Synthesis and characterization of Dy_2O_3 nano crystalline power by using a combustion Urea process

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ABSTRACT.

Urea assisted combustion process was used for the synthesis Dy_2O_3 nano crystalline powder. The formation of crystalline phase and structure of the Dy_2O_3 compound were identified by XRD, FTIR and SEM - EDX measurements.

1. INTRODUCTION

Ceramic materials with high chemical durability, high melting point, high hardness, wear resistance are attractive candidates for use as engineering components in extreme environmental conditions]. The potential of engineering ceramics for high temperature applications is not fully exploited because of the brittle nature of the ceramics [5-6]. One of the established methods of reducing brittleness of ceramics is toughening the ceramics by the controlled application of martensitic phase transformation. Rare earth oxides are the better potential candidates for development as transformation toughners as they undergo martensitic transformation between 2000 - 500°C accompanied by a volume change of ~9%, compared to tetragonally stabilized ZrO_2. Among the trivalent rare earth oxides, Dy_2O_3 exhibits the highest B→C martensitic transformation temperature at ~ 1900 °C. Metal oxides are usually produced by the thermal decomposition of inorganic precursors such as metal acetate, metal oxalate, metal nitrate, metal hydroxide, etc [1-3]. The step of decomposition often causes physical and chemical changes, which result in a variation in the stoichiometry, crystal structure, surface morphology and activity of the product oxide [4]. In Many wet chemical methods, like, sol-gel, combustion, polyol, etc., have been developed in order to overcome the above mentioned difficulties. Among the available, combustion process is found to simple and capable of producing nanocrystalline powder at a lower temperature in a short time. Therefore, in the present work, the Urea assisted combustion process have been investigated to prepare dysprosium oxide nano crystalline powder and characterized by XRD, FTIR and SEM - EDX.

2. EXPERIMENTAL

Analytical grade chemicals of dysprosium nitrate, citric acid (qualigens fine chemicals) and urea (qualigens fine chemicals) are used as the starting materials. The metal ions, citric acid and urea ratio was kept 1:1:1. The required amount of each precursor chemical was separately dissolved in distilled water. Citric acid, urea and dysprosium solutions were mixed under stirring at 90^{O}C up to 5 hours. The formed clear mixed solution was kept in oven to form the gel. The obtained gel was heated at 473K and 523K for 4 hours. The phase and the structural evaluation of the heat treated gel were monitoring respectively by XRD and FTIR techniques.

3. RESULTS AND DISCUSSION

X' Pert PRO MPD, PANalytical (Philips), X-ray diffractometer with Cu Kα radiation

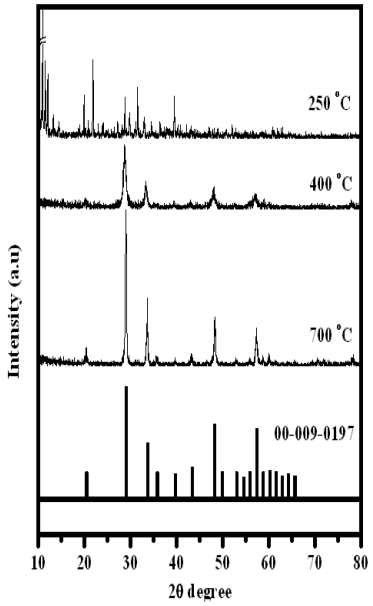

Fig 1. XRD patterns for Dy_2O_3 sample

of wavelength $\lambda = 1.5418$ Å was used to record the XRD patterns for Dy_2O_3 sample between 80° and 10°, 2θ values at a scan rate of 2° per minute. NBS silicon standard was used for the estimation of instrumental broadening. FTIR spectra were recorded using a Shimadzu FTIR-8300/8700 spectrometer in the range 4000–400 cm^{-1} at 40 scans for the pellets of Dy_2O_3 sample. All pellets were made with KBr powder and Dy_2O_3 sample using a KBr mini press. The shape and size of the prepared samples were investigated using a scanning electron microscope (SEM), JEOL-JSM6400 scanning electron microscope with an accelerating voltage of 20 keV.

