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## Time-resolved X-ray Diffraction Studies of Zr-doped CeO<sub>2</sub> Nano-particles

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Beamline(s): X7B

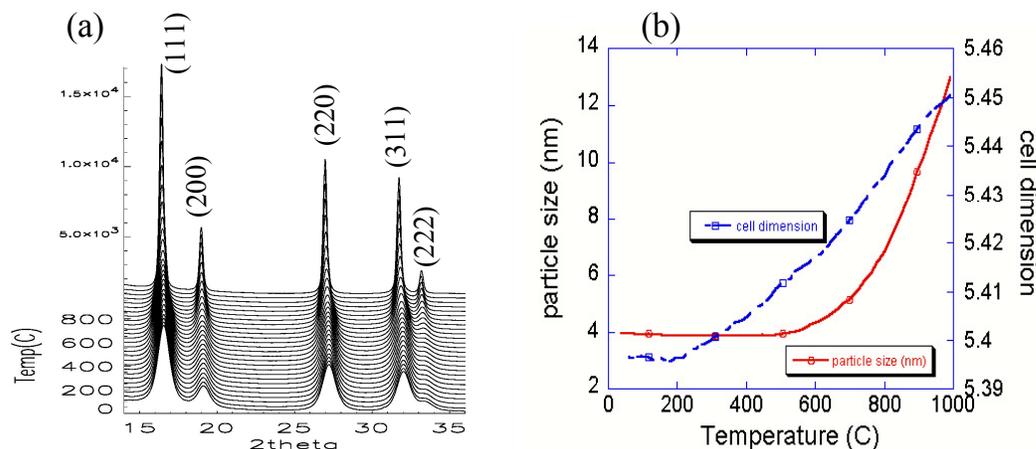
**Introduction:** Zr-doped ceria is an important catalyst used for three-way automotive exhaust converters. The studies on the thermal behavior of ZrCeO<sub>2</sub> nano-particles are the first in a series of investigations on doped metal oxide nano catalysis.

**Methods and Materials:** The Ce atoms in the CeO<sub>2</sub> nano-particles were replaced by Zr atoms by ratios of 1:9, 1:2 and 1:1 using a novel method developed at the Instituto de Catalisis, Madrid, Spain [1]. Preliminary TEM studies showed that these powders were in an order of nanometer sizes.

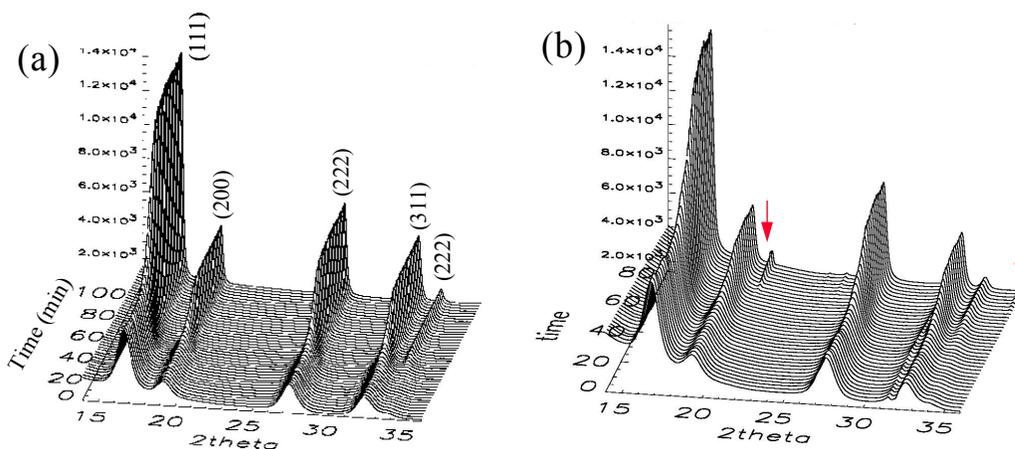
**Results:** The Zr<sub>x</sub>Ce<sub>1-x</sub>O<sub>2</sub> (where x=0.1, 0.33, and 0.5) powder samples were heated from 25°C to 1000°C in air by different heating conditions, and time-resolved synchrotron x-ray diffraction (XRD) data were recorded simultaneously in transmission using a MAR345 image plate based detector system at wavelength 0.9034Å. As shown in figure 1-a (for x=0.1), the CeO<sub>2</sub> phase peaks are getting narrower with increasing temperature (heating rate = 8°C/min), indicating that the coherence length of the phase is increased. For example, the FWHM of the (111) peak is decreased from 1.4 to 0.4 degree in 2θ, suggesting that the particles grow from 4 to 12 nanometer. The unit cell also increased with increasing temperature (see figure 1-b). Similar results were observed from the x=0.33 and 0.5 samples (figure 2-a and b, respectively) when they were heated to 1000°C and held at 1000°C for about one hour. For x=0.5 sample, however, extra peaks (indicated by arrows in figure 2-b) were found presumably due to a segregation of ZrO<sub>2</sub> phase.

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**References:** [1]. M. Fernandez, *et. al.*, J. Catalysis, **194**, 385 (2000).



**Figure 1:** Time-resolved x-ray diffraction patterns taken from Zr<sub>0.1</sub>Ce<sub>0.9</sub>O<sub>2</sub> nano-particle sample (a), and their changes in particle sizes and cell parameters (b). The CeO<sub>2</sub> phase peaks are indexed in (a).



**Figure 2.** Time resolved x-ray diffraction patterns obtained from Zr<sub>0.33</sub>Ce<sub>0.67</sub>O<sub>2</sub> (a), and Zr<sub>0.5</sub>Ce<sub>0.5</sub>O<sub>2</sub> (b). Extra phase peaks are indicated by arrows in (b).