown in Fig. 4. Measured conductivity is not equal to that of the irradiated surface layer, because it is the total conductivity including the irradiated surface layer and a highly conductive CFC bulk layer of $\sigma \sim 4.7 \times 10^4$ Ω^{-1} m^{-1}, so that we can expect that the actual conductivity of the surface layer itself will be definitely lower than the observed conductivity and that modification of the physical characteristics of the surface will be mainly associated with the reflectivity degradation. Up to now, it is not clear why the electrical conductivity has been changed owing to the ion-beam irradiation. With the ion beam irradiation, it was apparently seen that the surface morphology changed from a metallic lustrous surface to a blackened unlustrous one. Thus, we may consider that one of the reasons for the conductivity change is a formation of amorphous hydrocarbons or carbon clusters having a low conductivity which cause the drop of conductivity. We are now preparing to analyze the surface using the mass spectrometer or XPS in order to study the composition of the surface material. These experimental results will be presented in the near future.

In the present research, we carried out the reflectivity measurement at room temperature. As for the effect of surface temperature, we have already reported the experimental results of temperature dependence of reflectivity for graphite, CFC and metallic materials, where the temperature of the samples was varied from 300 to about 900 K [3,4]. Experimental results showed that the reflectivity was almost independent of temperature up to 900 K. Thus, we consider that the effect of the surface temperature on the reflectivity will be negligible in the present case.

3.4. Relation of synchrotron radiation power and reflectivity change

Lastly, we briefly discuss the synchrotron radiation power loss due to change of wall reflectivity. As in Ref. [1], a net of synchrotron radiation power loss taken into account of multiple-wall reflection, is given by

$$P_{\mathrm{syn}} = 4.14 \times 10^{-7} n_{\mathrm{e}}^{1/2} T_{\mathrm{e}}^{5/2} B^{5/2} a^{-1/2} (1-R)^{1/2} \times \left\{1 + 18/\left(A T_{\mathrm{e}}^{5/2}\right)\right\}^{1/2} V_{\mathrm{p}}\ [\mathrm{MW}] \qquad (1)$$

where n_{e} is the electron density in unit of 10^{20} m^{-3}, T_{e} the electron temperature (keV), a the minor radius (m), A the aspect ratio and V_{p} the plasma volume (m^3), respectively. In the ITER, for instance, it can be evaluated as roughly 65 MW, assuming the maximum values of plasma parameters and a wall reflectivity of 80%. If the reflectivity was varied from 80 to 50% as a result of the plasma–surface interaction, a net of synchrotron radiation power

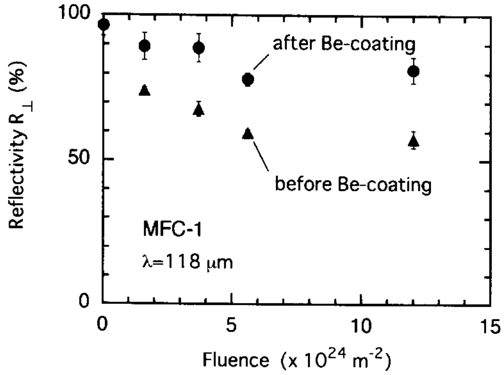

Fig. 5. Comparison of reflectivities of MFC-1 measured at $\lambda = 119$ μm before and after the beryllium-coating.

loss would increase from 65 MW to roughly 104 MW in the ITER-class plasma. Therefore, an improvement of reflectivity will be one of the important issues for the future fusion program. We have already proposed Be-coating as in-situ reflectivity recovery technique [11], preliminary results for ion-beam irradiated MFC-1 samples show a significant improvement of reflectivity from 60% to more than 80%, as shown in Fig. 5 at $\lambda = 119$ μm. Here, a thickness of beryllium coating was on the order of a few μm. A future issue of in-situ coating is to make a thick coating of ~ 100 μm with beryllium or tungsten.

4. Conclusions

In concluding the present paper, we presented the experimental results of the effect of ion-beam irradiation on reflectivity characteristics for the 1D CFC (MFC-1) and newly developed boron-doped CFCs (Tonen 1D CFC) in the synchrotron radiation range from 119 to 791 μm. The main results are summarizes as follows:

(1) Comparing the wavelength dependencies of 10 wt% B_4C-doped CFC and B_4C-coated graphite, we found that the B_4C-doped CFC had much higher reflectivity of $R >$ 70% in the whole wavelength range of 119 to 791 μm.

(2) The reflectivity of B_4C-doped 1D CFCs was insensitive to the ion beam fluence; in particular, the reflectivity of 5 wt% B_4C-doped CFC was almost independent of the fluences from zero to $7–8 \times 10^{24}$ m^{-2}. This result suggests that boron-doped CFC will be promising as the first wall material.

(3) To understand the mechanism of reflectivity degradation due to ion beam irradiation, we investigated the surface roughness and electrical conductivity. Experimental results suggested that the conductivity change was mainly associated with the deterioration of reflectivity.

(4) Lastly, we examined the usefulness of beryllium coating on the irradiated surface to improve reflectivity characteristics. Preliminary results showed that reflectivity of the beryllium-coated MFC sample was raised from 60 to $R > 80\%$, even at 119 μm.

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