SYNTHESIS AND CHARACTERIZAION OF $Ni_{0.75}Co_{0.25}Al_2O_4$

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Abstract

Nanocrystalline cobalt doped nickel aluminate ($Ni_{0.75}Co_{0.25}Al_2O_4$) synthesized by the solid-state reaction with Alluminum nitrate, Cobalt nitrate and Nickel nitrate as source of nickel, cobalt, aluminum respt. The prepared sample was characterized by thermogravimetry analysis, FTIR, X-ray diffraction and scanning electron microscopy. The FT-IR spectra of spinel $Ni_{0.75}Co_{0.25}Al_2O_4$ nanoparticles shows characteristics peaks spinal section at 715-508 cm^{-1} that ensure the cubical spinel structure. The formation of the specified $Ni_{0.75}Co_{0.25}Al_2O_4$ mixed metal spinel was confirmed by diffraction analysis. Prepared samples were pure crystalline materials have crystallite size 22.26 nm.

Introduction

Development of latest materials and their process has helped within the evolution of latest technologies in each field of science. In the above surroundings, the most focus of current analysis activities on mineral aluminates is twofold, viz. basic curiosity to understand however bulk properties transform because the size of the particle is reduced all the way down to nanoscale and therefore the study of doping effects of varied metal ions, thereby elaborating their structural, dielectric and electrical properties. For this purpose, an understanding of the dielectric properties of nano-crystalline $MgAl_2O_4$ [1-3], $NiAl_2O_4$ [4-6] and $SrAl_2O_4$ [7, 8] spinel aluminates in terms of particle size, temperature, and frequency of the applied field were developed [9]. Aluminate spinels generally present high thermal stability and melting point, mechanical stability, and resistance to alkalis and acids [13]. In particular, due to its defect chemistry, nickel aluminate is being considered in catalytic applications and proposed as a candidate material in high-temperature fuel cells [14]. $NiAl_2O_4$ is also a potential candidate in metal-ceramic composite because of its excellent strength and wettability with metal [15].

The present work successfully demonstrate the synthesis and characterization of spinel $Ni_{0.75}Co_{0.25}Al_2O_4$ nanoparticles through sol-gel combustion method. The impact of cobalt doping concentration on the structural, morphology and magnetization of spinel $Ni_{0.75}Co_{0.25}Al_2O_4$ nanoparticles has been investigated. The prepared samples were characterized by TGA, XRD, FT-IR, SEM.

Synthesis of $Ni_{0.75}Co_{0.25}Al_2O_4$

Material method:

For the synthesis of cobalt doped nickel aluminate $Ni_{0.75}Co_{0.25}Al_2O_4$ spinel system chemicals were of analytical grade obtained from Merck, India.

Experimental procedure for the synthesis of $Ni_{0.75}Co_{0.25}Al_2O_4$ spinel system:

The system comprises wet chemical mixing of the 4.567g of nickel nitrate and 18.75g of aluminum nitrate and glycine were slowly mixed with constant stirring till the homogeneous mixture of gel is formed. The resulting gel was heated on hot plate at 250ºC temperature for 15-20 minutes so that combustion of gel takes place with the great deal of foams produced and spark appeared at one corner which spread through the mass, yielding a dark brown voluminous and fuzzy product in the container heated till the formation blackish char. Then the char was collected in to the crucible and heated up to the 900ºC for 2 hours in muffle furnace which form sky blue coloured nano-particles.

Result and Discussion

TGA Study:

The thermogram shows three sections of weight loss as shown in fig 1. The primary section of mass loss taking place between concerning 100-150 ºC temperatures involved in decomposition of organic matter within the second step of obtained char was transform throughout continuous heating on 150 to 500 ºC as shown in region of second weight loss. The final weight loss takes place between the temperature 500-800 ºC as in the third section. The determined TGA Curve showed 61.92% as total weight loss that occurred at three fully altered temperature sections.

