Effect of Metallographic Preparation Process on Raman Spectrum of Pyrolytic Graphite

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1. Introduction

Nuclear graphite has been used as a moderator, reflector and core supporting structures in high temperature gas-cooled fission reactors. Radiation damage in graphite occurs by the impingement of energetic particles such as fast neutrons on the crystal lattice [2, 3]. Displaced carbon atoms in a cascade process create radiation defects such as lattice vacancies and interstitial atoms. The lattice strain and imperfection resulting from displacement damage causes dimensional change, irradiation creep and property change in the graphite. Therefore information on the atomistic structural radiation defects must be investigated more in detail to understand the effect of irradiation on the properties of nuclear graphite. One of the most suitable methods to study the properties of graphite is Raman spectroscopy [4].

Raman spectroscopy has been widely used for studying lattice disorder in materials. It is sensitive to both structural disorder and the resultant radiation damage. It is available to detect small change occurred on sample surfaces [5-9]. Graphite materials show that two dominant spectral peaks in first order spectral region, G- and D- bands. G-band due to the sp^2 graphite structure is located at about 1582 cm^{-1}. D-band induced from structural defects is located at about 1350 cm^{-1}. The intensity ratio of D band relative to G band (I_D/I_G) has a linear relationship with the crystallite size [8].

Sample preparation process such as polishing, crushing or etching make the sample to have lattice defects and cause the growth of high D band [5, 6, 7]. This could make errors in the determination of the intrinsic degree of disorder. However, to observe the optical microstructure of nuclear graphites, the polishing process is necessarily required. In this study, to investigate the effect of metallographic preparation process on Raman spectrum of nuclear graphite, we examined the Raman spectrum of pyrolytic graphite changing the degree of grinding and polishing.

2. Experiments

2.1 Materials

The material used in this study was a pyrolytic graphite manufactured by GE Advanced Materials-Quartz. Samples were mounted in an epoxy resin. To obtain several surface states, the samples were step-by-step ground and polished as the procedures summarized in Table 1. The applied force on the mount was about 20N and the grinding or polishing time was 1 minute for each step. After grinding and polishing, all samples were ultrasonically cleaned in ethanol to remove SiC or alumina particles which may be adhered to the surfaces.

Table 1. Grinding and polishing conditions

Step	Abrasive	Particle size	ID
1	P1200 SiC paper	15.3μm	S1530
2	P2400 SiC paper	6.5 μm	S0650
3	P2400 SiC paper	2.5 μm	S0250
4	1μm Alumina powder	1.0 μm	S0100
5	0.05 μm Alumina powder	0.05 μm	S0005

Raman scattering was obtained at room temperature using backscattering geometry. 532 nm Verdi 5.5 LASER was used to excite the samples. LASER beam size and power were 1.5 μm and 10 mW, respectively. Beam was focused on sample surface through objective lens of 60 magnifications. Scattered light from sample was dispersed through triple monochromators systems with 1800 grooves/mm. Two of three monochromators in the triple monochromator system were used for laser line rejection to filter out Rayleigh scattering from the sample upon laser excitation.

3. Results and Discussion

Fig. 1 shows the change of Raman spectra. The spectrum of unpolished reference sample is shown at the bottom. The samples have been then subjected to an increasing degree of polishing stages (spectra from the bottom to the top). All the spectra were normalized with regard to the G band. It appears clearly an increase of D band relative to G band due to polishing, whereas G band width seems to be practically unchanged.

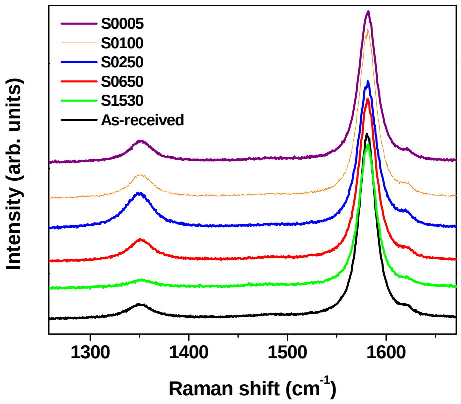

Fig. 1 Change of Raman spectra due to grinding and polishing processes

Fig. 2 shows the change of intensity ratio of D band relative to G band, I_D/I_G, due to the sample preparation process. The I_D/I_G value of the ground and polished samples was higher than that of the unpolished reference sample. As the grit number of SiC paper increased from P1200 to P4200, the I_D/I_G value increased continuously. However, the I_D/I_G value decreased after polishing with 1 μm and 0.05 μm alumina powder and the finer the alumina particle was, the smaller the I_D/I_G value was.

During the grinding process, the defects are supposed to be created on the sample surfaces due to shear deformation. As the grit of SiC paper increased, the deformation zone below the surface became thinner in proportion to the particle size of SiC particles. However, more defects were accumulated in the deformed surface and thus the I_D/I_G value increased up to grit P4200. It is well known that the penetration depth of a Raman laser into graphite is about 80~100 nm [10]. The deformed zone of the polished samples could be very thin compared with the penetration depth of the Raman laser In this circumstance, the Raman laser could react with not only the deformed zone but also the as-received region so that the I_D/I_G value of the polished samples was lower than that of the grinded samples

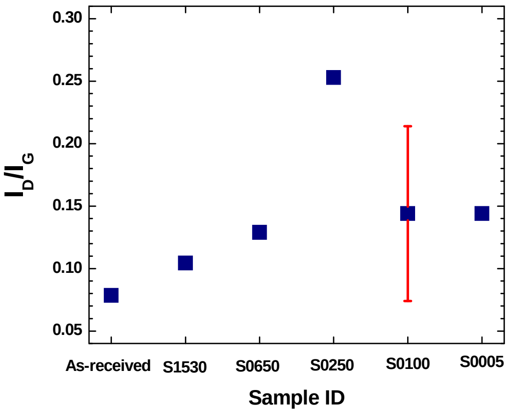

Fig. 2 Intensity ratio of D band relative to G band

4. Summary

This study investigated the artifacts occurring during the grinding and polishing process of a pyrolytic graphite. The intensity ratio of D band to G band appeared to be sensitive to the metallographic preparation process and consequently showed wrong information about the intrinsic structural disorder.

ACKNOWLEDGEMENT

This study has been carried out under the Nuclear R & D Program supported by the Korean Ministry of Education, Science and Technology.

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