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Application of optical techniques for in situ analysis of plasma facing carbon tiles

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ABSTRACT

Optical absorption/emission spectroscopy is considered to be used for in situ characterization of plasma facing carbon tiles and quantitative evaluation of tritium. In this paper we have applied ex situ laser Raman spectroscopy for carbon tiles used as first wall and divertor in JT-60 exposed to HH discharge plasma to verify the applicability of the technique. The analysis shows that the micro-structure of the carbon tile surfaces is modified in a similar way like damaging by energetic ion irradiation and that one can get information of how graphite structures are damaged by plasma exposure or what kind of structures the redeposited carbon obtains.

1. Introduction

Carbon-based materials (CBMs) are favourable for fusion applications because of a low atomic number, good thermo-mechanical properties, low coefficient of thermal expansion and the absence of melting. Fine grain graphite and carbon fibre composites (CFCs) are used in numerous existing fusion devices, and the latter is a candidate material for the vertical target tiles of the ITER divertor, where heat and particle fluxes are the highest [1]. Optical absorption/emission spectroscopy is a possible candidate method for in situ characterization of plasma facing carbon tiles and redeposited carbon materials, and quantitative evaluation of tritium. Raman spectroscopy is sensitive to detect the changes of graphitic structure and has been successfully applied to observe damaging process of CBM by irradiation of energetic particles (electrons, ions and neutrons), showing clear correlation with the Raman spectra and micro-structure observed by TEM [2–7]. In order to apply laser Raman spectroscopy as an in situ analysis technique for plasma facing carbon tiles and redeposited carbon layers in tokamak, we have to know how the Raman spectra of carbon materials are modified by plasma exposure and what kind of information they give on micro-structure change. In this work we have tried to get such information from graphite tiles exposed to JT-60 plasmas as first wall and divertor using ex situ laser Raman spectroscopy.

2. Experimental methods

2.1. Specimens

An isotropic fine grain graphite (Ibiden, ETP-10) blocks were machined as first wall and divertor tiles of JT-60 and exposed to HH (Hydrogen plasma with Hydrogen NBI) discharges in June 1988–October 1988 experimental campaign. The tiles were exposed to 1831 discharges (4 s plasma exposure for each discharge, 25% were disruption) with the maximum value for pasma current (I_p) 3.2 MA, and absorbed heat power (P_{abs}) 25.9 MW [8]. Operational temperature for the vacuum vessel was 573 K. The temperatures of the graphite tiles on the divertor and first wall were a little higher because of the plasma heat load [8].

Fig. 1 shows the schematic view of a poloidal cross-section with lower X point divertor geometry of the JT-60 vacuum vessel with indication of the locations of analyzed tiles [9]. Fig. 2 shows the detailed locations and the photographs of the divertor tiles in (a) and (b), and for the first wall in (c) and (d), respectively. Most of the divertor tiles were redeposited except near the divertor legs [8] (see Fig. 2(b)) and the first wall tile surfaces had mixed appearance of erosion (white and shinny area) and deposition (darker area) as seen in Fig. 2(d).

2.2. Raman spectroscopic analysis of the specimens

The Raman spectra were obtained using a 514.5 nm line of an argon ion laser with a back scattering geometry by a micro laser Raman spectroscopy (NRS-2100), which has a zero dispersive triple monochromator with a high sensitivity CCD alloy detector, and has a spatial resolution of 2 μm in minimum. The laser power was

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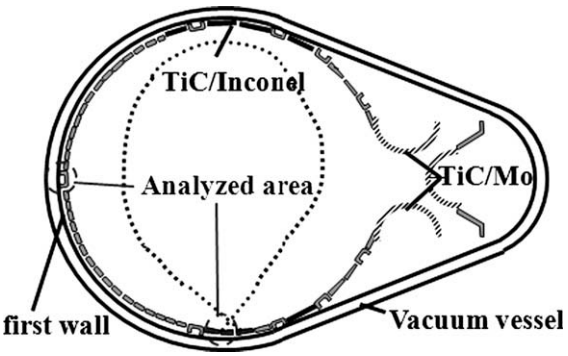

Fig. 1. The schematic view of the poloidal cross-section with lower X point divertor geometry of the JT-60 vacuum vessel (VV) in June 1988–October 1988 experimental campaign with indication for the analyzed tile's locations. Almost 80% of the inner surface of the VV was covered with isotropic graphite tiles, with the rest, 20%, armored mostly with TiC-coated Inconel liners [9].

controlled to be below 10 mW in order to avoid any laser heating effects. The analysis depth in the graphite tiles is considered to be in a range of 40–50 nm, judging from the optical skin depth of a laser with similar wavelength, 514.5 nm (2.42 eV) [10,11].

