Workshop PCCDI47

New advanced nanocomposites. Technological developments and applications

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Abstract Book

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Polymer nanocomposites for ballistic protection

R. Petre[1], N. Petrea[1], F. Neatu[2]
1 - Scientific Research Centre for CBRN Defence and Ecology, Bucharest, Romania
2 - National Institute of Material Physics, Magurele, Romania

In the recent years the fibre-reinforced polymer materials have been used successfully in a wide range of applications where can significantly improve the characteristics of protection. In this respect, our study aimed the possibility of using some novel composites of polymers and fibre-reinforced polymer nanocomposite materials for military application.
The polymer matrix is polyurea and the considered fibres are functionalized multi-wall carbon nanotubes. The reinforcement of a polymeric matrix with high strength and modulus fibres using the viscoelastic displacement of the matrix under stress transfers the load to the fibre; this resulting in a high strength and a high modulus composite material.
The aim was to produce a two-phase material in which the primary phase that determines the stiffness, is fibre-shaped and is well dispersed and bonded and protected by a secondary phase, the polymeric matrix. The fibre-reinforced polymer composite materials mechanical and physical properties are clearly determined by their constituent properties and by the micro-structural configuration. While the fibres are mainly responsible for strength and stiffness, the polymeric matrix contributes to stress transfer and provides microclimate protection.
The progress beyond the state of the art consists in producing of fibre-reinforced polymer materials with excellent mechanical resistance. Different types of reinforcement fibres and different thickness can be expressly used for specialized applications for the following purposes: improving ballistic protection of existing equipments; reducing blunt trauma for protection equipments; additional shield for armoured transport vehicles.
The performed determinations have confirmed a substantial improvement in the equipment protection capability.

Triboelectric devices/technologies based on electrospun fibrous materials

A. Evanghelidis, E. Matei, M. Beregoi, M. Enculescu, I. Enculescu
National Institute of Material Physics, Magurele, Romania

Triboelectric nanogeneration (TENG) devices are used to collect and transform mechanical energy from various environmental sources, energy that would otherwise remain unused, into a useful electrical energy source, for powering other devices or for sensing applications. To achieve this, TENGs are usually integrated into objects for everyday wear and use, or otherwise embedded in flexible and/or compliant hybrid structures which allow the triboelectric process to take place following a large variety of naturally occuring movement patterns, from bipedal walking to oceanic waves.

The use of nanostructured materials is crucial for achieving a high level of compliance and integration, and for enhancing the conversion efficiency of such devices to usable levels. The micro/nanofibrous mats that can be readily created in large amounts through electrospinning provide ample flexibility and multiple avenues for functionalization and customization, making them good candidates for the role of TENG substrates.

A triboelectric device in which the both the electrodes and the generating materials are based on electrospun fibers is presented. The fabrication process, as well as device performances, are discussed and potential applications are suggested.

Magneto resistors based on hybrid magnetorheological suspensions

M. Bunoiu, I. Bica, I. Mălăescu, G. Pascu
West University of Timisoara, No.3, Bd. V. Pârvan, 300223-Timișoara, Romania
email: madalin.bunoiu@e-uvt.ro

The focus of the project is on the advances made in the research area regarding the development of magnetorheological elastomers, membranes, and magnetic active fabrics for various applications. During the entire period, a multitude of new materials have been obtained and characterized, and their behaviour in external electric and magnetic fields has been evaluated, as well.

The magneto resistors (MRs) realized as part of this research are based on hybrid magnetorheological suspensions (hMRSs) and copper electrodes. The hMRSs are manufactured using silicone oil (SO), iron microparticles (Fe) and nano-microfibers (GB) consisting in cotton fibres (gauze bandage). 2.50 cm^3 mixtures of Fe with SO, in which the volume fraction Φ_{Fe} is 20% vol., 30% vol. and 40% vol., have been injected in GBs and then placed between the copper electrodes. Three types of MRs have thus been obtained, each having the dimensions 30x30x2 mm^3.

