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STUDY OF DECOMPOSED MN(II) BAKELITE METAL MATRIX COMPOSITES THROUGH DLS AND AFM

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ABSTRACT

The increase in demand of newer and smarter materials has incited the investigation and development of several of metal matrix composites and its nano synthesis through low cost. Many characterization techniques have effectively fulfilled their role in this investigation. AFM (Atomic Force Microscopy) is an emerging instrumentation technique to measure three-dimensional topography information from the angstrom level to the micron scale with extraordinary resolution. The DLS also help in identification of sub-microparticles at even nano scale. This paper presents the study of carbonized sample of Mn (II) Bakelite MMC through DLS and AFM.

Keywords: Atomic force microscopy, Bakelite, diffraction light scattering, Manganese, metal matrix composites.

I. INTRODUCTION

The demand for fabrication of newer and smarter material with low cost is highly elevated.So is the demand of analysing them, with competent and effective tools of characterization. Researchers are always keen to know the in-depth nature of micro particles in order to develop functional materials.Various characterization techniques have been developed to study the micro particle nature of Metal Matrix Composites (MMCs)[1]. Over the past 20 years Scanning Probe Microscopes (SPM) has emerged as an essential material characterization technique in various fields [2,3and 4]. AFM is capable of delivering unique 3D topography information from the angstrom level to the micron scale with unprecedented resolution. Dynamic light scattering is also a non-invasive technique for calculating the size of particles and molecules in suspension sizing down to 1 nm in very less time. It is also known as Photon Correlation Spectroscopy.

The MMC andtheir nano forms are developed to serve in different fields requiring high strength,thermal and electrical conductivity. Several low cost methods of formation ofinnovative nano products are also explored[5, 6, and 7]. Thermal decomposition of composites at high temperatures in muffle furnace has emerged as very nominal cost methods to produce nanotubes microparticles and magnetic nanocomposites [8 9].In this context of progress ofMMC development manganese compositesare also developed and extensively studied [10-12]. Recently formation, properties and carbonized products of manganese metal bakelite composites arestudied through various characterization techniques [13-16]. The aim of this paper is to probe the micro particle or nano behaviour of carbonized samples of Mn(II) Bakelite through competent tools such as AFM and DLS and give the depth of information provided through these techniques in composite analysis.

II. EXPERIMENTAL

2.1 Materials

Phenol was supplied by Qualigens Fine Chemicals (India). Formaldehyde and Hydrochloric Acid was supplied by Fisher Scientific, Qualigens (India). Glacial acetic Acid was purchased from Central Drug House (P) Ltd (India). Manganese (II) Chloride tetrahydrate 98% ($MnCl_2.4H_2O$) was supplied by Sigma-Aldrich ACS reagent (India).Distilled water which was used to prepare composites was of chemically pure grade. Metal solutions were prepared by dissolving appropriate amount of its chloride salt in distilled water. The samples were decomposed using muffle furnace KLS 03/10 with T max 1000^{o}C.

2.2 Sample preparation

The composites were prepared by simple vortex method. The homogenous solution of matrix salt was prepared by adding 0.5gm and 2gm $MnCl_2.4H_2O$ in 5 ml distilled water. The monomers of bakelite reaction are further mixed to prepare MMC [14]. The lustrous pinkish red MMC of 0.5% Mn-(II)-Bakelite is mentioned as MB1 and 2% Mn-(II)-Bakelite composite is mentioned as MB2 respectively. The MB1 composite was decomposed at 750 ^{o}C and MB2 composite was decomposed at elevated temperature of 950^{o} C for 6 hours. The black carbonized product was obtained. It was then crushed to powdered state using pestle and mortar and characterized.

2.3 Characterization

Atomic force microscopy (AFM) was done usingAFM -XE 70, Park Systems - Korea in vibrating mode. Particle size was determined by Diffraction Light Scattering (DLS) Zetasizer Malvern, Nano ZS90 – UK.