For comparison with previous Raman studies of carbon materials, the obtained Raman spectra were deconvoluted into two Lorentzian peaks at around 1355 cm^{-1} (so-called Disordered-peak, D-peak) and 1580 cm^{-1} (so-called Graphite-peak, G-peak), which were observed in Raman spectra for hydrogen and helium ion irradiated graphite [3–6,12,13], and the peak intensities (I_{1355} and I_{1580}) and full width at the half maximum for the D-peak ($FWHM_{1355}$) were determined.

3. Results and discussion

Fig. 3 shows typical Raman spectra for the first wall and divetor tiles. The spectra composed of two broad peaks centered at around 1580 cm^{-1} (G-peak) and 1355 cm^{-1} (D-peak) are very similar to those for hydrogen and helium ions irradiated graphite of which the peak structures change systematically with damaging by the ion irradiations [3–6,12,13].

Fig. 4 shows plots for the full width at the half maximum (FWHM) of the D-peak, $FWHM_{1355}$ versus the peak intensity ratio of the D-peak and G-peak, I_{1355}/I_{1580} for the eroded and redeposited areas of the divertor and first wall. The data for the first wall are from No. 7 and No. 10 tiles with the location numbers in Fig. 2(c). For example, the plot 7-1 in Fig. 4 means the data for location 1, No. 7 tile in Fig. 2(c). Although the data points in Fig. 4 did not seem to vary systematically in the toroidal or poloidal directions, they show clear tendency on how the graphite structures were modified. In addition, the data for the eroded area and the deposited area of the first wall tiles are clearly separated. The data for the highly eroded area corresponding to white colored area on No. 10 tile in Fig. 2(c) are located at lower left area in Fig. 4, while those for the deposited tile (No. 7), upper right. The data for the divertor tiles show a little different tendency, i.e. those for the deposited area appeared in the lower region, while those for eroded ones, i.e. divertor legs, are in the upper area indicating heavier damage. This is probably owing to higher flux irradiation at the divertor area, as discussed later.