By placing the MRs between the magnetic poles of a direct current electromagnet, using an 8846A multimeter, the electrical capacity C_p and resistance R_p of the MRs have been measured respectively, in a continuous electric field superposed over the static magnetic field with different fixed magnetic flux densities, B.

It has been shown that depending on the values Φ_{Fe}, these magneto resistors are devices in which the partition of the components C_p and R_p can be selected based on the applied values of the magnetic flux density. In the continuous electric field superposed over the static magnetic field, the quantities C_p and R_p depend on the quantities Φ_{Fe} and B, and are constant during the application of the magnetic field.

By using the dipole approximation model, the mechanisms that take part in the observed effects, are described. In connection to these mechanisms, the main technical characteristics of MRs have been elaborated and defined. The magnetocapacitive and magneto resistor effects of MRs have also been illustrated.

The results obtained can be useful in the research concerning the passive electrical devices for electric circuits, devices which are low cost, magnetically controllable with stable technical characteristics over time, and with high response speed to the application of magnetic fields.

New phospho-tellurite glassy systems with applications for magnetic sensors

M. Elisa[1], S. M. Iordache[1], A-M. Iordache[1], C. R. Stefan[1], I. C. Vasiliu[1],
B. A. Sava[2], L. Boroica[2], C. M. Dinca[2], A. V. Filip[2], V. Kuncser[3], C. Bartha[3],
A. C. Galca[3], M. Eftimie[4]

1 - National Institute for Research and Development for Optoelectronics - INOE 2000, 077125, Magurele, Romania
2 – National Institute for Laser, Plasma and Radiation Physics, 077125, Magurele, Romania
3 – National Institute of Material Physics, 077125, Magurele, Romania
4 – University Politehnica of Bucharest, 313 Spl. Independentei, 060042 Bucharest, Romania

Within the project 47PCCDI/2018 (Advance Nano), new phospho-tellurite vitreous materials were synthesized and investigated having application as Faraday rotator type magnetic field sensors. These glassy materials belong to the following compositional systems: (50-x) ZnO-$10Al_2O_3$-$40P_2O_5$-$xTeO_2$ (x = 5; 10; 15), (40-x) Li_2O-$10Al_2O_3$-$5TiO_2$-$45P_2O_5$-$xTeO_2$ (x =5; 10; 15), $35Li_2O$-$10Al_2O_3$-$5Nb_2O_5$-$45P_2O_5$-$5TeO_2$ and $39Li_2O$-$10Al_2O_3$-$1ZrO_2$-$45P_2O_5$-$5TeO_2$. Phospho-tellurite glasses were prepared by an unconventional wet method of reactant processing, followed by a preliminary heat treatment, melting-refining, molding by casting, heat treatment for stress releasing and optical processing. The method ensures a high chemical and optical homogeneity of the prepared vitreous materials. Phospho-tellurite materials were characterized as follows: optical spectroscopy (UV-Vis transmission, refractive index dispersion), structural spectroscopy (FTIR absorption, Raman scattering), phase analysis (X-ray diffraction), dilatometry (thermal expansion coefficient and characteristic temperatures), chemical stability (electrical conductivity of the powder glasses dispersed in water, as a function of time), magnetic characterization (magnetization depending on the applied magnetic field and temperature) and magneto-optical characterization (variation of the Faraday rotation angle and the Verdet constant depending on the wavelength). The magnetic characterization showed a diamagnetic behavior of these glassy materials and there was an increase in the values of magnetic susceptibility, Faraday rotation angle and Verdet constant with increasing TeO_2 content. It was found that the tendency of crystallization of these vitreous materials increases with increasing TeO_2 content, which required low melting, refining and annealing times (approx. 30 min). The values of the Faraday rotation angle are ranging between 0.087^o-0.158^o at 633 nm and of the Verdet constant are ranging between 0.012-0.156 min/Oe/cm at 633 nm.

