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Recent Developments in the Processing and Testing of Nanocomposites: A Review

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---ABSTRACT---
This review paper discusses on the recent developments in the fabrication methods of nano-composites (NC) and subsequent testing of their properties such as mechanical, thermal, tribological, wear, morphological, chemical and dielectric. The conducted review reveals a significant amount of work on the fabrication techniques and testing of three different Nanocomposites which are Polymer-Matrix, Metal-Matrix and Ceramic-Matrix based. Coupled with the use of Carbon Nano Tubes (CNTs) as reinforcement phases, NCs have enhanced unique properties which make them materials of the future.

Keywords: *nanocomposites (NCs), nanomaterials, filler phases, process parameters*

I. Introduction

Nanocomposites are the recently developed materials which have unique processing technologies with different property combinations that are not found in any conventional composites [1]. In Nanocomposites, at least one of the phases shows dimensions in nanometer. Nanocomposites can be classified based on the matrix materials employed in three different categories:

- Ceramic Matrix Nanocomposites (CMNC) employing Al_2O_3, SiC and SiN as matrix materials
- Metal Matrix Nanocomposites (MMNC) employing Al, Mg, Pb, Sn, W and Fe as matrix materials
- Polymer Matrix Nanocomposites (PMNC) employing mostly vinyl polymers and polyolefins as matrix materials

With the advent of carbon nanotubes (CNTs) in early nineties, they are being used on a wide scale to reinforce the matrices in nanocomposites to exhibit unique mechanical, thermal and electrical properties. Nanocomposites reinforced with CNTs have been extensively researched for the last two decades and there has been a continuous increase in the number of publications on the subject [1].

II. Processing Technologies

The processing technologies of the different NCs are summarized below:
Ceramic Matrix Nanocomposites (CMNC): The most common methodologies used are conventional powder method, polymer precursor route, spray pyrolysis, chemical vapour deposition(CVD) and physical vapour deposition (PVD) and chemical methods, which include the sol-gel process, colloidal and precipitation approaches [1].
Metal Matrix Nanocomposites (MMNC): There are different manufacturing techniques for reinforcing alloy such as spray decomposition, liquid metal infiltration, powder metallurgy, squeeze casting, mechanical alloying and compocasting [3].

Polymer Matrix Nanocomposites (PMNC): The common methods to produce PMNC are intercalation (Figure-1) of the polymer or pre-polymer from solution, In-situ intercalative polymerization, melt intercalation, direct mixture of polymer and particulates, in-situ polymerization and sol-gel process [1].

