Theme:

Anisotropic Nanocomposites for High Magnetic Energy Densities Permanent Magnets

Contract: CEEX 19/2005
Contracting Authority: **MATNANTECH**, Polytechnical University Bucharest
Contractor: *INCDIE ICPE – CA*, Bucharest
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Programme: CEEX – Excellence Research
Project Category: MODULE I – Complex Research & Development Projects
Project Type: P-CD
Acronym: **NANOMAG**

Project Coordinator: *INCDIE ICPE – CA*, Bucharest
Partners: *BABES-BOLYAI University*, Cluj-Napoca
INCDFM, Bucharest
INCDFT, Iasi

Thematic areas: 4.1 Nanosciences, Nanotechnologies
4.2 Materials
4.3 New Production

Technological Platform: Advanced Engineering Materials and Technologies

General objectives: The project propose the study of the physical properties of the nanocrystalline materials having a high potential to be developed into high–performance permanent magnets, with very high energy product, finalising with an anisotropic nanocomposite system *demonstrator*, which represents a novelty at the national level.

Project finalizing:
- Functional model performing;
- Patent for the original technic solutions

Project planning / Time table

Stage I/2005: Rare-Earth (RE) -Transition Metal (TM) Nanocomposite Systems: Physical Properties and Technical Performances
Period: 01.10.2005 – 10.12.2005

Stage II/2006: Preparation And Studies Of The Isotropic Nanocomposites Systems, Magnetic Hardened By Exchange Interactions
Period: 11.12.2005 – 15.05.2006

Stage III/2006: Identification Of The Precursors Systems For The Anisotropic Permanent Magnets
Period: 16.05.2006 – 30.11.2006

Stage IV/2007: Study of The Influence of the Alloying Elements on the Physical Properties of the Nanocomposites Alloys
Period: 01.12.2006 - 30.04.2007

Stage V/2007: Induced Magnetic Anisotropy by Different Techniques and the Study of the Physical Properties of the Anisotropic Systems
Period: 01.05.2007- 30.11.2007

Stage VI/2008: Anisotropic Nanocomposite Magnet, Hardened by Exchange Interactions – Demonstrator

Period: 01.12.2007 - 15.05.2008

Potential end-users: producers of high magnetic performances materials and devices, for electrical engineering, electronics, automative industry, computer and medical techniques.

The technical, scientific, economic and social impact. The magnetic materials were used in many devices and equipments: in industry and also, at home: motors, generators, PC.

The advantages of such anisotropic nanocomposite system are:

- *the RE content can be lower with* 15 – 50 % *as the RE content of the sintered or bonded magnets*;
- *the price is lower*, in comparison with other type of permanent magnets (PM) based on RE;
- *the magnetic performances are higher*;
- the corrosion resistance is higher, due to the reduced content of RE;
- have *a better mechanical resistance* (fracture tensile), due to the structure with fine nanograins and to the existence of a relative soft phase, alpha-Fe.

Also, we can mention the exploitation possibilities of the technological and economic advantages offered by this new type of PM, by their capitalization at the end-users, with lower fabrication prices, simultaneously with high performances. The increasing of the magnetic performances and the improvement of the processing parameters will lead to the increasing of the efficiency: for example, the use of this magnets, based on RE in efficient motors can «save» ~ 150,000 Euros at national level. For this reason, we consider that the finalization of the project leads to the increasing of the Romanian products competitivity, and to assure a long – stable development in the economic plan.

By the realization of the final objective of the project – preparation of the anisotropic nanocomposite systems, based on RE-TM, useful for the fabrication of the permanent magnets - will be created *new working places*, on the PM and devices equipped with PM producers.