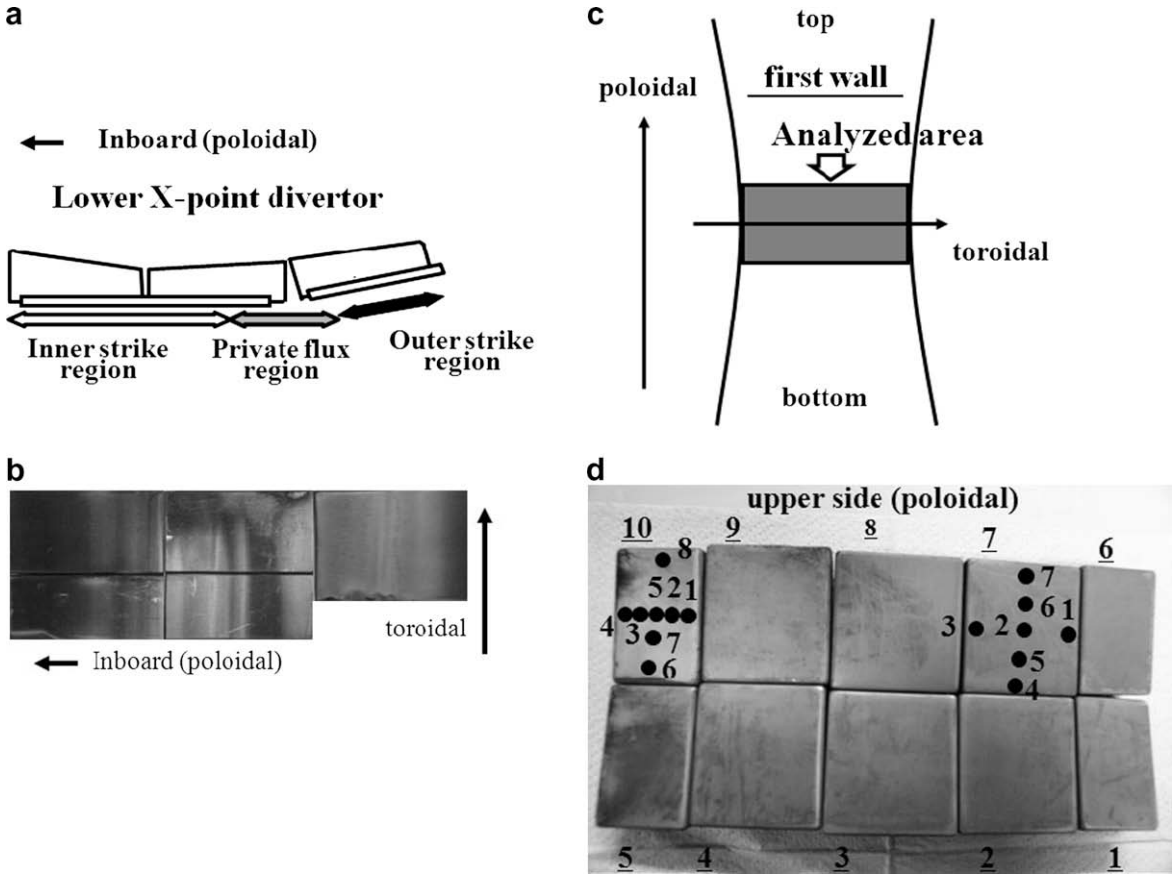

Fig. 2. Detailed views and the photographs of the divertor tiles in (a) and (b), and for the first wall in (c) and (d), respectively. In both photographs of (b) and (d), white shiny areas are highly eroded zones and dark areas are redeposition zones. Numbers in (d) are indicating analyzing points of tiles Nos. 7 and 10.

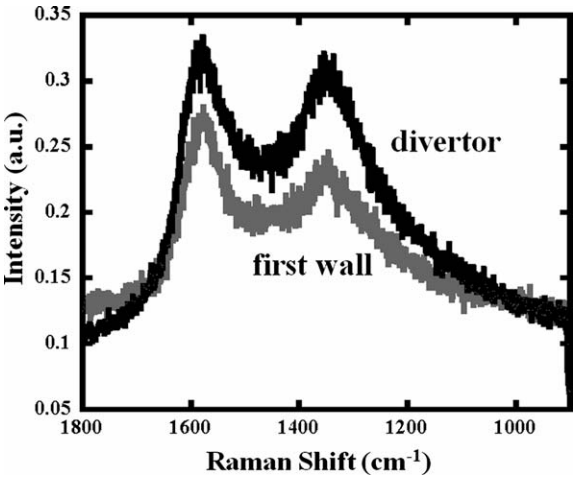

Fig. 3. Typical Raman spectra of the JT-60 first wall and divertor graphite tiles. Both spectra showed two broadened peaks, G-peak (centered at around 1580 cm^{-1}) and D-peak (centered at around 1355 cm^{-1}), which are always seen ion irradiated graphite [3–6].

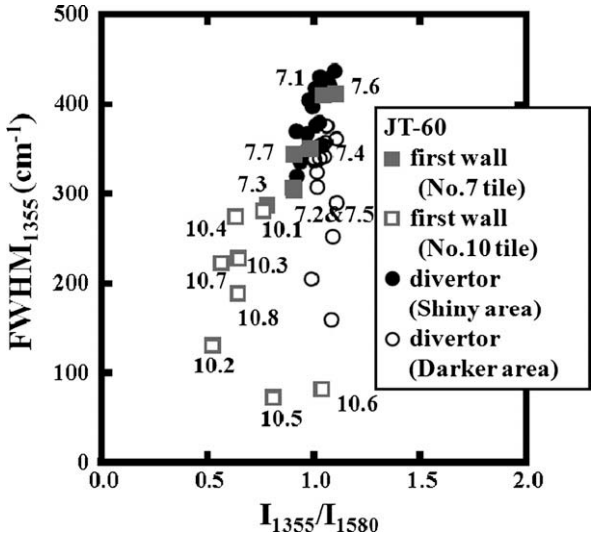

Fig. 4. Plots of the peak intensity ratio I_{1355}/I_{1580} vs. full width at the half maximum (FWHM) of the 1355 cm^{-1} peak for the JT-60 first wall and divertor graphite tiles.

Previous Raman studies have shown that the change of the Rama peak structures given by energetic ion irradiation corresponds well to the damaging process of micro-structures observed by TEM [4,12,14]. In particular, the peak intensity ratio I_{1355}/I_{1580} is well correlated to the crystallite size of damaged graphite with a equation $La = 41.9\ (I_{1355}/I_{1580})^{-1}$ (Å) [15,16]. Moreover, in the data plots of $FWHM_{1355}$ vs. I_{1355}/I_{1580} (similar to Fig. 4), variation of the data points to horizontally right direction corresponds to accumulation of defects within a graphite plane (graphene) or decrease of the width of the graphene (referred as two dimensional disordering), while that to vertically upper direction accumulation of three dimensional damage or destruction of layered structure (refereed as three dimensional disordering) [15,16].

