Chemistry

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XPS study on Ce-Zr-Y oxide nanoparticule catalysts

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Introduction

Nanoparticule catalysts of solid solution of ceria and zirconia doped with yttria are expected to be useful for clean up of an automobile exhaust gas. Surface layer of the nanoparticule catalysts were characterized by a non-destructive depth profiling analysis with a variable excitation XPS using synchrotron radiation. The high surface sensitivity of this technique has allowed us to measure the ultra thin thickness of the surface layer on the nanoparticule without damage. The XPS of 3d of Zr and Y were measured in the excitation x-ray energy range of 100 - 900 eV at the BL-13C.

Experimental

The samples of Ce-Zr-Y oxide nanoparticle catalysts were prepared by precipitation from homogeneous solution and colcined at 873 – 1473 K under atmosphere. The XPS of 3d of Zr and Y were measured in the excitation x-ray energy range of 100 - 900 eV at the BL-13C. Powder samples were measured with a slit opening 20 μm. and the resolution was estimated E/ΔE = 4000 at 400 eV by evaluation of S2p of MoS_2 crystal.

The measurement of the incident x-ray intensity (I0) was measured by drain current from the gold mesh refreshed by evaporation (20 mesh). XPS spectra were measured by PHI ESCA-5500 modified for synchrotron radiation. The analysis area of XPS was ϕ0.8 mm with an aperture of ϕ4 mm and collection angle of 7 degree. To suppress of charging up, electron neutralizer combined with Ar ion gun was used [1].

Results and Discussion

Figure 1 shows XPS spectra including Zr3d and Y 3d of CZ-Y(20) 1473 K excited by various x-ray energy of 250-860 eV. The peak around 170 eV observed at low excitation energy is not assigned but it may be surface contaminants.

Figure 2 shows retios of peak area of Y3d and Zr3d vs. incident x-ray energy on the Ce-Zr-Y samples. The peak areas were obtained by Gauss-Lorentzian fitting. The concentration of Y is high and unstable at 873 K of calcine temperature. This means crystallization of ZrO2 is not complete while it complete for the samples calcined at higher temperature. It was not clear that concentration of Y from this results. We will follow the samples of other preparation condition and other doping elements.

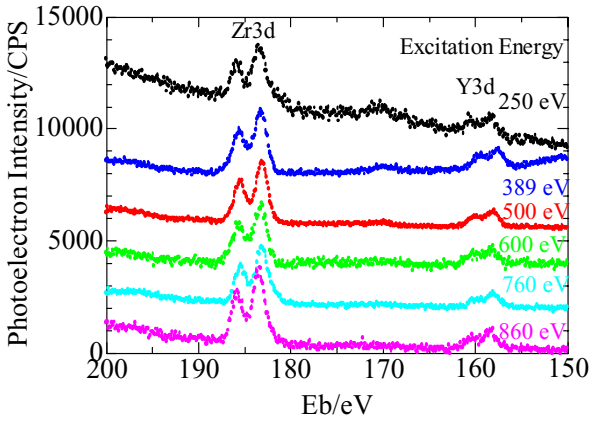

Fig. 1 XPS spectra of Zr 3d and Y 3d of CZ-Y(20%) culcined at 1473 K excited by various x-ray energy of 250 - 860 eV.

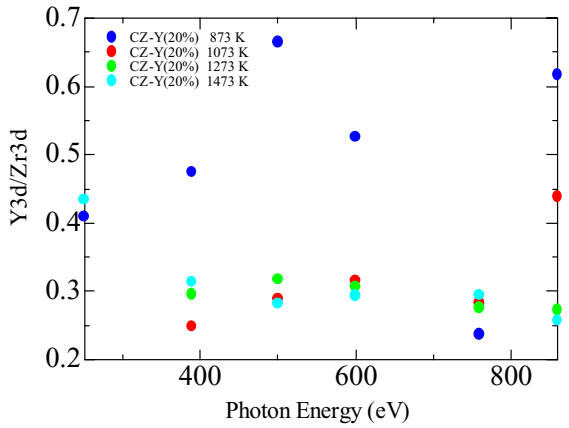

Fig. 2 The ratios of Y 3d/Zr 3d of XPS of CZ-Y(20%) calcined at 873 – 1473 K ploted vs excitation energy of 250 - 860 eV.

References

[1] T. Tanaka *et al.,* J. Elec. Spec. Rel. Phen., **114-116**, 1077 (2001).

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