III.RESULTS AND DISCUSSIONS

3.1 DLS Analysis

The Particle size analysis through DLS gave volume plot for MB1MMC decomposed at 750 ^{o}C with Z average 1176 d.nm. The volume plot of DLS for MB2MMC decomposed at 950 ^{o}C gave Z-average 768.7 d.nm. The results clearly illustrate that on increasing the decomposition temperature the particle size of prepared composite decreases as revealed by the Peak 1 values of Fig 3.1 and 3.2 respectively.The diameter and width of MB1 MMC appeared at 546.4 d nm and 98.4 nm. The diameter and width of MB2 MMC appeared at 504.9d.nm and 83.9nmrespectively. The result clearly reveals that on increasing decomposition temperature the composite show a decreasing trend of size and even nano size range is obtained through simple decomposition process at high temperatures.

Results

			Diam. (nm)	% Volume	Width (nm)
Z-Average (d.nm):	1176	Peak 1:	546.4	100.0	98.40
PdI:	0.921	Peak 2:	0.000	0.0	0.000
Intercept:	1.00	Peak 3:	0.000	0.0	0.000
Result quality :	Refer to quality report				

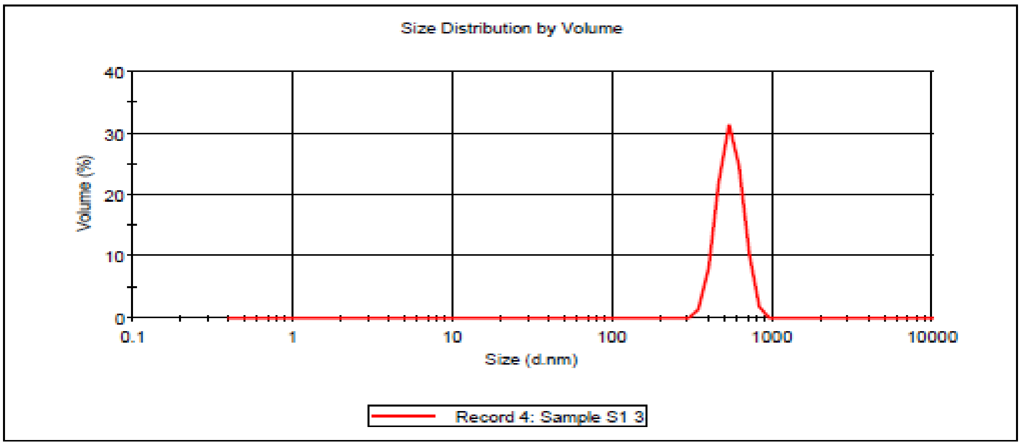

Fig 3.1Volume plot of MB1MMC

Results

			Diam. (nm)	% Volume	Width (nm)
Z-Average (d.nm):	768.7	Peak 1:	504.9	100.0	83.91
PdI:	0.505	Peak 2:	0.000	0.0	0.000
Intercept:	0.946	Peak 3:	0.000	0.0	0.000
Result quality :	Refer to quality report				

