

§11. Ion-beam Induced Luminescence Spectra of Er_2O_3 at Elevated Temperatures

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This study is undertaken to develop optical methods for *in-situ* characterization of radiation-induced defects in Er_2O_3 by energetic ion bombardment. The characterization is important for qualification of Er_2O_3 coatings as electric insulation of Li/V-alloy blanket systems and as hydrogen permeation barriers.

A potentially useful luminescence band in 640–690 nm is identified as $4f^{11} \ ^4F_{9/2} - \ ^4I_{15/2}$ transition of Er^{3+} at C_2 cation sites. It has been demonstrated that ion bombardment on a plasma splay coating sample and a sintered bulk sample quenches preferentially the luminescence band.

The blanket material will be used at elevated temperatures in fusion reactors. Annealing effects in damaged ceramics at the elevated temperatures are issues to be investigated. The annealing effects may be inferred from ion-beam induced luminescence spectra measured with heated samples. In this work, the first preliminary measurement of the luminescence has been performed with an Er_2O_3 sample heated at 300°C.

The Er_2O_3 sample (high purity (3N) sintered plate, 15mm×15mm wide and 1mm thick, TEP CORP.) is mechanically attached on a micro ceramic heater equipped with a thermocouple (25mm×25mm wide, MS-1000R, SAKAGUCHI E.H VOC CORP.). Ceramic fiber papers are used to thermally insulate the ceramic heater from a target holder on which the ceramic heater is set. Temperature control of the heater is performed automatically with the aid of a thermoregulator (SCR-SHQ-A, SAKAGUCHI E.H VOC CORP.). With this experimental setup, temperatures of the target sample were gradually increased up to 600°C in vacuum. The temperature of the target sample is deduced assuming thermal equilibrium with the ceramic heater.

Ion-beam induced luminescence of the target sample is measured with an experimental apparatus at NIFS consisting of an ion-beam source, a collision chamber, and a UV-visible spectrometer equipped with a Peltier cooled CCD camera. The ion source is a part of medium current ion implanter (ULVAC IM-200MH-FB) originally used for semiconductor production (Freeman-type). Ion-beams extracted from the Freeman ion source are introduced into the collision chamber after analyzing the mass to charge ratio by a magnet. In the ion source, a mixture gas of H_2 and Ar (or Kr) was used to extract H^+ ion-beams of $\sim \mu\text{A}$ in current at about 30 keV in kinetic energy.

Photon emission is measured at a right angle to the ion-beam axis. The sample surface is leaned 45 degrees toward the line of sight so that the photons emitted in

a area of the ion-beam spot are collected by a condenser lens focused on a slit of the spectrometer.

Fig. 1 shows measured luminescence spectra induced by H^+ ions bombardment on the Er_2O_3 sample with and without heating, respectively. Wavelength calibration of the spectra was done by using Balmer- α and - β emission lines from a hydrogen discharge tube. Bunch of emission lines seen in the measured spectrum without heating are assigned to Stark components of the luminescence due to the $4f^{11} \ ^4F_{9/2} - \ ^4I_{15/2}$ transition. In the spectrum of a heated sample, a strong line appears at 655 nm which is absent in the spectrum without heating. This line can be assigned to the Balmer- α (656.28 nm) emitted by neutral hydrogen atoms released from the target. Correspondingly, the Balmer- β line is also identified at 485 nm. Since the apparent blue-shift and the Doppler broadening correspond to an kinetic energy as high as ~ 1 keV, the origin of the emitters should not be ascribed to thermal desorption enhanced at elevated temperatures but to backscattering of energetic incident ions.

Since reflection coefficients of the energetic H^+ ions on Er_2O_3 are small, the apparently strong Balmer- α may indicate that the luminescence of Er^{3+} ions are decreased substantially at 300°C. Excited states population of Er^{3+} ions in the oxide is dissipated before emitting photons at higher temperatures.

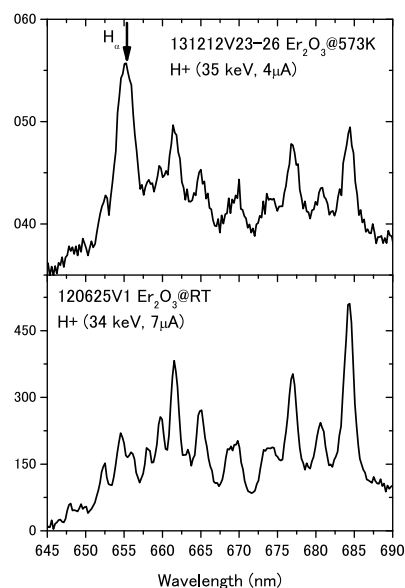


Fig. 1: H^+ ion-beam induced luminescence spectra of Er_2O_3 . Upper: A spectrum for a sintered sample heated at 300°C. Arrow indicates the blue-shifted hydrogen Balmer- α line, H_α . The spectrum is obtained by adding photon counts in four successive measurements; 2 min. sampling in the each measurement. Lower: A spectrum measured without heating.