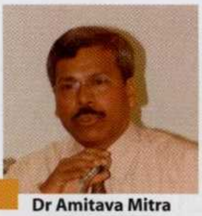
Nanostructured Materials Research at NML

— Dr Amitava Mitra, Dr Arvind Sinha, Dr Suman Kumari Mishra & Dr K L Sahoo



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NML is coordinating the CSIR Network Project on 'Nanostructured Advanced Materials'. Eight other participating CSIR labs are AMPRE, Bhopal; CEERI, Pilani; CGCRI, Kolkata; CMERI, Durgapur; IMMT, Bhubaneswar; NAL, Bangalore; NCL, Pune; NPL, New Delhi. The project has been divided into four modules: (i) Biomaterials and Processes (ii) Ceramics, Composite and Hard coatings (iii) Magnetic Materials and (iv) Structural Materials. The major emphasis on the project has been given on the development of newer process, product and devices.

(i) Biomaterials & Processes: Akin to a matrix mediated biomineralisation process, nanoparticles of hydroxyapatite (HA) has been synthesized by a biomimetic route, using a hydrophilic polymer, namely poly (vinyl alcohol) (PVA) as a matrix. A simple surface chemistry modification could translate these HA nanoparticles into a self setting inject able gel. This nanocomposites gel can be used as an inject able bone graft. The critical performance parameters for the inject able graft like water stability, micro and macroporosity, setting time and mechanical strength along with inject ability have also been studied as a function of solid – liquid ratio and surface treatment. Bioactivity test at Srichitra Trinual Institute of Biomedical Science confirms the biocompatability of the product. The industrial viability of the product is now jointly carried out with IFGL, Kolkata.

Our study on ferrofluid is focused on the single step in situ biomimetic synthesis of superparamagnetic iron oxide nanoparticles (SPION) using synthetic polymers, biopolymers and a combination of both, in an aqueous environment. This gives a high degree of control in shape, size and morphology and allows synthesis in bulk. Till now we have been able to synthesize highly stable aqueous ferrofluids in water with controlled particle size (<10nm). Experiments are on to make nanofluids suitable for hyperthermia. Studies have been initiated with Delhi University and Bhabha Atomic Research Center.

A Process has been developed for bioleaching of low grade sphalerite ((Zn,Fe)S, a chief ore of Zn) tailings using microbes in 2L bio-reactor with yield of 98 per cent purity and 55-60nm size ZnO. Industry is being contacted to go for larger size bioreactor.

Fig.1: Injection of HA nanoparticles(a), water stability of self-setting nanoHA exhibiting bubble formation due to porous structure (b) and porosity confirmed by SEM studies (c).

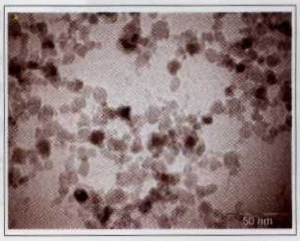

Fig-2 (a). TEM image ferrofluid

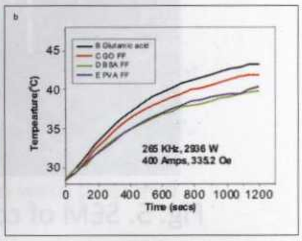

Fig -2 (b) Radio frequency heating of ferrofluid

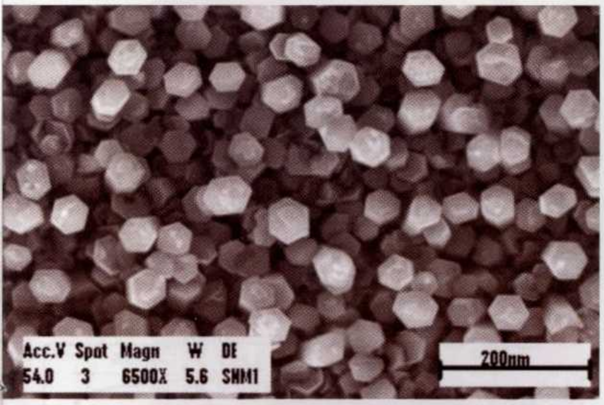

Fig-3: Zno nanoparticles obtained through bio-leaching using microbes

(ii) Ceramics, Composite and Hard Coatings: A process has been developed for multilayer Si-C-N coating on steel substrate using single SiC-C target by suitable alteration of plasma parameter. Three layers having hard-soft-hard have been deposited with an overall hardness 25GPa and modulus of elasticity as 343. The percentage of elastic recovery is 54 percent. The films did not show fracture till 1000gf load, rather substrate starts yielding at those loads and film failure took place mainly at higher loads due to plastic deformation of substrate indicating both hard and tough nature of film.

