Preparation and Study of Finely Dispersed Magnetic Oxide in Polymer Matrix

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Preparation and Study of Finely Dispersed Magnetic Oxide in Polymer Matrix

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Abstract: The paper describes the synthesis and characterisation of a composite of Fe-Co with copolymer of aniline formaldehyde by chemical processing. The resulting material was studied by XRD, X-ray microradiography, TEM, Mossbauer effect and magnetisation measurements. Uniform dispersion of spherical shaped nano-sized particles of spinel magnetic oxide in polymer matrix is inferred.

1. INTRODUCTION

Composites based on nanoparticle sized magnetic materials have been of great interest to the researchers in the recent years. The potential of the nanocomposites for atomic engineering of the materials for tailoring and control of their magnetic properties and their possible applications in refrigeration [1] and high density information storage [2] are well recognised. The routes often employed for preparation of these materials are by co-sputtering of magnetic and non-magnetic species[1] and chemical processing [3]. The latter method is comparatively simpler to adopt and cost effective for production of bulk quantities. The paper reports the synthesis of a composite of iron and cobalt with copolymer of aniline formaldehyde and the results of its characterisation.

2. SYNTHESIS AND CHARACTERISATION

The composite material was synthesised by treating a solution of aniline hydrochloride and formaldehyde with aqueous solution of halides of iron and cobalt taken in the molar ratio 70:30. Addition of 10% sodium hydroxide yielded brownish yellow precipitate. Powder of the resulting composite material was obtained by filtering the precipitate followed by washing with distilled water and drying. The entire processing was carried out at room temperature. The density of composite powder was $\sim 1.5 g/cm^3$.

The average particle size of the powder was found to be around 10 μm by sedimentation method using MICRON PHOTO SIZER (Seishin, Japan). The spread of particle size ranged from submicron to 40μ. Debye-Scherrer patterns of powder sample were obtained by using CuKα radiation. ^{57}Fe Mossbauer spectrum was recorded in standard transmission geometry. The magnetisation measurements were carried out on a PAR 155 Vibrating Sample Magnetometer.

3. RESULTS AND DISCUSSION

The powder of the composite material was found to be attracted by a simple magnet. Attempts made to separate the magnetic component from the polymer present in the composite by application of high magnetic field did not succeed. The XRD pattern of the powder revealed the presence of two phases - one set of lines corresponding to a crystalline phase and the other a halo of the polymer. All the diffraction lines could be indexed and were found to belong to a spinel structure, in close agreement with JCPDS data for compounds like Fe_3O_4 and $CoFe_2O_4$. The only exception was simultaneous existence of halo at a Bragg angle of 10°. The diffraction lines were quite diffused in appearance. Since the crystalline phase, corresponding to magnetic material, and the polymer phase have widely different densities, attempt was made to separate them by floatation method. The powder was mixed with methanol intensely and allowed to settle for a few minutes. The liquid containing the finer particles was decanted and dried. The material settled at the bottom was separately collected. The two samples were examined by XRD. The diffraction patterns were identical with the one obtained earlier. No difference was observed in

the magnetic behaviour either. It showed that the magnetic phase consisted of fine particles evenly distributed in the polymer matrix. The diffused nature of the diffraction lines indicated that crystalline particles were essentially of dimensions less than half a micron.

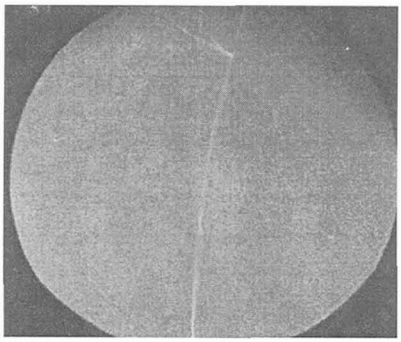

Fig. 1. X-ray microradiograph (x 5)

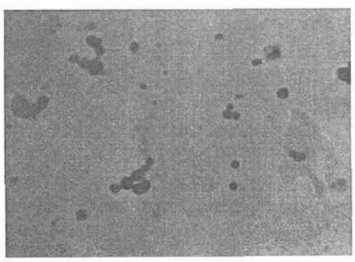

Fig. 2. TEM micrograph (x 10000)

The two phases present in the composite material have widely different densities. The distribution of fine particles of crystalline phase in a polymer matrix of such a sample is expected to be revealed by X-ray microradiography because of large difference in the densities of the two phases. A thin pellet (~0.5mm thick) of the composite powder was prepared by cold pressing. The microradiograph was obtained using MoKα radiation from a microfocus X-ray source with an effective focal spot of 50x50μm^2 on a fine grained X-ray film placed in contact with the pellet. The estimated spatial resolution in this method is better than 1 μm. The slightly increased blackening towards the top of the micrograph shown in figure 1 is due to inhomogenious intensity distribution of the source. The microradiograph has a contrast free appearance showing absence of any crystalline particles of size > 1μm. A final confirmation was obtained by taking a TEM micrograph of a spec of the powder (Fig. 2). The randomly distributed high density particles of spherical shape with dia around 50nm are clearly seen. The Mossbauer spectrum showed a six line pattern indicating presence of iron ions in a magnetically ordered environment. It could be resolved into two sextets corresponding to iron ions at tetrahedral and octahedral sites in the spinel structure. The isomer shift values of 0.32mm/sec and 0.42 mm/sec indicate presence of iron in trivalent state at both the sites. Zero quadrupole splitting values indicate high symmetry around the iron nuclei.

The magnetisation of the composite material was measured as a function of the applied magnetic field. The behaviour is typical showing saturating effect at high applied field of > 7kOe. Saturation magnetic moment value of 21emu/g is obtained.

4. CONCLUSIONS

The characterisation of the synthesised powder of iron and cobalt in copolymer of aniline formaldehyde by XRD, X-ray microradiography and TEM reveals the formulation of a composite material. It essentially consists of a crystalline phase of spherical shaped nanosized particles of magnetic oxide with spinel structure uniformly dispersed in the polymer matrix. The magnetic oxide can possibly be γ-Fe_2O_3/Co_3O_4/$CoFe_2O_4$ or a combination of them. Presence of Fe_3O_4 is ruled out since Mossbauer effect studies have shown that iron exists only in trivalent state. It is, however, not possible to differentiate among the various oxidic compounds from XRD data due to very close proximity of their cell constants and the diffused nature of the diffraction lines.

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