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Synthesis and characterization of Cu- doped ceria nanopowders

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Abstract – Nanopowdered $Ce_{1-x}Cu_xO_{2-\delta}$ samples ($0 \le x \le 0.15$) were synthesized by a self-propagating room temperature synthesis. Samples were characterized by XRD, Raman spectroscopy and nitrogen adsorption-desorption measurements. $Ce_{1-x}Cu_xO_{2-\delta}$ particles exhibit a fluorite-type structure with and average particle size of 4 nm. Raman spectroscopy shows that these compounds in low doping regime (up to 7.5 % of Cu), form a solid solution that maintain fluorite structure of CeO_2. With further increase of Cu concentration additional mode appears at ~ 620 cm^{-1} which is ascribed to CuO structure. Nitrogen adsorption-desorption measurements reveal that doping increases the specific surface area of nanoparticles.

$Ce_{1-x}Cu_xO_{2-\delta}$ nanopowders ($0 \le x \le 0.15$), were synthesized by a self-propagating room temperature synthesis (SPRT) using metal nitrates and sodium hydroxide as the starting materials. The synthesis involves hand-mixing of precursors and exposure to air for three hours after which mixture is suspended in water. Rinsing out of reaction byproduct ($NaNO_3$) was performed by centrifuge. This procedure was repeated three times with distilled water and twice with ethanol [1].

Characterization of structural properties, phase stability and particle size was performed with X-ray diffraction (XRD) spectroscopy at room temperature. $Ce_{1-x}Cu_xO_{2-\delta}$ particles exhibit a fluorite-type crystal structure characteristic for CeO_2. Average particle size of about 4 nm was determined from Ritveld analysis of XRD data.

First order Raman active F_{2g} mode, in $Ce_{1-x}Cu_xO_{2-\delta}$ samples, shifts to lower frequencies with increase of dopant concentration up to 7.5% Cu. Further increase of Cu content leads to increase in F_{2g} mode frequency and additional mode appears at ~ 620 cm^{-1} which is ascribed to B_g Raman mode characteristic for nanocrystalline CuO structure [2]. From Raman scattering results we have concluded that these compounds are solid state solutions in low doping regime (until 7.5% Cu). Additional mode ascribed to intrinsic oxygen vacancies in ceria lattice is located at 600 cm^{-1}. Intensity of this mode increases with doping content as a consequence of increased oxygen vacancy concentration.

Nanopowders were characterized by nitrogen adsorption-desorption measurements and it was revealed that the radius of pores varies between 2 and 6 nm which means that all samples are mostly mesoporous according to IUPAC classification. Presence of Cu increases the specific surface (≈ 100 m^2g^{-1}) compared to pure ceria (70 m^2g^{-1}).

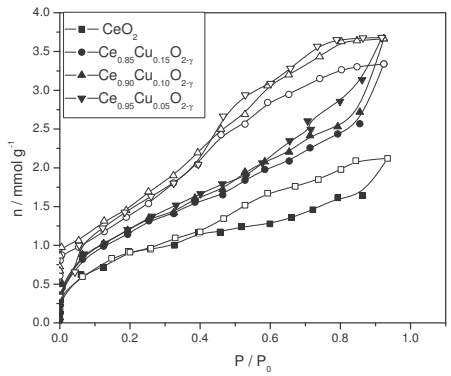

Figure 1: Nitrogen adsorption isotherms for CeO_2 sample and samples of CeO_2 with different amount of Cu. Solid symbols - adsorption, open symbols – desorption.

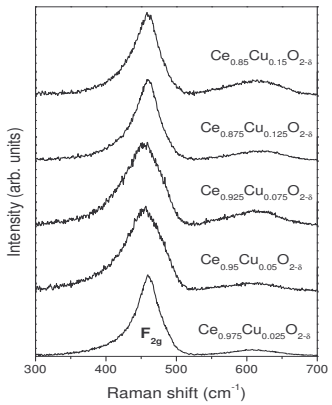

Figure 2: Room temperature Raman spectra of $Ce_{1-x}Cu_xO_{2-\delta}$ samples ($0 \le x \le 0.15$)

References

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[2] J. F. Xu, W. Ji, Z. X. Shen, W. S. Li, S. H. Tang, X. R. Ye, D. Z. Jia and X. Q. Xin, J. Raman Spectrosc. 30, (1999) 413–415.