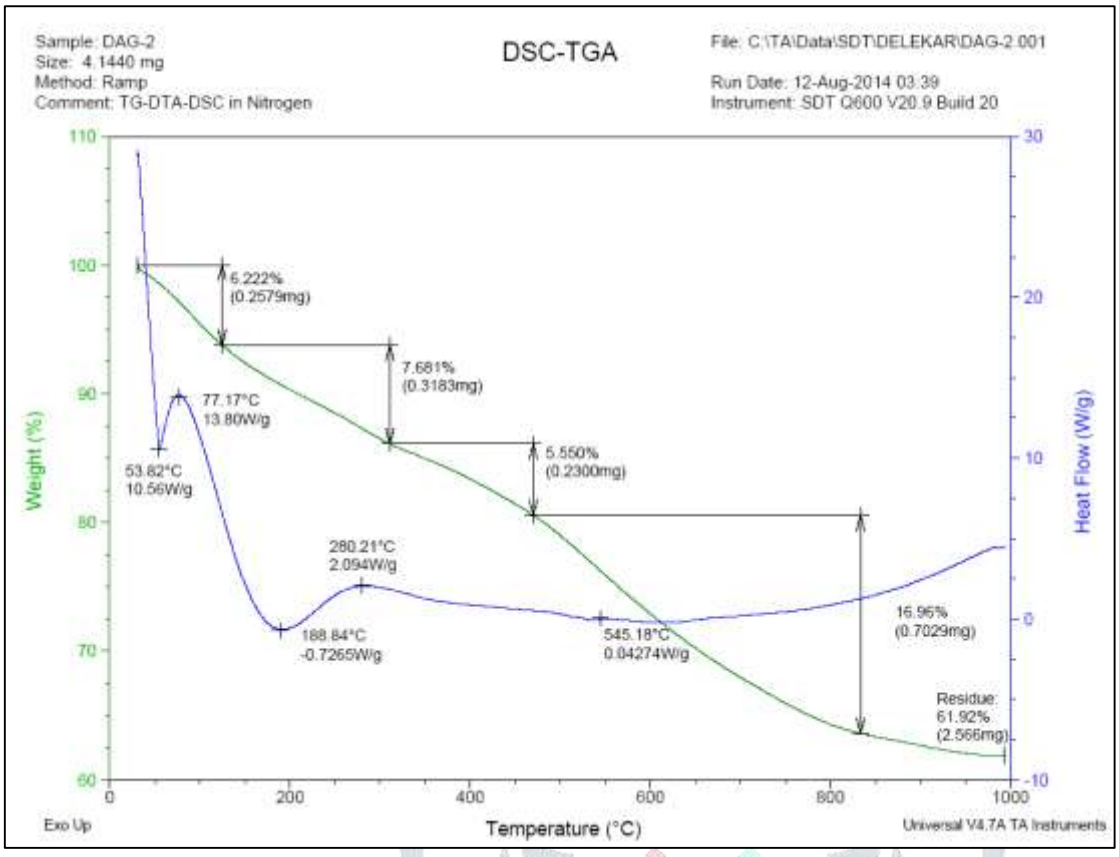

Fig 1. TGA $Ni_{0.75}Co_{0.25}Al_2O_4$ spinel

FT-IR Study

The FT-IR spectra of $Ni_{0.75}Co_{0.25}Al_2O_4$ spinel nanoparticles as shown in fig. 2, An influential and broad absorption band observed at all over the place within the region 3453 cm^{-1}, which may be allocated to the vibrations of binary compound molecules. A band observed at around 1631 cm^{-1} is present altogether compositions, which might be appointed to the H−O−H bending vibration. FTIR spectrum shows characteristics peaks spinal fragment at approximately 711-516 cm^{-1} that confirm the cuboidal spinel structure. The detected peak at 516 cm^{-1} was associated with broadening vibration mode of Al−O for the octahedral coordinated Al^{3+} ions.