The project has identified and created the research consortium, with all the teams with excellence in the field of the hard magnetic materials; the teams are composed by prestigeous scientists *recognised on the national and international level.* By optimal and corect allocation of the human and materials resources, the project will realise a *technological networking*, stimulating the formation of a scientific and technological *cluster* in the actual field of *anisotropic magnetic nanocomposites systems*. The results of the project will be the increasing of the knowledge level, by the understanding of the physical phenomena at tehnical level of the nanostructures. The fundamental interest results will increse the scientific potential and the visibility of the four entities involved in the project, the national consortium having big chances to the integration in the ERA.

The project creates opportunities to learn, especially for the young researchers. The young scientists can learn:

- to work with the SEM,
- to measure the magnetic characteristics by vibrating sample magnetometer at different temperatures and fields,
- to work with the vacuum equipment for thermal treatments (annealing, homogenizations, recrystallisation, melting, melt-spinning);
- to consolidate their knowledge by the bilateral agreements facilitated access in the description of the magnetic phenomena;
- to sustain master dissertations or Ph.D. theses in the field of the project;

- to participate at the international and national conferences, workshops, congress;
- to publish their papers in scientific prestigious journals;
- to take part at the inventors team.

The technological impact will be *major on the technological media*, by:

- association of research teams, with the same research interests, from national R-D institute and universities, in an strong consortium;
- stimulation of the formation, on the project partners level (R-D and pilot plants), of an laboratory network involved in the elaboration of the new concepts and techniques for the processing of the advanced nanocomposite materials;
- stimulation of the formation of a multidisciplinary, scientific and technological service center, specific to he domain

Environmental impact. The new processing technique is an *ecological technique*, *without negative impact to the environment* - in comparison with the classical techniques (air melting or powder metallurgy ways) - case in which some noxes, fine powders, volatiles were free in the environment. In this ways, the conditions regarding the bioethics and biosecurity are accomplished. Due to the specific energy, significant more, in industry can be realized the same effects (the same remanence in an certain air gap, the same magnetic energy in an decreased volume) with miniaturized PM (volume lower with ~ 33 %); that means the *saving of the natural resources* – a condition for the environment protection and an fundamental principle for *the long –durable development*.

By the modernization of the developed material quality, the project can contribute to the scientific and technological, sustained and accelerating integration of Romania to the UE structures.

BACK

Stage I / 2005

RARE-EARTH (RE) - TRANSITION METAL (TM) NANOCOMPOSITE SYSTEMS:
PHYSICAL PROPERTIES AND TECHNICAL PERFORMANCES
Period: 01.10.2005 – 10.12.2005

Objectives:
- Studies concerning the preparation methods of the RE-TM nanocomposites systems;
- Analysis of the systems realized up to now by the consortium teams
- Characterization methods of the nanocomposite systems
- Design of the hot working devices

Obtained results:
- Documentation activities;
- Activities related to the design and the execution of the experimental units and the specific devices for hot pressing / hot deformation of the RE-TM melt-spun powders.

The heating system, with quartz lamps (see figure 1), has been finalized and will equippe the experimental unit for hot pressing of the RE-TM melt-spun powders, in inert atmosphere, in order to avoid the oxidation and inflammation phenomena of this powders.

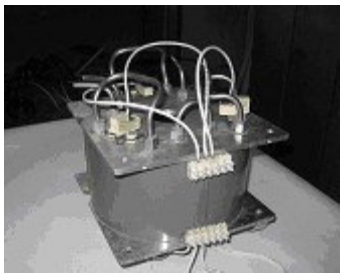


Fig. **1** Heating system with quartz lamps

BACK

II. PREPARATION AND STUDIES OF THE ISOTROPIC NANOCOMPOSITES SYSTEMS, MAGNETIC HARDENED BY EXCHANGE INTERACTIONS

Period: 11.12.2005 – 15.05.2006

III. IDENTIFICATION OF THE PRECURSORS SYSTEMS FOR THE ANISOTROPIC PERMANENT MAGNETS

Period: 16.05.2006 – 30.11.2006

Stage objectives:

- Synthesis of the RE-TM isotropic nanocomposites systems (RE – rare earths, TM-transition metals);
- Preliminary characterization (structural and magnetic) of the prepared samples;
- Selection of the methods and synthesis procedures for the RE-TM nanocomposite systems, precursors of the anisotropic permanent magnets;
- Selection of the methods and characterisation procedures for the RE-TM nanocomposite systems;
- Dissemination of the results;
- Publication of scientific results in ISI quoted reviews journals.