Photodetectors based on Ge nanocrystals in oxide matrix

M.L. Ciurea[1], R. Muller[2], D. Ursutiu[3]
1 - National Institute of Materials Physics, Magurele, Romania
2 - TRANSILVANIA University of BRAȘOV
3 - National Institute for R&D in Microtechnologies, București, Romania

In Ge nanocrystals (Ge NCs), the transitions between energy levels give the possibility to fabricate infrared (IR) photodetectors for wavelengths up to 5 μm that can be extended to 10 μm by controlling the traps density located at Ge NC / oxide matrix interface. Also, the quantum confinement contributes to the increase of radiative recombination efficiency. For obtaining high performance optoelectronic devices the NCs size, density and dispersion uniformity in matrix are decisive characteristics and, therefore the preparation method has to insure a good control of these parameters.

We have prepared films formed of Ge NCs embedded in SiO_2, TiO_2 and HfO_2 matrices on p-Si wafers. For this, amorphous Ge-oxide films (different compositions) were co-deposited by magnetron sputtering and then were thermally annealed (rapid thermal annealing RTA) for nanostructuring.

The films morphology and structure were investigated by using high resolution TEM that shows besides Ge NCs formation, the oxide matrices are nanostructured (TiO_2 and HfO_2 NCs). Also, we have prepared films with GeSi NCs with low Si content to hinder Ge atoms fast diffusion. The photosensitivity properties of these films were studied by electrical and photoelectrical measurements taken on films.

Finally we obtained matrices of photodetectors by using metallic (Cr/Au) interdigitated contacts designed in 2 versions and optimized by taking into account results obtained from functional characterization.

In conclusion, we have obtained **matrices of photodetectors with a significant extension of spectral photosensitivity at room temperature up to 1400 – 1500 nm.**

Magnetic diluted oxide nanosystems decorated with magnetic nanoclusters

V. Mihalache, C. Negrila, I. Mercioniu, N. Iacob, A. Kuncser, G.Schinteie, A.Leca, G. Filoti and V. Kuncser
National Institute of Materials Physics, 077125, Magurele, Romania

Significant research efforts have been done for improving the magnetic properties of diluted magnetic oxides (DMO) or diluted magnetic semiconductors (DMS). One usual way was to dope DMO systems with transition metal TM ions with the aim of inducing additional magnetic defects in the neighborhood of the doping TM ions. As regularly observed by our studies the TM ions present a pronounced tendency of clusterization, the final product being in form of nanoparticles of DMO decorated by TM oxide clusters. The idea of the present studies was to exchange couple the ferromagnetic phase specific to DMO systems with the antiferromagneic TM oxide nanoclusters assumed to be formed with suitable size if using an increased doping percentage. Two systems were considered in this respect: (i) TiO2 nanoparticle systems doped with a high amount of Fe as prepared by laser pyrolysis and (ii) Zn-Fe-O nanoparticle systems (Z3F, Z20F and Z60F) produced by changing the Zn:Fe ratio (0.97:0.03, 0.8:0.2 and 0.4:0.6 in at.%) in Zn(II)-Fe(III)-carboxylate precursors.

The influence of doping Fe ions on the magnetic behavior of each phase is discussed in detail. Evidences for interface exchange couplings (with unidirectional anisotropy in specific conditions) between the long-range ferromagnetic phase and the fine clusters (antiferromagnetic in nature), which become frozen below temperature of 50, K, are provided in the case of the Fe doping TiO_2 nanoparticles. Concerning the second system, depending on the Zn/Fe ratio either nanocomposites or magnetic clusters decorating the DMO nanoparticles can be formed. We found evidence for a $ZnFe_2O_4$ spinel of high inversion degree (80-100%) and superparamagnetic (SPM) behaviors in all three samples by a remarkable correlation between HRTEM, FTIR, XPS, Mössbauer and magnetization analyses. Iron modifies the decomposition process of the precursor and enhances its viscosity, which appears to favour Zn- and Fe-rich phases separation and two-phase (ZnO and $ZnFe_2O_4$) systems of individual nanocrystals/nanoparticles are formed. For Z3F with lowest ampunt of Fe, a portion of iron was found in the Fe^{2+} state, presumably substituting for Zn^{2+} in zinc oxide. This sample behaves like a system of $ZnFe_2O_4$ SPM nanoclusters/nanoparticles (directly observed via HRTEM) decorating the ZnO nanoparticles rather than a Zn(Fe)O dilute magnetic semiconductor. All the mentioned systems can be further developed for specific applications: gas sensors, magnetic (nano)sensing, antibacterial agents, and (magnetic) catalysts.