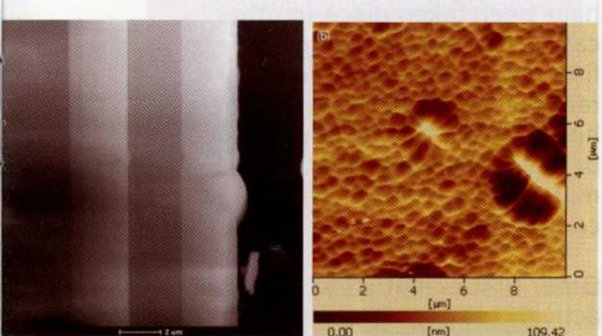

Fig.-4: Three layered Si-C-N with hard-soft-hard phases (a) and the surface topography of top layer (b).

8 Mol% Yttria stabilized Zirconia (YSZ) and $La_2Zr_2O_7$ (LZ) nano powders are made by solution combustion process. YSZ powder was granulated by spray dryer and plasma sprayed on bond coated substrates. Study of grain growth behaviour of TBC coating (YSZ and LZ) and plasma spry deposition TBC coating on turbine blade is in progress.

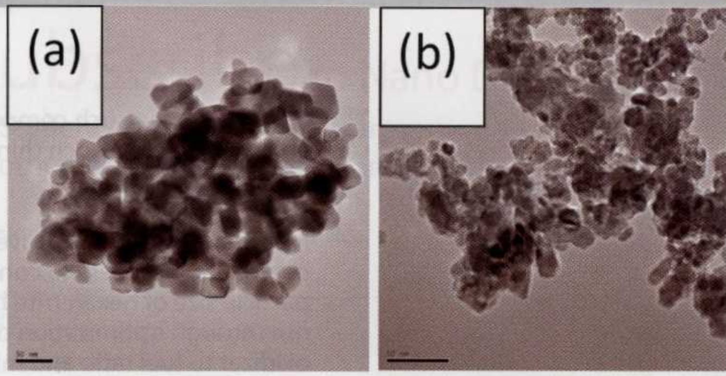

Fig.-5: TEM micrographs of nano thermal barrier coating materials (a) YSZ and (b) LZ

(iii) Magnetic Materials: A Giant magneto-impedance (GMI) based sensing device has been prepared using nanostructured magnetic wires of 120 m diameter prepared in the laboratory by in-water quenching technique. The GMI sensing device has been taken to a petrochemical industry and tested for the detection of carburization in Johnson screen used in its catalytic converter reactor unit (CCRU). Development of sitewarthy sensing device for structural health monitoring is in progress in collaboration with the R&D Division of Indian Oil Company Limited.

NML carried out activities on ferromagnetic shape memory alloys and found more than 300ppm Magnetic Field Induced Strain (MFIS) in NiMnGa based bulk alloys produced in the form of ribbon by melt spinning technique. A thin film has been

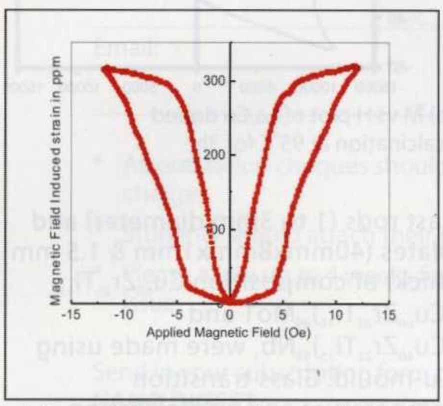

Fig-7: Magnetic field induced strain in rapidly solidified and annealed crystalline NiMnGa alloy

Fig.- 6: Catalytic reactor unit of oil Industry where NML team carried out structural health monitoring using GMI based sensing device prepared using such composition and charaterisation of such thin film is under progress.