In Fig. 5, all the present data for the JT-60 tiles superimposed on those obtained by the helium ion irradiation [12]. One can note that the data points for the first wall tiles just follow the damaging process observed in the ion irradiated graphite [3–6], i.e. subjected to the similar micro-structure change, with more disordering for

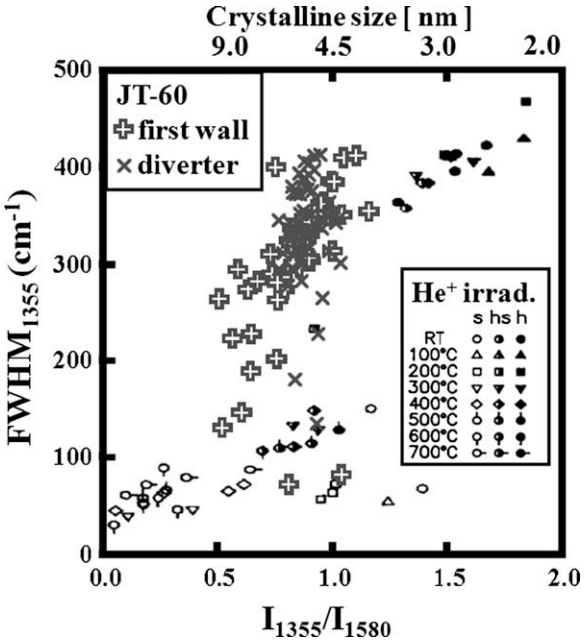

Fig. 5. Comparison of the Raman data for the JT-60 first walls and divetor tiles with those for irradiated highly oriented pyrolytic graphite (HOPG) foils by 25 keV He^+ ions at various temperatures [12]. The conditions of the diffraction pattern of the foils were investigated by TEM [14] and are indicated by the marks. I_{1355}/I_{1580} is correlated to crystallite size with a equation, $La = 41.9\ (I_{1355}/I_{1580})^{-1}$ (Å) [15,16].

the redeposited area. The data for the divertor area are somewhat different. As previously mentioned, the data for the redeposited area are located separately. This can be attributed to the in situ annealing effect of the redeposited layers on the divertor under high flux irradiation, which has been demonstrated in the previous work in which graphite tiles were irradiated with very high fluxes and at high temperatures resulting in very fine grains but keeping good crystallinity and their data points located at more lower right [13]. Actually the TEM observation of the deposition layer on the JT-60 divertor tiles showed layered structures with very small width [9].

Because the graphite tiles used in the JT-60 did not contain large amount of hydrogen (H concentration, around 3–5% [8]), the effect of hydrogen on the modification of the micro-structure would be small. Hence the influence of H contents on the Raman spectra remains as a future work. Nevertheless, the present results are very much promising for in situ Raman analysis to detect structure modification and /or to identify the structure of the redeposited layers on the plasma facing wall.

4. Conclusion

We have applied laser Raman spectroscopy to characterize plasma facing carbon tiles at the first wall and divertor of JT-60 exposed to HH discharges in June 1988–October 1988 experimental campaign. The tiles showed Raman spectra similar to those for damaged graphite by ion irradiation with two broad peaks, graphite-peak (G-peak) and disordered-peak (D-peak), centered at around 1580 and 1355 cm^{-1}, respectively. The spectra were a little different to each other depending on the analyzed location. Although the variation of the peak structures could not be well correlated to horizontal and vertical locations, data plots for the peak intensity ratio of D-peak and G-peak, (I_{1355}/I_{1580}) and the full width at the half maximum of D-peak ($FWHM_{1355}$) overlap quite well on the same plots for the ion irradiated graphite showing systematic change with temperature and irradiation dose. It

should be noted that the irrespective of tile nature i.e. used either as the first wall or divertor, and deposited or eroded, the microstructure of the carbon tile surfaces are modified through similar way as damaging by energetic ion irradiation. Since the Raman spectra can be easily taken in situ if we could introduce fibers in tokamak both for probing laser light and collecting reflecting light, the technique will be very useful for analysis of plasma facing carbon tiles.

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