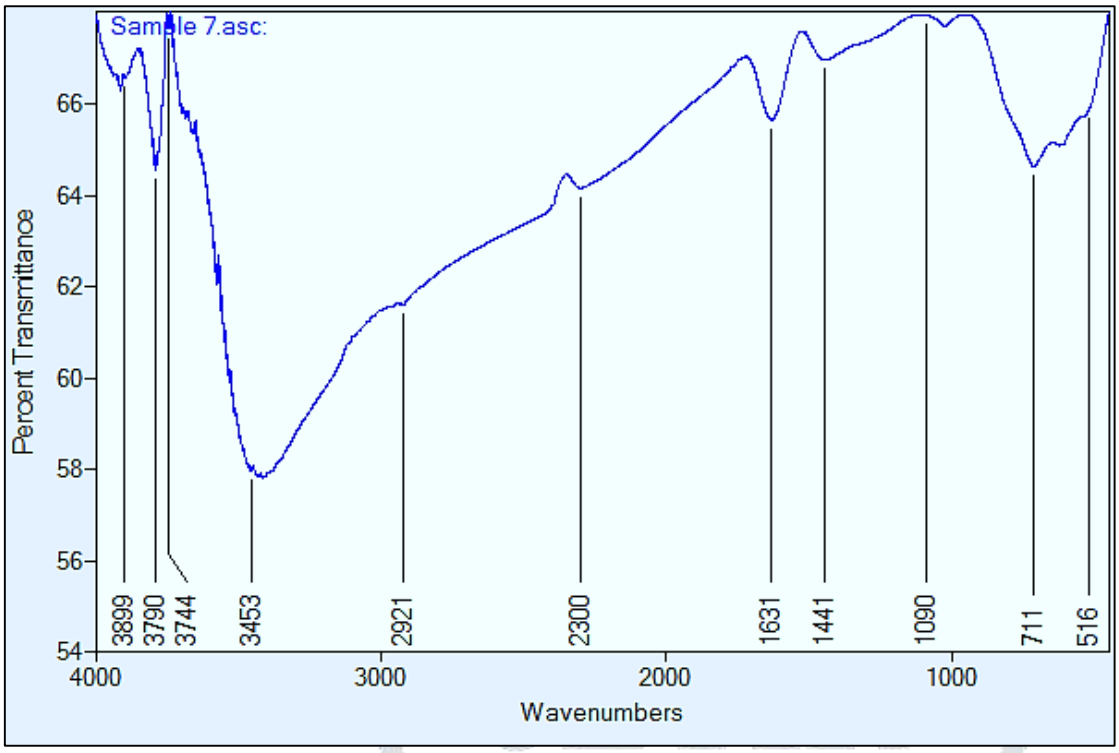

Fig. 2 FTIR of $Ni_{0.75}Co_{0.25}Al_2O_4$ spinel

XRD Study :

Development of the specified $Ni_{0.75}Co_{0.25}Al_2O_4$ mixed metal spinel was confirmed by diffraction study. The XRD pattern shown depicts broad peaks because of the nano-sized of particles and equivalent to the reported (JCPDS card No. 010-0339) data. The observed peaks at 2θ = 31.38°, 36.96°, 45.02°, 55.78°, 59.66°, 65.58° and 77.44° that appropriately be assigned to the reflections reminiscent of (220), (311), (400), (422), (511), (440) and (533) miller indices planes of $Ni_{0.75}Co_{0.25}Al_2O_4$ correspondingly X-ray diffraction pattern that indicates that the synthesized sample was pure crystalline materials.