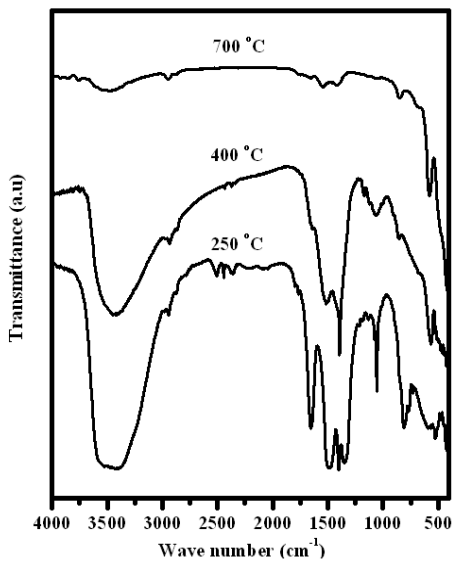

Fig 2. FTIR spectra for Dy_2O_3 sample

Fig 1 shows the XRD patterns of the heat treated gel at 250^0C, 400^0C and 700^0C. From Fig 1, the observed peak free XRD patterns of gel heated at 250^0C confirm the amorphous nature. For the gel heated at 400^0C and 700^0C, the observed XRD peaks and its analysis revealed the formation of nanocrystalline Dy_2O_3 phase was confirmed on comparison with the JCPDS. File number #09-0197[9]. The crystallite size was calculated using the Scherer equation[10],

$$D = \frac{k\lambda}{\beta \cos\theta}$$

where D is the crystallite size, k is a constant (=0.9 assuming that the particles are spherical), λ is the wavelength of the X-ray radiation, β is the line width (obtained after correction for the instrumental broadening) and θ is the angle of diffraction. The average crystallize size obtained from XRD data is found to be ~28 nm.

Fig. 2 shows the FTIR spectra of the heat-treated gel at 250^0C, 400^0C and 700^0C. In Fig. 2, the observed variation of IR band positions and also band intensities revealed that the structure of sample changes when it was heated at different temperatures. For the sample heated at 700^0C, the observed IR band positions revealed the formation of Dy_2O_3 structure and also the removal of water molecules [11].

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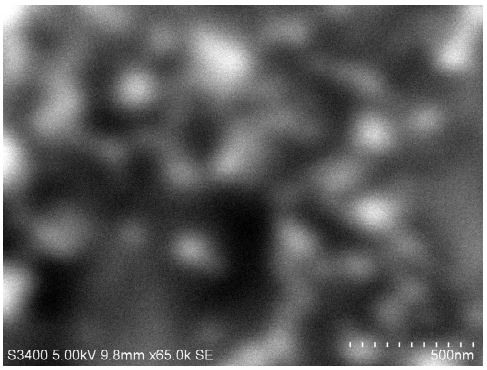

Fig 3 SEM EDX Image

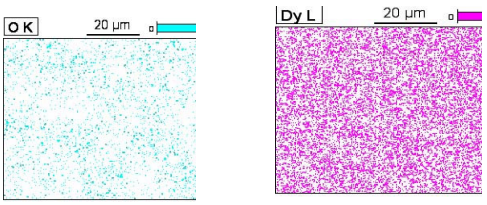

Fig 4. SEM–EDX mappings

The microstructure and elemental analysis of the Dy_2O_3 formation was conform using SEM - EDX measurements. SEM micrograph of fig.4 showed an agglomerated spherical particles of Dy_2O_3 and their particle size is ~200nm. SEM–EDX spectrum and mappings shown in fig.5 & 6 are respectively confirm the existence and uniform distribution of O and Dy in the Dy_2O_3 . SEM- EDX results confirm the formation of the Dy_2O_3, shown in table.1, and it is free from organic contamination.

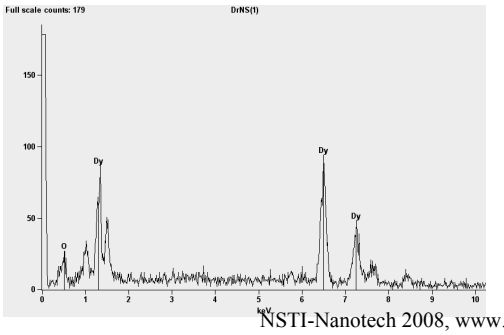

Fig 5. SEM–EDX spectrum

Element	*Net Counts*	*Weight %*	*Atom %*	*Formula*
O	339	11.45	56.77	O
Dy	2524	88.55	43.23	Dy
Dy	1367	---	---	
Total		100.00	100.00	

CONCLUSION

SEM–EDX spectrum and mappings SEM–EDX spectrum and mappings Nano crystalline Dy_2O_3 power was Synthesized by using combustion process. The formation of nanocrystalline phase and structure of the Dy_2O_3 compound were identified by monitoring the heat treated dried gel, at various temperatures, from amorphous to crystalline growth through XRD and FTIR measurements. Crystallite size of the Dy_2O_3 powder, calculated using its XRD data and the Scherer equation, $D = k\lambda/\beta cos\theta$, is found to be ~28 nm. SEM micrograph of fig.4 showed an agglomerated spherical particles of Dy_2O_3 and their particle size is ~200nm. SEM–EDX spectrum and mappings, shown in fig.5 & 6, are respectively confirm the existence and uniform distribution of O and Dy in the Dy_2O_3 . SEM- EDX results confirm the formation of the Dy_2O_3, shown in table.1, and it is free from organic contamination

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