Results:

Have been prepared magnetic isotropic nanocomposites samples, based on NdFeB alloys, hardened by exchange interaction. This new materials have been structural (by XRD and AFM) and magnetic characterised (by VSM and hysterezisgrah measuring), in order to identify the precursors systems, which can be used the further stages of the project for magnetic anisotropy induction.

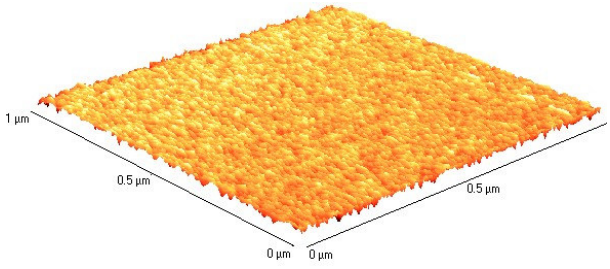


Fig. **1** NdFeB ribbons characterized by AFM

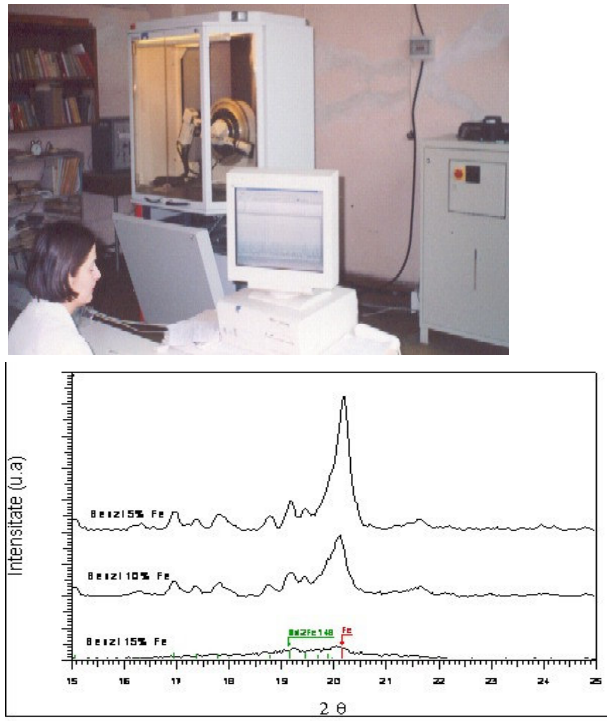
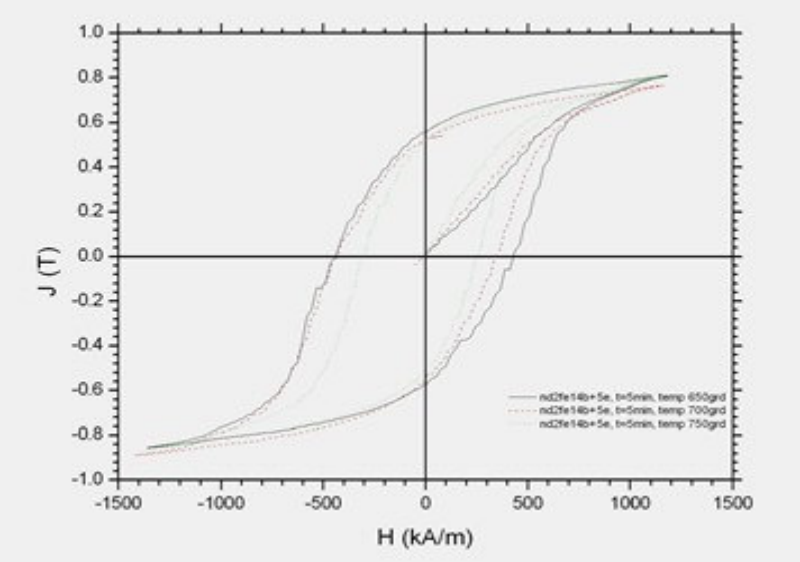