NML developed microwave assisted combustion process to control the particle size of hexaferrite below 200 nm through optimization of the oxidizer to fuel ratio and microwave exposure time. Besides, it is observed that the developed process avoids the formation of other secondary phases, thereby the prepared powders using La and Co as doping elements exhibit coercivity and magnetization values of ~7800 Oe and ~3300 G, respectively at room temperature. The coercivity value is much superior then the powder available with some of the leading producers of India. Such good value obtained due to increase of the magnetocrystalline anisotropy constant value caused by smaller particle size of the prepared powder than the theoretically predicted single domain particle size (~840 nm).

(iv) Structural Materials: Suction

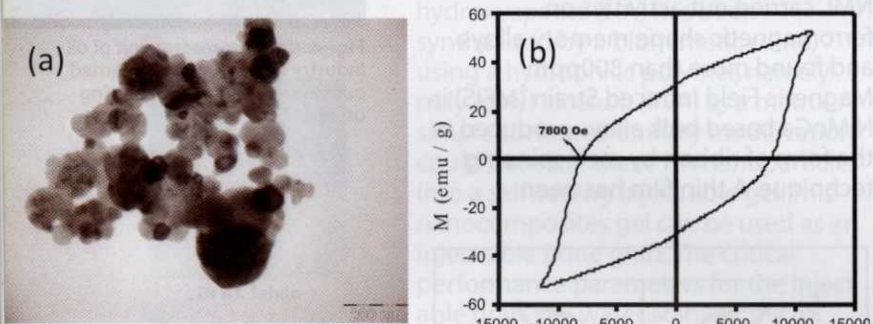

Fig.-8: (a)TEM image and (b) M vs H plot of La,Co doped $SrFe_{12}O_{19}$ powder after calcination at 95°C for 3h

cast rods (1 to 3mm diameter) and plates (40mmx8mmx1mm & 1.5 mm thick) of composition $Cu_{60}Zr_{25}Ti_{15}$, $(Cu_{60}Zr_{25}Ti_{15})_{99}$Mo1 and $(Cu_{60}Zr_{25}Ti_{15})_{99}Nb_1$ were made using Cu-mould. Glass transition temperature and crystallization behaviour were evaluated using differential scanning calorimeter.

Maximum compressive fracture strength of 2200MPa and plastic strain 2.75 per cent (elongation 5 per cent) were achieved in $(Cu_{60}Zr_{25}Ti_{15})_{99}Nb_1$ alloy. Efforts are now being made to develop gear using the bulk metallic glass.

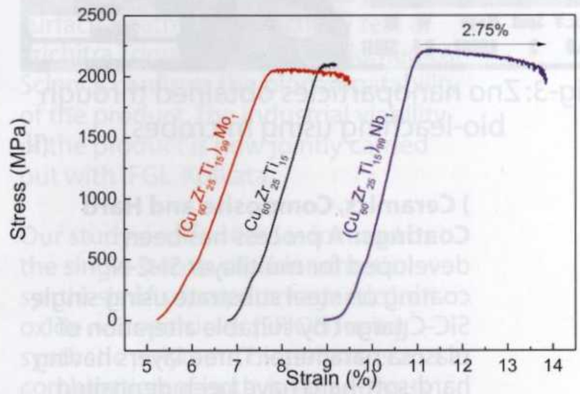

Fig.-9: Compressive Stress strain curve of developed Cu- based bulk metallic glass

Challenges

The product, process and device oriented activity on nanostructured materials have been carried out at National Metallurgical Laboratory. In some cases industry collaborations have been taken place. In other cases attempts are being made to find out suitable partners. Now the real challenges to the researchers how to make their product, process or device using nanostructured materials internationally competitive.

Other NML Scientists who are actively involved in the CSIR network project on Nanostructured Advanced Materials are: Dr S Nayar, Dr AK Pramanick, Abhilash, Dr Lokesh Pathak, Dr AK Panda, Dr R Roy,
Dr RK Sahu, Dr J Chakrabarty, Dr S Palit Sagar, Dr M Bhattacharya and Dr A Kailath. ■