§15. Applicability Study of Spectroscopic Measurement Methods in Characterization of Functional Materials for Fusion Blanket

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In the development of fusion blanket systems, usage of various oxide, carbide, nitride, hydride functional materials have been proposed for electrical insulation, suppression of tritium fuel permeation, suppression of corrosion, radiation shielding, etc. The present study has been conducted to investigate applicability of spectroscopic measurement methods for inspection of initial condition of the functional materials and for detection of property changes in a reactor operation. Spectroscopic measurements are expected to be significantly effective for the features of (1) non-contact, non-destructive, (2) acquisition of information on a crystal condition such as band structure and crystallinity and (3) high spatial resolution of < 1 μm.

In our previous studies, cathodoluminescence spectrum (CL) measurement has been performed on SiC, B_4C, Er_2O_3 and AlN with a general 12 keV electron gun. In fusion blanket development, SiC and B_4C has been studied as a structural material and neutron shielding material, respectively. Er_2O_3 and AlN have been studied as electrical insulator and tritium fuel permeation barrier. In the fiscal year of 2012, CL measurement system was newly installed to a scanning electron microscope (SEM) and its applicability was examined for characterizing blanket functional materials with a high spatial resolution. In the SEM-CL system, a electron beam is focused to a spot of <1 μm in diameter on a sample surface, and luminescence light is collected by a mirror in an optical column. The light is guided out of vacuum chamber of SEM with an optical fiber and the spectrum is obtained with a CCD spectrometer.

SEM-CL spectra have been obtained for various blanket functional materials of Li_2CO_3, Li_2ZrO_3, Li_2O, $CaZrO_3$, Y_2O_3, Flinak, NaF, LiF and KF. While the luminescence intensities were different with the composition and crystallinity, spectra data could be obtained within 10 second for most samples. This is because the electron beam current in SEM can be changed by more than three orders. The SEM-CL measurement could achieve the spatial resolution of <1 μm even in measurements with a high electron beam current. It was also confirmed that Er_2O_3 and Y_2O_3 coatings fabricated by the metal organic decomposition (MOD) method showed sufficient luminescence intensity for spectrum measurement, although such coatings have low crystallinity due to the limitation of calcination temperature. In the previous study, it has been found that the intensity in 640-700 nm had a correlation with crystallinity in Er_2O_3 coatings [1]. In the present study, relation between crystallinity and luminescence spectra in Y_2O_3 coatings fabricated by the MOD method and sintered B_4C were examined.

SEM-CL spectra for an Y_2O_3 coating and sintered Y_2O_3 sample are shown in Fig. 1. A luminescence peak is observed around 350 nm in sintered bulk sample. On the other hand, in coating sample, a peak around 450 nm is observed and intensity of the peak around 350 nm increases with calcination temperature. The balance of the peaks of 350 nm and 450 nm could be used for examination of crystalinity of the Y_2O_3 coating.

In a sintered B_4C sample, SEM-CL spectrum was compared before and after a 34 keV H^+ beam irradiation. As shown in Fig. 2, the luminescence peak position shifted from 550 nm to 570 nm in the irradiated part. It can be considered that the red shift in luminescence occurs when the crystallinity degrades with defects.

The correlation between the crystal conditions and luminescence spectra will be continuously examined, and comparison with the change in the band structure obtained from theoretical calculation will also be studied.

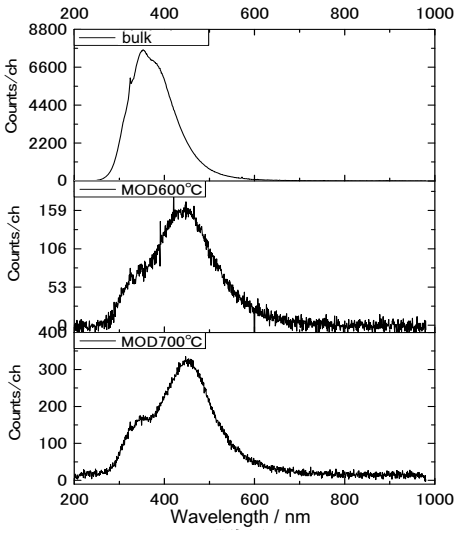

Fig. 1. SEM-CL spectra of sintered bulk Y_2O_3 sample and Y_2O_3 coating sample fabricated by MOD method.

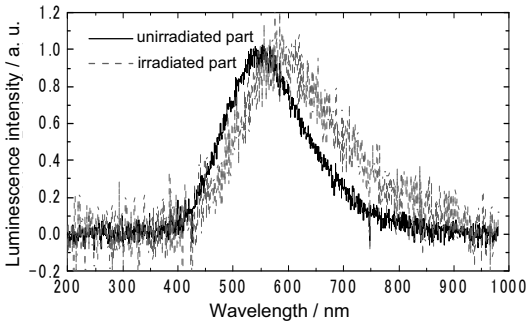

Fig. 2. Change in SEM-CL spectrum of 34 keV H^+ beam irradiated sintered B_4C sample.

[1] Tanaka, T., et al., Journal of Nuclear Materials 417 (2011) 794–797.