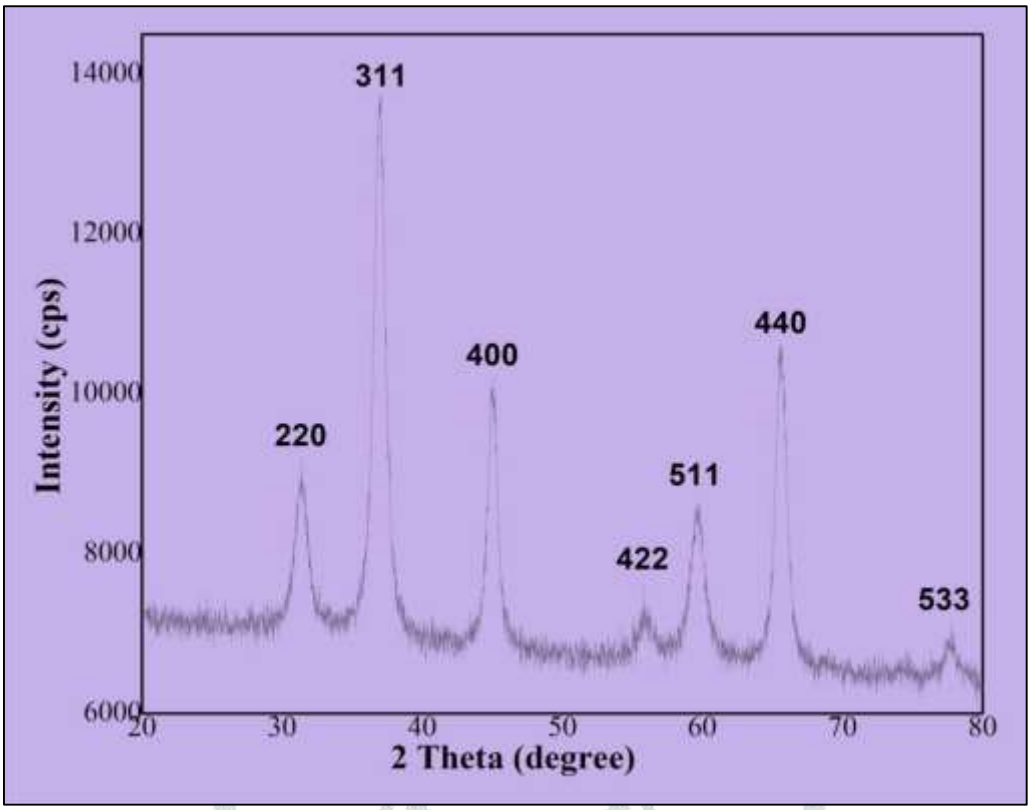

Fig 3. XRD of $Ni_{0.75}Co_{0.25}Al_2O_4$ spinel

The average crystallite size was projected by applying the Debye Scherrer's equation on the three main intense peaks at 2θ = 36.96°, 45.02° and 65.58° of the $Ni_{0.75}Co_{0.25}Al_2O_4$ spinel concentration. The average calculated crystallite size was found in the 22.26 nm range.

SEM Study:

The structure and morphologies of spinel $Ni_{0.75}Co_{0.25}Al_2O_4$ nanoparticles were confirmed by the scanning electron microscope (SEM). Fig. 4 containing the magnification ranges shows SEM images of $Ni_{0.75}Co_{0.25}Al_2O_4$ 20kV 500 magnifications for 50µm range, 2000 magnifications for 10µm range, 5000 magnifications for 5µm range and 10000 magnifications for 1µm range respectively. The SEM images consist of particle-like nanostructures with irregular grain size smaller than 50 nm.