Fig. **2** XRD spectra of the $Nd_{11}Fe_{83}B_6$, $Nd_{10.5}Fe_{84}B_{5.5}$ and $Nd_{10}Fe_{85}B_5$ ribbons, prepared by melt-spinning


J (T)
H (kA/m)
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
-0.4
-0.6
-0.8
-1.0
-1500
-1000
-500
0
500
1000
1500
nd2fe14b+5e, t=5min, temp 650grd
nd2fe14b+5e, t=5min, temp 700grd
nd2fe14b+5e, t=5min, temp 750grd

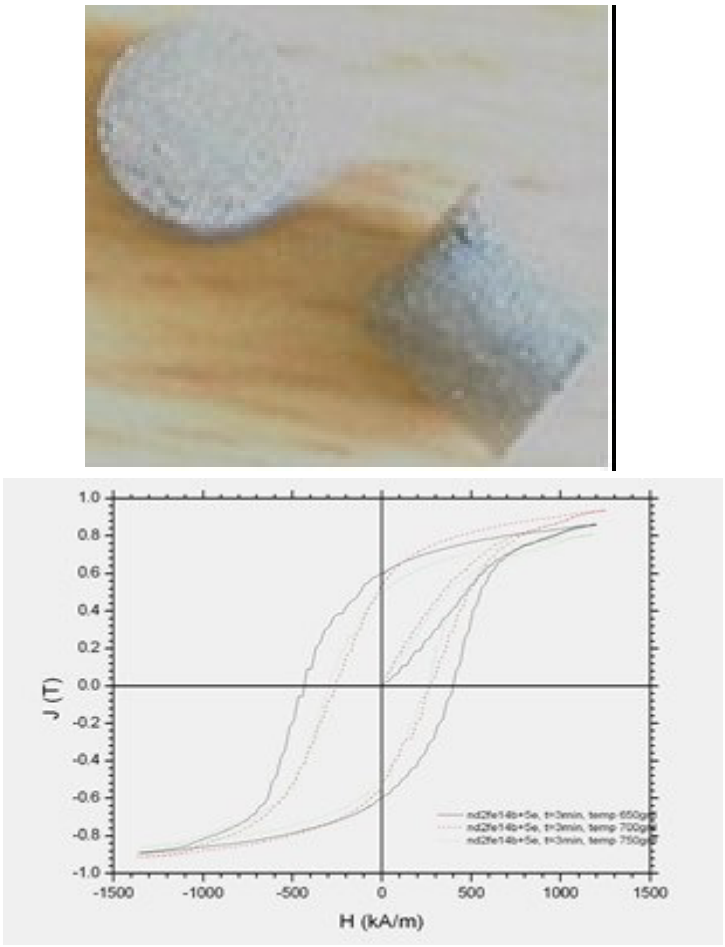


Fig. **3** Hysteresis curves of the magnetic isotropic $Nd_{11}Fe_{83}B_6$ nanocomposies samples,
prepared by annealing in different conditions

Have been studied the influence of the parametres of the processing on the structure and the magnetic properties (see the figures **1** - **3**).

Have been performed a *Product Card* (see link) for the institute catalogue, which describe the physical and technical and functional characteristics of this new material:

- Remanence $B_r > 0.7$ T;
- Coercivity $H_{cJ} > 500$ kA/m;
- M_r / M_S ratio is more than 0.58 (up the 0.5 value, the magnets are nanocomposites, hardened by exchange interaction).

Have been designed the hot pressing and deformation unit, in vacuum/inert atmosphere (argon), and also the performing of the heating systems of this unit.