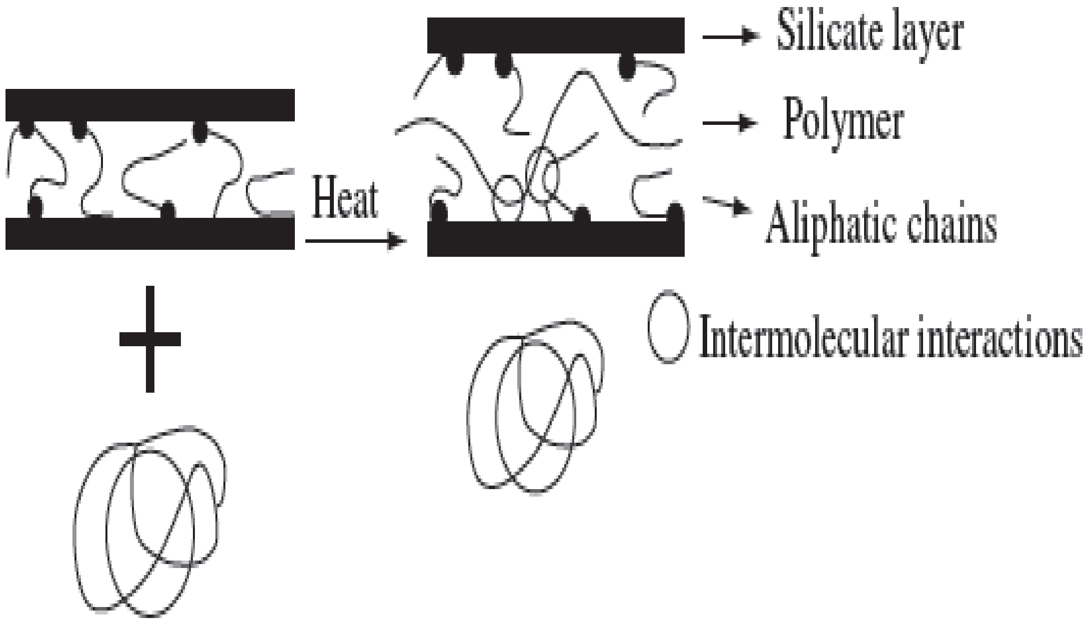

Figure 1: Melt intercalation synthesis of polymer/clay nanocomposites [1]

Two important processing methods to form nanocomposite layers on the surface of the substrates are PVD (Physical Vapor Deposition) and CVD (Chemical Vapor Deposition). Both the techniques are shown below in Figure 2 and Figure 3.