L10 Phase Formation and Stability in FePt and FePtMn Nanocomposite Magnets

A.D. Crisan [1], P. Palade [1], A. Leca [1], D. Pantelica [2], I. Dan [3], O. Crisan [1]
1 – National Institute for Materials Physics, P.O. Box MG-7, 077125 Magurele, Romania
2 – National Institute for Physics and Nuclear Engineering, P.O. Box MG-6, 077125 Magurele, Romania
3 – R&D Consulting and Services S.R.L., 023761 Bucharest, Romania

Magnetic nanoscale materials exhibiting the $L1_0$ tetragonal phase such as FePt or ternary alloys derived from FePt show most promising magnetic properties as a novel class of rare earth free permanent magnets with high operating temperature. With the aim of demonstrating phase coexistence of two magnetic phases in an intermediate annealing regime and obtaining highly coercive FePt nanocomposite magnets, two alloys of slightly off-equiatomic composition of a binary Fe-Pt system were prepared by dynamic rotation switching and ball milling. The alloys, with a composition $Fe_{53}Pt_{47}$ and $Fe_{55}Pt_{45}$, were subsequently annealed at 400 °C and 550 °C and structurally and magnetically characterized by means of X-ray diffraction, ^{57}Fe Mössbauer spectrometry and Superconducting Quantum Interference Device (SQUID) magnetometry measurements. Gradual disorder–order phase transformation and temperature-dependent evolution of the phase structure were monitored using X-ray diffraction of synchrotron radiation. It was shown that for annealing temperatures as low as 400 °C, a predominant, highly ordered $L1_0$ phase is formed in both alloys, coexisting with a cubic $L1_2$ soft magnetic FePt phase. The coexistence of the two phases is evidenced through all the investigating techniques that we employed. SQUID magnetometry hysteresis loops of samples annealed at 400 °C exhibit inflection points that witness the coexistence of the soft and hard magnetic phases and high values of coercivity and remanence are obtained. For the samples annealed at 500 °C, the hysteresis loops are continuous, without inflection points, witnessing complete exchange coupling of the hard and soft magnetic phases and further enhancement of the coercive field. Maximum energy products comparable with values of current permanent magnets are found for both samples for annealing temperatures as low as 500 °C.

On the other hand, a granular alloy derived from binary FePt with low Pt content and the addition of Mn with the nominal composition $Fe_{57}Mn_8Pt_{35}$ has been synthesized in the shape of melt-spun ribbons and subsequently annealed at 600 °C and 700 °C for promoting the formation of single phase, $L1_0$ tetragonal, hard magnetic phase. Proton-induced X-ray emission spectroscopy PIXE has been utilized for checking the compositional effect of Mn addition. Structural properties were analyzed using X-ray diffraction and diffractograms were analyzed using full profile Rietveld-type analysis with MAUD (Materials Analysis Using Diffraction) software. By using temperature-dependent synchrotron X-ray diffraction, the disorder–order phase transformation and the stability of the hard magnetic $L1_0$ phase were monitored over a large temperature range (50–800 °C). A large interval of structural stability of the $L1_0$ phase was observed and this stability was interpreted in terms of higher ordering of the $L1_0$ phase promoted by the Mn addition. It was moreover found that both crystal growth and unit cell expansion are inhibited, up to the highest temperature investigated (800 °C), proving thus that the Mn addition stabilizes the formed $L1_0$ structure further. Magnetic hysteresis loops confirmed structural data, revealing a strong coercive field for a sample wherein single phase, hard, magnetic tetragonal $L1_0$ exists. These findings open good perspectives for use as nanocomposite, rare earth free magnets, working in extreme operation conditions.