Results dissemination

- W. Kappel, M. M. Codescu, N. Stancu, J. Pintea, E. Patroi, D. Patroi - *High Energy Density Magnetic Materials for Electronic Packaging*, Proc. of the 1st Electronic Systemintegration Technology Conference ESTC2006, 5-7 September 2006, Dresden, Germany, p. 338 – 43, ISBN 1-4244-0553-X, awarded paper (Best Award Poster) (see link).
- W. Kappel, M. M. Codescu, N. Stancu, J. Pintea - *Influence of the Recrystallization Processes on the Magnetic Properties of the $Nd_2Fe_{14}B$/alpha-Fe Nanocomposites*, Scientific Workshop "Materials for Electrical Engineering" MmdE 2006, June 15 – 17 2006, Bucharest
- W. Kappel, M. M. Codescu, N. Stancu, E. A. Patroi, D. Patroi, M. Valeanu J. Pintea, A. Jianu, F. Lifei - *Influence of the Recrystallization Processes on the Structural and Magnetic Properties of the $Nd_2Fe_{14}B$/alpha-Fe Nanocomposites,* International Conference for Advanced Materials ROCAM 2006, September 11 – 14, 2006, Bucharest, in press in Journal of Optoelectronics and Advanced Materials
- W. Kappel, M. M. Codescu, N. Stancu, J. Pintea, E. A. Patroi, D. Patroi, S. Hodorogea – *Applications of High Energy Magnetic Materials, the* 4 International Conference for Materials and Manufacturing Technologies, September 21 – 23, 2006, Cluj-Napoca.
- W. Kappel, D. Patroi, M. M. Codescu, E. A. Patroi, N. Stancu, J. Pintea - *Magnetic Nanocomposites based on NdFeB*, Romanian – German Workshop, October 26 – 27 2006, Bucharest
- F.Tolea, B. Popescu, M. Valeanu, A. Birsan - *Exchange Coupling Effect in $R_2Fe_{14}B$/a-Fe Nanocomposite Magnets (R=Nd, Pr),* 7th International BalKan Conference on Applied Physics, Constanta, July 5-7, 2006
- H. Chiriac, M. Grigoras, M. Urse - *The Additions Effect on he Microstructure and Magnetic Properties of [NdFeB/M]´n Thin Films*, Proceedings of the Materials For Electrical Engineering Conference - MmdE 2006 & ROMSC 2006, Bucharest, June 15-17, 2006, Journal of Optoelectronics and Advanced Materials, vol. 9, issue 4, 1165 – 8, 2007
- H. Chiriac, M. Grigoras, M. Urse - *The Co Content and Stratification Effect on the Magnetic Properties, Microstructure and Phase Evolution of [NdFeBNbCu/Co] x n Thin Films*, 10th Joint MMM-INTERMAG Conference, Baltimore, S.U.A, MD, January 7-11, 2007
- In the frame of the Bucharest International Fair TIBCO 2006, on the Research and Innovation Fair, the specialists team involved in this project has participate with isotropic magnetic nanocomposites samples, based on Nd-Fe-B alloys, and also with the Product Card, describing the physical and functional characteristics of the of this new nanocomposite hard materials.

Conclusions

We intent to use the DTA investigations, corroborated with the XRD analysis, in order to minimise the range of the annealing temperatures and to establish the optimal parameters for the hot processing of this materials.

Will be finalised for the folloing stage the experimental unit for hot pressing / hot deformation, in order to indice the magnetic anisotropy for the nanocomposite materials.

Will be continued the experimental researches for the preparation of the melt-spun RE-TM alloys, in the anisotropy inducing way by hot pressing, followed by hot deformation, the both performed in inert atmosphere (Ar), during a very short time, at strict temperatures, determined by recrystalisation treatments and DTA studies.

Will be studied, also, the influence of some alloying elements, as: Pr, Dy, Ga, Al, Co, which favorise the magnetic properties development and the hot presing/ deformation.

BACK