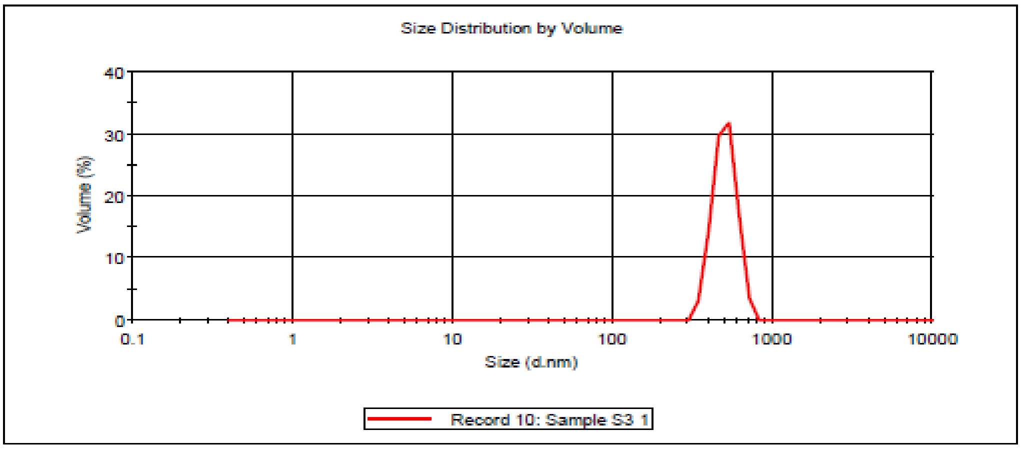

Fig. 3.2Volume plot of MB2MMC

3.2 AFM analysis

The Prepared thin films of composite sample of Mn(II)Bakelite composite were analyzed on 50 × 50 μm^2 area under AFM to study the particle size transformation at varying temperatures. The AFM profile images Fig.3.3 and 3.4 displays the film surface of MB1MMC decomposed at 750^o C and MB2 MMC decomposed at 950^{o}C respectively. In the Fig. 3.3 the profile image extends up to 50 μm with a smooth curve. The AFM profile image clearly indicates that at lower decomposition temperatures the particle height at points 10.45,20.31 and 38.84 were 7.154, 7.34 and 6.148 respectively for MB1MMC. It indicates almost globular shape of decomposed

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particle. In Fig.3.4 the profile image is irregular indicating the surface to be rougher and particle to be more proximate. The curve is extended till 30 µm indicating decreasing trend of particle size. At Points 4.12,13.43, 25.88 the height of particle were 1.27, 0.684,1.733 µm. The average roughness values Ra is1.07 and 0.280µm for MB1MMC and MB2MMC respectively[16].Theresult are comparable with DLS as on increasing temperature the size of particle decreasesand metal proximity within MMC increases[16].This behavior indicated inculcation of new properties in MMC after decomposition which can be useful in microelectronic devices [17].

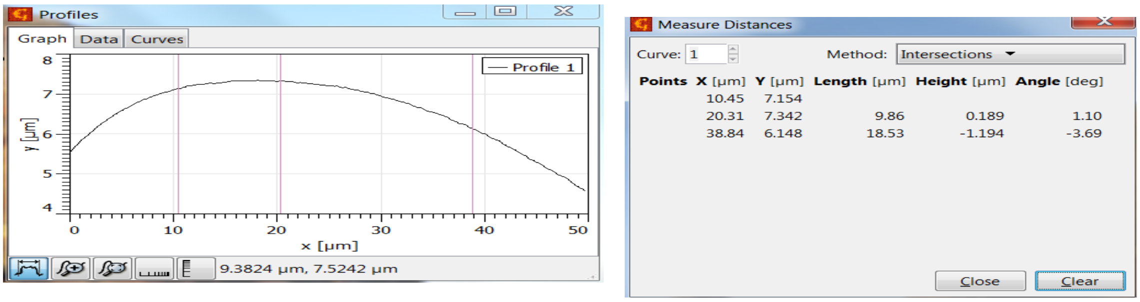

Fig 3.3 AFM profile of MB1MMC

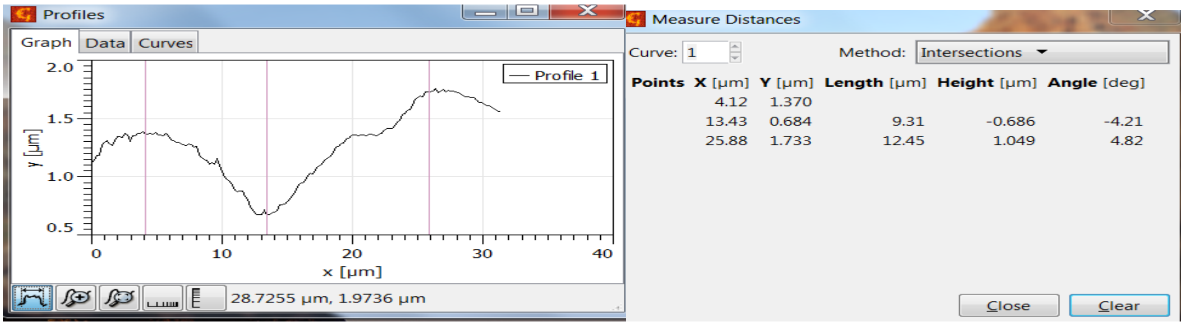

Fig. 3.4AFM profile of MB2MMC

IV. CONCLUSION

The AFM and DLS technique is effective in studying the three dimensional micro details of particles, The characterization techniques effectively explained the decreasing trend upto nanometers in particle size of Mn(II) Bakelite composited with variable concentration under high decomposition. The reduction in size of MMC particle at higher temperature leads to increase in proximity of metal matrix. This attribute can be used in preparing microelectronic devices.