New “exchange spring”-type systems

T. Roman[1], M. Grigoraș[1], G. Gurău[2], C. Gurău[2], N. Lupu[1]
[1]*National Institute of Research and Development for Technical Physics, Iași, Romania*
[2]*”Dunărea de Jos” University of Galați, Faculty of Engineering, Galaţi, Romania*

With the fast development of green-energy technologies, the consumption of Nd-Fe-B permanent magnets (PMs) is expected to double or even triple in the near future. Moreover, today's modern applications are pushing the limits of the properties associated with conventional NdFeB-based PMs to operate at elevated temperatures. Therefore, the development of NdFeB-based PMs with high magnetic performance and thermal stability becomes more important. MnBi alloys are one of the promising candidates for PM applications, since the MnBi low-temperature phase (LTP) has high magneto-crystalline anisotropy and a remarkable positive temperature coefficient of coercivity [1].

In this work, we have developed a new “exchange spring”- type system consisting of both MnBi and NdFeB magnetic phases. While the classical concept of “exchange spring” PM considers two magnetic phases with enhanced coercivity and B_s, coupled by exchange interactions, in our new approach the coercivity is “borrowed” from NdFeB, while MnBi enhances the thermal stability. The powders prepared from precursor melt-spun ribbons, were mixed in different ratios (NdFeB/MnBi = 100/0, 90/10, and 80/20 wt.%) and compacted by spark plasma sintering (SPS) or cold die-compaction technique. The studies done on different compacts revealed that the cold pressed sample containing 90%wt. NdFeB and 10%wt. MnBi exhibits an enhanced “exchange spring" behaviour, with $H_c > 1.2$ T, $M_s \sim 120$ Am^2/kg and the average temperature coefficient of coercivity, $\beta = 0.3812$ (%/°C) in the 298–423 K temperature region.

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Engineering of permanent magnets based on Co-rich Co-Zr alloys

F. Tolea[1], P. Palade[1], B. Popescu[1], M. Sofronie[1], A. Kuncser[1], C. Gurau[2], G. Gurau[2], V. Pop[3], R. Hirian[3], D. Benea[3], A. Ferenczi[3]

[1] *National Institute of Materials Physics, Atomistilor 405A, 077125, Magurele-Bucharest, Romania*
[2] *Faculty of Engineering,"Dunărea de Jos" University of Galati, Domnească Street, 47, RO-800008, Galati, Romania*
[3] *Faculty of Physics, Babes Bolyai University from Cluj*

