PART III
MATERIAL SCIENCES

Aayushi International Interdisciplinary Research Journal (AIIRJ)
ISSN 2349-638x Impact Factor 4.574 Special Issue No. 26
UGC Approved Sr.No.64259 Website :- www.aiirjournal.com Email id:- aiirjpramod@gmail.com

SYNTHESIS OF CuO MODIFIED NANOCRYSTALLINE CR_2O_3

Chandak, P.M.[1], Raghuwanshi, F.C.[2] & Kapse, V.D.[3]

[1] Department of Physics, B.B. Arts, N.B. Commerce and B.P. Science College, Digras-445203, Maharashtra State, India

[2] Department of Physics, Vidyabharati Mahavidyalaya, Amravati-444602, Maharashtra State, India

[3] Department of Physics, Arts, Science and Commerce College, Chikhaldara-444807, Maharashtra State, India

ABSTRACT

Nanocrystalline Cr_2O_3 was successfully synthesized by co-precipitatipon method. Synthesized Cr_2O_3 was examined with the help of X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and energy dispersive X-ray (EDX) spectroscopy. Thick films of pure Cr_2O_3 were prepared by screen-printing technique. The surfaces of these films were modified by dipping them into a 0.01M aqueous solution of cupric chloride ($CuCl_2$) for different intervals of time, followed by firing at 550 ^{0}C for 30 min. The firing resulted in the oxidation of the $CuCl_2$ additive into. CuO i.e Cu0 modified Cr_2O_3 thick films.

Keywords: *Chromium Oxide; Nanocrystalline; X-ray diffraction; Thick films; CuO.*

INTRODUCTION

Chromium oxide (Cr_2O_3) is one of the widely studied transition metal oxide because of to its wide band gap (~3.3 eV) [1]. Cr_2O_3 is an intrinsic semiconductor at high temperature (>1000°C), whereas extrinsic p-type semiconductor at lower temperatures [2,3]. This kind of p-type wide band gap oxide semiconductors may be a good candidate for numerous applications.

Different preparation techniques for synthesis of Cr_2O_3 nanoparticles have been reported in the literature. For most of the techniques, highly explosive reactants, more complex processes, environmentally sensitive, more expensive reaction apparatus and higher calcination temperature is required. Moreover, most of methods produced a nonhomogenous particle size distribution, highly agglomerated and low yields. Among them, co-precipitation is considered as a cost effective and a less time consuming route. This technique can be used for the production of high purity nanocrystalline Cr_2O_3 on large scale.

So, the aim of the present work is to prepare nanocrystalline Cr_2O_3 by co-precipitation route to investigate the effect of CuO modification on structural and morphological behaviour of Cr_2O_3 based thick films fabricated by screen printing technique.

EXPERIMENTAL

2.1. Synthesis of nanocrystalline Cr_2O_3

In present work 25·50gm $Cu(NO_3)_3{\cdot}9H_2O$ was dissolved in 50 ml double distilled water and then kept on magnetic stirrer at 80^{0}C for 1 h, a transparent solution was formed. In this solution ammonia was added drop wise until a precipitated of pH 9 was formed. After ageing at room temperature for overnight the Chromium hydroxide was recovered by filteration, washing with double distilled water and drying at 110^{0}C for 24 h. Cr_2O_3 nanomaterial was obtained by calcinig Chromium hydroxide at 600^{0}C for 5 h. The synthesized Cr_2O_3 nanostructure product was used for further study.

2.2. Preparation of thick films

Thick films of Cr_2O_3 nanostructure were prepared by using screen printing technique. In this process, paste was formulated by mixing the synthesized Cr_2O_3 nanostructure with ethyl cellulose (a temporary binder) in mixture of three organic solvents. The ratio of inorganic to organic part was kept as 75:25 in formulating the pastes. The ready pastes were screen printed on a glass substrate in desired patterns. The films prepared were fired at 500^{0}C for 12 h. Prepared thick films termed as pure Cr_2O_3 thick films.

2.3. CuO modified Cr_2O_3 thick films

Surface of pure Cr_2O_3 thick films were modified by dipping them into 0.01M aqueous solution of $CuCl_2$ (99%ARgrade, Merck) for different intervals of time (3, 6, 9 min.). Dipped thick films were dried under IR lamp for 1 h. Dried thick films were fired at 500^{0}C for 30 min. The $CuCl_2$ dispersed on the film surface was oxidised to CuO in firing process and sensor elements with different mass % of CuO on the surface of Cr_2O_3

thick films were obtained. These surface modified thick films are termed as CuO modified Cr_2O_3 thick films.

RESULTS AND DISCUSSION

3.1. X-ray diffraction studies

Fig. 1 shows X-ray diffraction (XRD) patterns of synthesized Cr_2O_3 powder samples, the observed peaks are matching well with JCPDS data of Cr_2O_3. The characteristic peaks observed in the spectrum are higher in intensity which indicates that the as-synthesized samples are of good crystalline nature. The average crystallite size (D) was estimated from

the Debye–Scherrer's equation: $D = 0.9 \lambda / \beta \cos \theta$; where λ is the wavelength of X-rays (1.54056 Å), β is the FWHM, θ is the diffraction angle at which the full width at half maximum (FWHM) measured.