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REFERENCES

[1]. D. Brandon, W. D. Kaplan, microstructural characterization of materials (John wiley & Sons Ltd, England, 2008)

[2]. G. Binning, C.F. Quate, Ch. Gerber, Atomic force microscope. Phys. Rev. Lett., 56(9), 1986, 930-934.

[3]. S. Dror, *scanning force microscopy: with applications to electric, magnetic and atomic forces*(Oxford University Press, 1994).

[4]. D. Bonnell, *scanning probe microscopy and spectroscopy*, (Wiley-VCH, 2001).

[5]. K. Bratek, H. Marsh, Carbonizations under pressure of chloroform soluble material of coking vitrinite.Fuel, 59, 1980, 339−342.

[6]. M. A Forrest,H. Marsh, The carbonization of blends of pitches and resins to produce anisotropic carbonand the effects of pressure, Journal of Material Science, 18, 1983, 991−997.

[7]. P. D Green, I. A. S Edwards, H Marsh, K. M Thomas, and R. F Watson, Coal thermoplasticity and cokestructure as related to gasification. Effect of inorganic additives on high pressure dilatometric properties andreactivity towards hydrogen, Fuel, 67, 1988, 389−395.

[8]. B.Tiwari, I. P. Tripathi, and Sanjay Saxena, Synthesis, characterisation and study of properties of CNTsprepared by egg protein and metal salt, Indian Journal of Applied Research, 3(2),2013, 14-16.

[9]. Sujittra Daengsaku, Charusporn Mongkolkachit, Chunpen Thomas., A simple thermal decompositionsynthesis, magnetic properties,and cytotoxic

[10]. D. Al-Khafagiy, F.L Farhan Lafta Rashid, A. Hashim, and Md. A Mahdi, Preparation and characterization of (PS-CoCl2) and (PS-MnCl2) Composites. Elixir Applied Chemistry, Vol. 48, 2012, 9585-9587.

[11]. P. Jia, M. Zhang,C. Bo, and L. Hu, and Y. Zhou, The plasticizing effect of properties of manganous chloride and glycerin on poly (vinyl alcohol). Polish Journal of Chemical Technology,17(2), 2015,29-33.

[12]. Šljukić, B., Stojković, I., Cvijetićanin, N. and G. Ćirić-Marjanović, Hydrogen peroxide sensing at MnO2/carbonized nanostructured polyaniline electrode. Russian Journal of Physical Chemistry A, 85(13), 2011, 2406-2409.

[13]. R. Raj, and B. Tiwari, Synthesis and Structural Study of Manganese (II) Bakelite Composites by X-Ray Diffraction. International Journal of Metallurgical & Materials Science and Engineering, 3(4), 2013,23-26.

[14]. R. Raj, and B. Tiwari., Thermal analysis of Manganese (ii) Bakelite composites. International Journal of Mechanical Engineering and Technology (IJMET), 6(1), 2015, 104-110.

[15]. R. Raj, and B. Tiwari,. Spectroscopic Investigation of Manganese (II) Bakelite Composite., ICRTIET-2014 Conference Proceeding, Divya Jyoti College of Engineering & Technology, Modinagar ,2014.

[16]. R. Raj, and B. Tiwari, Study of microstructure of manganese (ii) bakelite composite after carbonization, Internatrional Journal Of Science Resereach And Methodology, 5(3),2017,23-31.

[17]. N.L., Singh, S., Shah, A. Qureshi, A. Tripathi, F. Singh, D.K. Avasthi, and P. M. Raole, Effect of ion beam irradiation on metal particle doped polymer composites. Bulletin of Material Science, 34(1), 2011, pp. 81–88