The prospect of producing medium-energy permanent magnets without Rare Earths, using Co-rich Co-Zr alloys and unconventional synthesis methods under non-equilibrium conditions, was considered. There is experimental evidence that the rhombohedral phase of Zr_2Co_{11} has hard magnetic properties [1]: a high uniaxial anisotropy which can yield values of the coercitive field as high as 7 kOe, a high Curie temperature (500^{o}C), and a room temperature magnetization at saturation of about 60emu/g. There are however big challenges if one is interested to obtain a certain phase, given the polymorphic nature of Zr_2Co_{11} with the pseudohexagonal, orthorombic or rhombohedral crystalline structures having closely competitive energies [2]. Our strategy has been to synthesize the alloy by several processing methods and to study which one is more favorable for the formation of the rhombohedral Zr_2Co_{11} hard phase. Thus, we studied the Co-Zr alloy obtained by melt spinning, subsequently subjected to severe plastic deformation and via mechanical alloying. The ingots, prepared by arc melting in protective Ar atmosphere from high purity (99,99%) constituent elements, were melt-spun into ribbons, via the "melt spinning" method - the ribbons thus obtained were denoted as-quenched (AQ). We extend the research and evaluate the possibility to induce the formation of the hard magnetic Zr2Co11 rhomboedral phase by the high-speed, high-pressure torsion (HSHPT) novel technique [3]. Depending on the deformation level, the method should promote grain nanostructuring with superior mechanical properties, better plasticity, and increased fatigue limit. Alternatively, synthesis methods involving mechanical milling were performed to establish and optimize the specific parameters to induce nanocrystallization and tune the magnetic coupling starting with the nominal compositions Co82Zr18 and Co80Fe2Zr18. Also, we tried to stabilize the non-cubic high anisotropy structure of Zr2Co11 by the partial substitution of Co with alloying elements such as: Cr, Mo, C, B, Fe. Finally, our studies addressed as well the effect of thermal treatments on the nanostructure, mechanical and magnetic properties of Zr2Co11, again witn an amphasis on the enhancement of the magnetocrystalline anisotropy field of the hard magnetic phase.

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Magnetic nanoparticles dispersed in liquid media

V. Socoliuc, L. Vekas, N. Iacob, V. Kuncser
1 – Romanian Academy – Timişoara branch, Mihai Viteazu 24 Bv., Timişoara, 300223, România
2 – National Institute of Materials Physics, Atomistilor 405A Street, Magurele, Romania

We present the *Romanian Academy – Timisoara Branch* (ARFT) research and dissemination activities in the project *UEFISCDI PN-III-P1-1.2-PCCDI-2017-0871*. Spin-off cooperations will be briefly presented. Special attention will be given to two research directions related to liquid media dispersions of magnetic nanoparticles:

1. Fe_2O_3/SWNT magnetic nanocomposites synthesized at partner IC-INCDFM were dispersed in a high grade transformer oil based ferrofluid synthesized at P1-ARFT. The result was two samples of magneto-rheological fluids with different Fe_2O_3 to SWNT mass ratios. The magneto-rheological properties of the two samples are presented and discussed in correlation with magnetic and structural characterization data obtained in the project.

2. Fe_3O_4/FeCo magnetic nanocomposites were synthesized at INCDTIM-Cluj Napoca by means of miniemulsion, using a toluene based ferrofluid with Fe_3O_4 synthesized at ARFT in the project and FeCo nanoparticles synthesized at ICP-CA Bucharest. The high magnetization Fe_3O_4/FeCo nanocomposites were dispersed in a high grade transformer oil based ferrofluid synthesized at P1-ARFT to obtain two magneto-rheological fluids with different nanocomposites mass weights. The magneto-rheological properties of the two samples are presented and discussed in correlation with magnetic and structural characterization data obtained in the project by ARFT and IC-INCDFM, as well as in comparison with similar magneto-rheological fluids already investigated in this project.

Carbon nanotubes loaded with magnetic nanoparticles

A. Nila, A. Udrescu, M. Dinescu, M. Cristea, P. Ganea, I. Zgura, I. Mercioniu, N. Iacob, A. Leca, V. Kuncser, M. Baibarac
National Institute of Materials Physics, Atomistilor 405A Street, Bucharest, Romania, R-077125

Hematite (α-Fe_2O_3) is considered the most thermodynamically stable phase from all iron oxide phases, but the use of this in electronic applications is limited due to its low conductivity. [1] To overcome these limitations, the iron oxide has been combined to carbonaceous materials to improve the conductivity and thermal stability. In this manner, composites based on iron oxide and carbon nanotubes have been used as the anode materials for rechargeable lithium ion batteries [2], the active materials in supercapacitors cells [3] and remove of phenols from aqueous solutions [4].