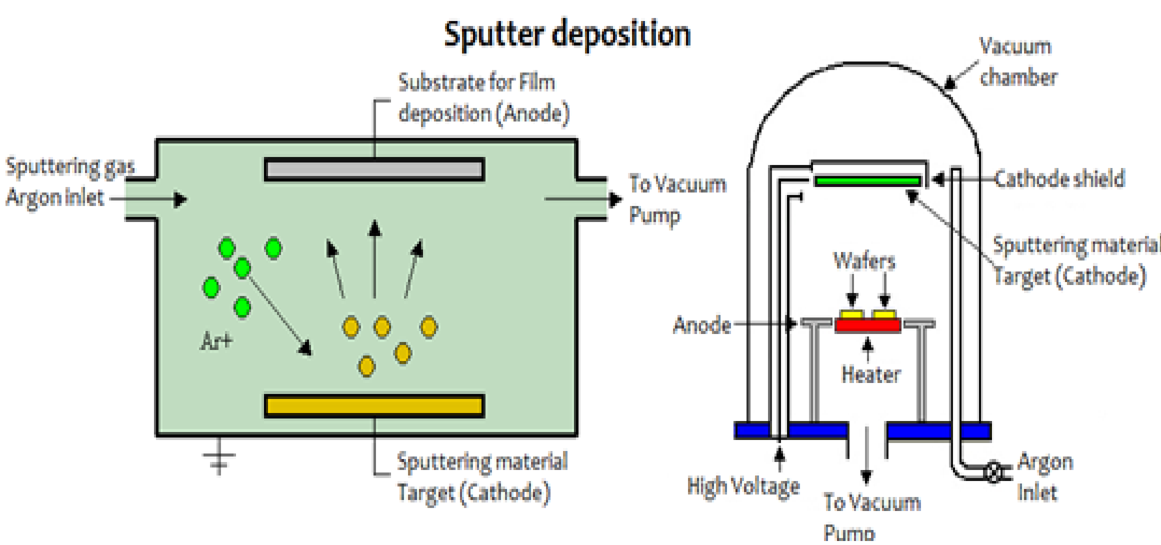

Figure 2 PVD Method

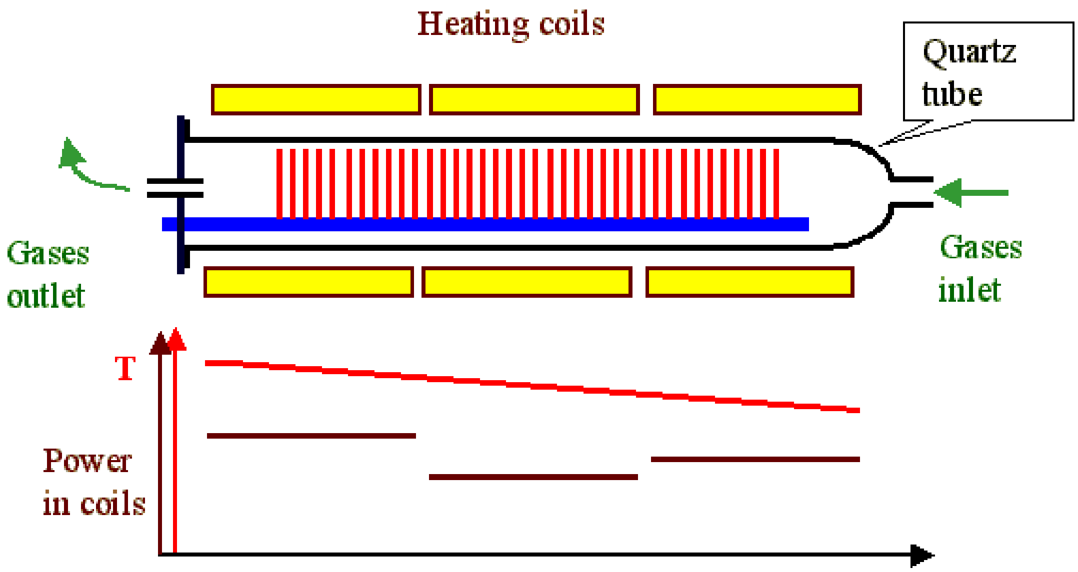

Figure 3 CVD Method

III. TESTING METHODS

The testing methods employed for NCs include the tests for compressive and tensile strength, hardness, density, porosity, ductility, fatigue strength, energy storage efficiency, thermal stability, heat resistance, modulus of elasticity, coefficient of friction, wear resistance, solid-particle erosion, hardness, diffusion properties, thermochemical properties, microstructures, toughness, electrical and thermal conductivity, dielectric properties, tribological behavious, cohesion and handling properties among others. Different authors have investigated different properties and established their usage in practical applications.

IV. RECENT DEVELOPMENTS IN PROCESSING AND TESTING

Camargo et al [1] have done an exhaustive study of the synthesis, structure and properties of NCs by reviewing the work on NCs upto the year 2009. They have done an overview of the three types of NCs underlining the need for these materials, their processing methods and the results on structure, properties and potential applications, perspectives including need for such materials in future space mission and other interesting applications together with market and safety aspects. Nassar and Nassar [2] fabricated pure aluminum nanocomposite reinforced with nano titanium dioxide by powder metallurgy method and measured properties like tensile strength, hardness, and density which exhibited an increase in porosity and tensile strength of composites with an increase in volume fraction of nanoparticles; however ductility of aluminum reduced. Senthilkumar et al. [3] fabricated powder metallurgy based aluminium alloy with reinforcements of micro and nano-sized alumina particles and subjected to low cycle fatigue test with a constant strain rate which exhibited enhanced mechanical properties when compared to the micron sized alumina reinforced composites. Abdalla et al. [4] explored NC films which are being researched to develop the energy-storage efficiency of electrostatic capacitors. It was found that polymer purity, nano-particles size, and film morphology drastically affect the electrostatic efficiency of the dielectric material that form an insulating film between conductive electrodes of a capacitor. This in turn affects the energy storage performance of the capacitor. Zhao et al. [5] fabricated Polyimide (PI)/nano-SiO_2 composites via a novel in-situ polymerization and investigated the microstructure, thermal properties, mechanical performance and tribological behaviors of these composites. The testing revealed that compared with pure PI, thermal stability and heat resistance get increased with the addition of nano-SiO_2. Compressive strength and modulus of composite with 5 wt% nano-SiO_2 increase by 42.6 and 45.2%, respectively. The coefficient of friction (COF) of composite with 5 wt% nano-SiO_2 decrease by 6.8% owing to the thick and uniform transfer films. The tests also revealed that excess nano-SiO_2 could adversely affect the COF of PI/nano-SiO_2 composite. Besides, wear resistance deteriorates obviously since transfer film exfoliates easily and nano-SiO_2 aggregates on the surface of transfer films.