The average crystallite size of the synthesized Cr_2O_3 nanoparticles was measured from XRD patterns using Scherrer equation and was found to be ~23 nm.

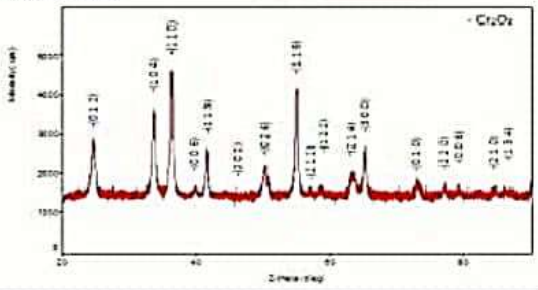
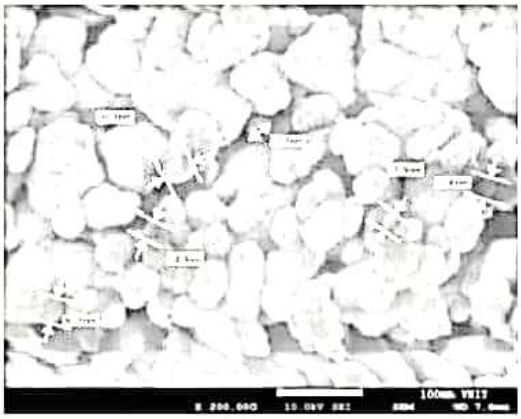


Fig. 1: X-ray diffraction pattern of Cr_2O_3 powder sample calcinated at 600 ^{0}C.

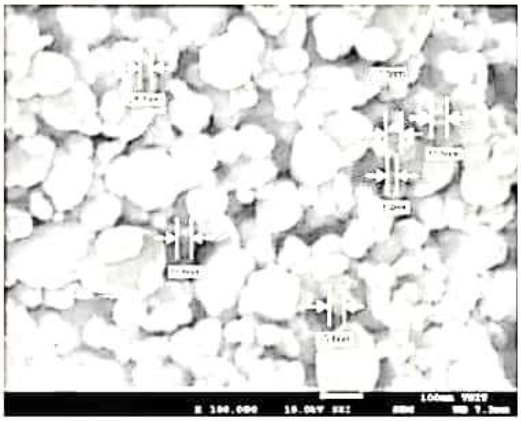
3.2. Scanning electron microscopic study

Fig. 2 (a-d) shows typical FE-SEM images of the pure and CuO modified Cr_2O_3 thick films prepared by screen printing technique. It can be seen from Fig. 2 (a) that the pure Cr_2O_3 nanoparticles were nearly uniform spherical shapes and very small particles in evidently dispersed without large agglomerates. Fig. 2 (b-d) depicts the microstructure of

CuO modified film for 3 min., 6 min. and 9 min., respectively, consist of particles with smaller size and shape associated with the Cr_2O_3 grains. Moreover, it can be seen that there is decrease in the agglomerations with the increase in the content of Cu. The average grain size of the fabricated thick films is observed to be in the range of 24 nm to 32 nm.

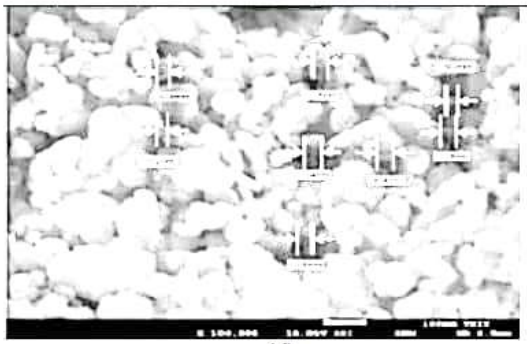


(a)



(b)

(c)



(d)

Fig. 2: FE-SEM microstructures for (a) Cr_2O_3 nanoparticles (b) CuO modified Cr_2O_3 thick films (3 min.) (c) CuO modified Cr_2O_3 thick films (6 min.) (d) CuO modified Cr_2O_3 thick films (9 min.).

CONCLUSIONS

In this paper, nanocrystalline Cr_2O_3 has been successfully prepared by co-precipitation method. The average crystallite size of as-prepared Cr_2O_3 has been estimated to be ~23 nm. The as-prepared nanoparticles are high purity, composition and produced with minimal agglomeration. The crystallite sizes calculated from XRD data show good agreement with those particle sizes obtained by FE-SEM. The morphological characterization of pure and CuO modified Cr_2O_3 thick films reveals that there is decrease in the agglomerations with the increase in the content of Cu.

REFERENCES

1. R.E. Kirby, E.L. Garwin, F.K. King, A.R. Nyaiesh, Surface properties of Cr_2O_3, J. Appl. Phys. 62 (1987) 1400.
2. L.N. Cojocaru, Electrical Properties of Non-Stoichiometric Cr_2O_3, Zeitschrift Für Phys. Chemie. 64 (1969) 255.
3. L.N. Cojocaru, T. Costea, I. Negoescu, Electrical Properties of Non-Stoichiometric Oxides of CrO_3-Cr_2O_3 Systems, Zeitschrift Für Phys. Chemie. 60 (1968) 152.