In this communication, using the hydrothermal synthesis of the Fe_2O_3 particles in the presence of carbon nanotubes (CNTs) and the mechano-chemical interaction of the Fe_2O_3 particles with CNTs, the composites of the type Fe_2O_3/CNTs having the CNTs concentration equal to 8 and 27 wt.% have been prepared. X-ray diffraction studies performed using the Fe_2O_3/CNTs composites indicated that the addition of carbon nanotubes during the synthesis of the Fe_2O_3 particles does not affect their high crystallinity [5]. Regardless of synthesis method of the Fe_2O_3/CNTs composites, Raman spectra highlight an increase in the intensity of the D band, assigned to the disorder state or the defects from the graphitic lattice of CNTs. This fact was correlated with the assembling process of the Fe_2O_3 particles onto CNTs surface. According to the SEM studies, the hydrothermal method leads at: i) the Fe_2O_3 particles with size from 1.23 µm and ii) as increasing the CNTs concentration in the reaction mixture of the Fe_2O_3/CNTs composites at 27 wt.%, a gradual increase in the size of the resulted structures up to 6.2 µm takes place. Using dielectric spectroscopy, an assessment of the conductivity of the Fe_2O_3/CNTs samples is reported. Both in the case of the Fe_2O_3/CNTs samples prepared by the hydrothermal method and the mechano-chemical way, an increase of the conductivity of the Fe_2O_3 particles with increasing of the concentration of CNTs in the mass of the Fe_2O_3/CNTs composite takes place. The decrease of the thermal coefficient of the conductivity of the Fe_2O_3 particles with two or three orders of magnitude in the presence of CNTS indicates an electronic conduction typical of metals or semiconductors. A magnetic-fluid behavior is reported for the first time in the case of the Fe_2O_3/CNTs composites.

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Magnetic nanoclusters dispersed in conductive nonmagnetic metallic thin films

C. Locovei, A. Stanciu, S. Greculeasa, G. Schinteie, P. Palade, A. Kuncser,
N. Iacob, A. Leca, A. Alexandru-Dinu, V. Kuncser,
National Institute of Materials Physics, Atomistilor 405A Street, Bucharest, Romania, R-077125

The necessity of new materials with novel properties has been leading to the synthesis of nanomaterials with multifunctional features. In this regard, we obtained Au_xFe_{1-x} nanocomposite thin films of different thicknesses and compositions. For a narrow interval of both thicknesses and elemental ratios, Transmission Electron Microscopy evidenced the self-organization of metallic iron nanoparticles into lamellar forms embedded in the gold matrix. MOKE magnetometry shows the in-plane uniaxial magnetic anisotropy in case of the lamellar 2D organization of the magnetic clusters and no in plane magnetic texture of the same randomly distributed clusters with average size specific to a superparamagnetic behavior. SQUID magnetometry performed in two geometries (with the field in the film plane and perpendicular to it, respectively) and at various temperatures pointed for a strong magnetic in plane uniaxial texture in those samples with a specific amount of Fe (close to 30% at.), for mainly superparamagnetic behavior in case of samples with a lower amount of Fe and for a magnetic domain structure without magnetic texture in case of samples with a higher amount of Fe. The data collected by Conversion Electron Mossbauer Spectroscopy were fitted with a hyperfine magnetic field distribution corresponding to Fe configurations with Au impurities in case of samples with intermediate Fe content (30% at.). For samples with a higher amount of Fe, an additional magnetic sextet was considered to account for larger particles of α-Fe. Two forms of crystalline structures were evidenced for the Au matrix, their relative amounts depending on the Fe content. To note that in cases where uniaxial magnetic anisotropy is present the less usual hexagonal structure.is evidenced for Au. Magneto-transport measurements taken at different temperatures and in different geometries have proven specific magneto-resistance effects, in direct relation to the type of self-organization of the magnetic clusters and the stabilized crystalline structure of the Au matrix. Thus, the multifunctionality of Au_xFe_{1-x} nanophasic system is highlighted by the combination of properties presented in this study.