Shirazia et al. [6] investigated the effects of graphite (Gr) content on the dry sliding wear, solid-particle erosion and corrosive wear of Al6061 based hybrid NCs fabricated via powder metallurgy route containing SiC nano-particles and micron-sized Gr particles. It was found that the hardness of these composites decreased almost linearly with their Gr content, accompanied with decreased erosion resistance at 90° particle impingement. The Al6061/2SiC/2Gr nano-composite exhibited the minimum wear rate and friction coefficient. Tavakol et al. [7] investigated the mechanical properties of nano-composites produced by shock wave sintering of aluminum and silicon carbide nano-powders using Molecular Dynamics (MD) simulations and concluded that increasing the shock pressure leads to an enhancement in the mechanical properties as a result of the formation of fiber reinforced nano-composites. The initial pressure exerted on the nano-particles, however, results in weakening of the sintered nano-composite. It was also found that an appropriate cooling rate can result in the activation of the diffusion mechanism after the shock wave passage which is helpful in increasing the bonding strength of nano-particles. Sui et al. [8] investigated on the thermochemical properties and microstructures of layered aluminum (Al) and iron oxide (Fe_2O_3) through thermogravimetric analysis (TGA), differential scanning calorimetry (DSC) and scanning electron microscopy (SEM) for developing a new type of metastable intermolecular composites (MIC). SEM images demonstrated a layered structure with the intimate contact between Al and Fe_2O_3. It was found that the decomposition of Fe_2O_3 nanoparticles, diffusion path of oxygen and interfacial contact between Al and Fe_2O_3 layers determined the energetic properties of layered thermite composites.

Yin et al. [9] incorporated SiC nano-powders and nano-wires with excellent toughness as well as high strength in $Mg_{2.16}(Si_{0.3}Sn_{0.7})_{0.98}Sb_{0.02}$ and characterized the effect of the morphology and phase fraction of nano-SiC additives on the thermoelectric (TE) as well as mechanical properties of the composite. Xua et al. [10] investigated the nano-rubber toughening effects on neat epoxy and epoxy/carbon fiber composites under a range of temperatures from −80 °C to 50 °C and studied the influence of temperature on the delamination fracture toughness of epoxy/carbon fiber composites with nano-rubber filled epoxy matrices compared to those with neat epoxy matrices mirrored the same trend of nano-rubber particles on epoxy. Akmal et al. [11] synthesized NiTi-HA composites using powder metallurgy route and observed new phases such as $NiTi_2$, Ni_3Ti and Ni_4Ti_3 for sintered composites. It was found that mechanical properties enhanced with the increasing content of HA and new phases. No martensitic transformation was observed for all composites by DSC analysis. Nayaka et al. [12] worked on Nano TiO_2 particles which are one of the promising inorganic nano fillers used in polymer matrix composites to enhance the mechanical properties and investigated the addition of nano

TiO_2 filler on water sorption, residual strength and thermal properties of glass fiber reinforced polymer (GFRP) composites. The results revealed that addition of 0.1 wt% TiO_2 reduced water diffusion coefficient by 9%, improved residual flexural strength by 19% and residual interlaminar shear strength by 18% among all the nano TiO_2 modified composites. The improvement of mechanical properties in hydrothermal environment creates opportunity and reliability to be used in different engineering applications. Pitchan et al. [13] investigated Polyetherimide (PEI) reinforced with multi-walled carbon nanotube (MWCNT) using novel melt blending technique modifying the surface of MWCNTs by acid treatment as well as by plasma treatment. PEI NCs with 2 wt% treated MWCNT showed about 15% improvement in mechanical properties when compared to unfilled PEI. There was a significant improvement in mechanical properties and thermal properties for surface functionalized MWCNT reinforced.