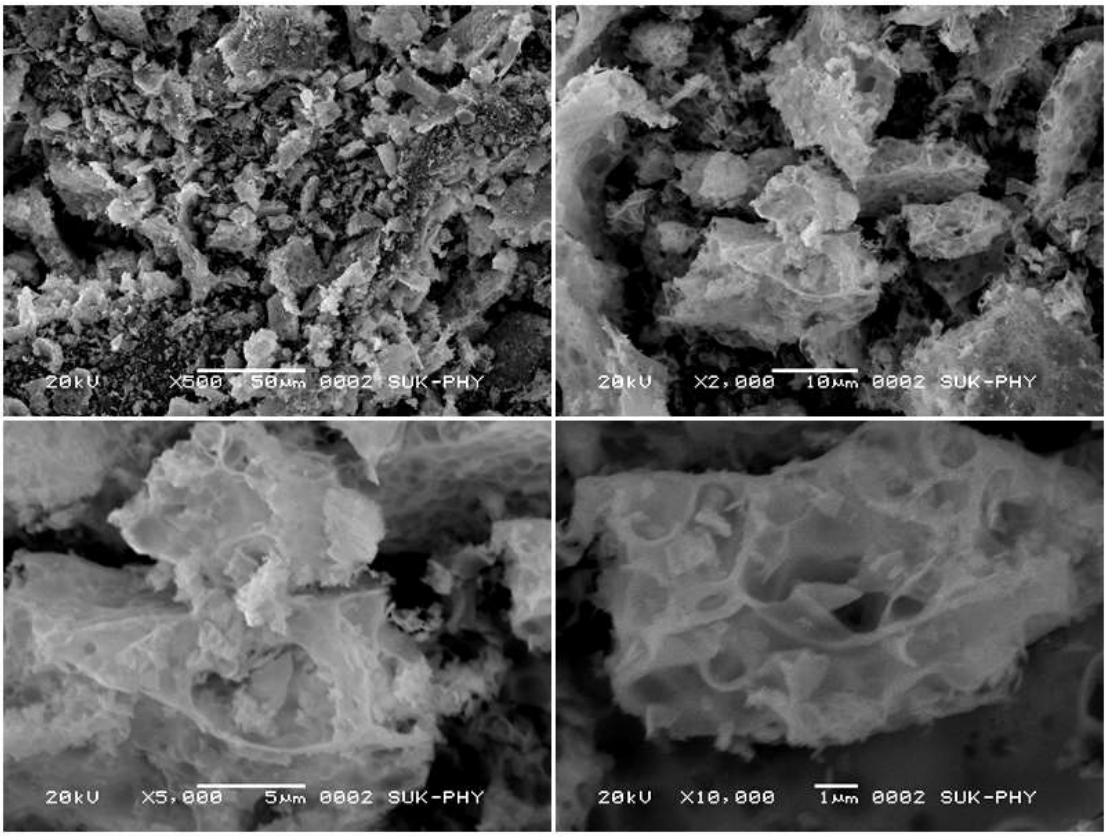

Fig 4. SEM of $Ni_{0.75}Co_{0.25}Al_2O_4$ spinel

Conclusion

In this paper, we have investigated the Nanocrystalline blue coloured cobalt doped nickel aluminate ($Ni_{0.75}Co_{0.25}Al_2O_4$) at an annealing temperature of 900 °C using the solid state reaction. According to TGA it was shown that, The weight loss was terribly negligible at on top of 900^0C. The X-ray diffraction planes and all peaks are completely indexed as an intensive cube-shaped spinel $Ni_{0.75}Co_{0.25}Al_2O_4$ nanostructure having 22.26 nm crystallite size. The particle morphology is suggesting irregular porous grain having possible applications in the field of nanotechnology.

References:

[1] Pinga L. R., Azadb A. M. and Dunga T. W., Materials Research Bulletin, 36, (2001), 1417-1430

[2] Ganesh I., Bhattacharjee S., Saha B. P., Johnson R. and Mahajan Y.R., Ceramics International, 27(7), (2001), 773-779 (doi.org/10.1016/S0272-8842(01)00029-3)

[3] Guo J., Lou H., Zhao H., Chai D. and Zheng X., Applied Catalysis A: General, 273, (2004), 75-82 (doi.org/10.1016/j.apcata.2004.06.014)

[4] Otero Arean C., Penarroya Mentruit M., Lopez Lopez A. J. and Parra J. B., Colloids and Surfaces, A: Physicochemical and Engineering Aspects, 180, (2001), 253-258 (doi.org/10.1016/S0927-7757(00)00590-2)

[5] Escalona Platero E., Otero Arean C., Parra J. B., Res. Chem. Intermed., 25 (1999), 187

[6] Fung Yi-Lan E. and Wang H., Journal of Membrane Science, 444, (2013), 252-258 (doi.org/10.1016/j.memsci.2013.05.025)

[7] Sahner K., Wickles M., Schonauer D., Rettig F., Roosen A. and Moos R., Funktionskeramik, DKG-82, 13, (2005), 170-173

[8] Dutczak D., Justel T., Ronda C. and Meijerink A., Phys. Chem. Chem. Phys., 17, (2015), 15236 (doi.org/10.1039/c5cp01095k)

[9] Kurien S., Structural and electrical properties of certain nanocrystalline Aluminates, Ph.D. Thesis, Mahatma Gandhi University, Kottayam, India, (2005)

[10] Zawadzki M. and Wrzyszcz J., Materials Research Bulletin, 35(1), (2000), 109-114.

[11] Kou, L. and Selman J., Journal of Applied Electrochemistry, 30(12), (2000), 1433-1437.

[12] Kim J. W., Shin P. W., Lee M. J. and Lee S. J., Journal of Ceramic Processing Research, 7(2), (2000), 117-121.