Kundalwal and Kumar [14] focused on the mechanical properties and stress transfer behavior of multiscale composites containing nano- and micro-scale reinforcements. They found out that the Orientation of CNTs has significant influence on stress transfer behavior and CNT-epoxy interface weakening significantly affects the radial stress. Javier et al. [15] studied the Iron oxyhydroxide and graphite oxide (GO) composites with either 1 or 10% of GO modified with Ag using a single step in-situ procedure. Barari [16] investigated the plant-derived cellulose nano-fibers (CNFs)/bio-based epoxy composites manufactured using the Liquid Composite Molding (LCM) process and found enhanced tensile strength of the composites reinforced by silyliated CNFs. The tests also showed that composites reinforced by cellulose fibers have lower coefficient of friction and wear. Pedrazzini et al. [17] reported a study of the structure-processing property relationships in a high strength $Al_{93}Fe_3Cr_2Ti_2$ nano-quasicrystalline alloy and composites containing 10 and 20 vol% ductilising pure Al fibres and observed an yield strength of 544 MPa substantiated from a combination of solid solution strengthening, work hardening, precipitation hardening and Hall-Petch grainsize dependent effects. The processing condition employed in this study provided micron-sized grains with a strong [111] preferential orientation along the extrusion direction and a bimodal size distribution of the icosahedralnano-quasicrystalline precipitates. Both were deemed to be a significant contributor to the high yield strength observed. The addition of pure Al fibres was found to decrease the yield strength linearly with increasing Al content, and to augment the ductility of the composites. Saba et al. [18] prepared epoxy based hybrid nanocomposites by dispersing the nano filler (nano OPEFB filler, MMT, OMMT) at 3% loading through high speed mechanical stirrer followed by wet hand lay-up technique. Tests for mechanical and morphology properties of hybrid nanocomposites were carried out which concluded that nano OPEFB hybrid composites will provide alternative constructional materials respect to steel, bricks and cement for Malaysia. Tang et al. [19] attempted to improve the thermal conductivity of the form–stable phase change materials (FSPCM) by adding Nano–Al_2O_3 and nano–graphite. Analysis through Scanning electronic microscope, Fourier transformation infrared spectroscope and X-ray diffractometer revealed that when the mass fraction of nano–powder additives is 12%, the thermal conductivities increase by 95% (NAO) and 121% (NG) at 30 °C which implies that the FSPCM possess a potential application for thermal energy storage.

Kim et al. [20] prepared the core–shell polypyrrole@polyimde (PPy@PI) particles and investigated on their dielectric properties for all-organic dielectric composites which showed that at the 15 wt% PPy in PPy@PI composite, the dielectric permittivity of over 100 was obtained with the electrical conductivity of 10^{-8} S/cm. High fractional use of PPy in dielectric layer as over 30 wt% PPy gave the ultra-high dielectric permittivity of 487. Czechowsk et al. [21] combined titanium doped nano-hydroxyapatite with chitosan and calcium sulfate to obtain cement-type materials with improved cohesion and handling properties. It was proven that the presence of nano-TiHA influenced setting, hardening, microstructure and bioactivity of the cement samples. In the developed composites chitosan played a role of the cohesion promotor and created homogenous organic layers on the materials surfaces. It was suggested that the resorption rate of materials based on calcium sulfate can be optimized by addition of both titanium doped nano-hydroxyapatite and chitosan. Porwal et al. [22] produced Graphene Nano-Sheets (GNS) with different lateral sizes (193, 373 and 1070 nm) using liquid phase exfoliation and controlled centrifugation. The investigations showed that the fracture toughness, hardness and elastic modulus of the composites decreased with increasing GNS size. Microstructural investigation of the composites suggested that their mechanical properties were directly related to the average number of GNS present per unit volume in composite and the relative size of GNS to alumina grains. The smaller size GNS produced various toughening mechanisms, while the larger size GNS, with similar size to the alumina grains, resulted in grain boundary sliding.

IV. CONCLUSION

The survey of the contributions by different authors shows a wide spectrum of nanocomposites being investigated for mechanical, morphological, thermal, electrical and microstructural properties. The authors have suggested different ways to improve the properties of nanocomposites so as to elevate their utility in various applications. This brings out the possibility of enhanced utilization of nanocomposites in the near future.

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