



European Science Foundation

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ESF Exploratory Workshop, 21 - 25 September 2003

Organized by the Institute of Electronics, Bulgarian Academy of Sciences

**Microwave and optical behaviour of nanogranular ferroxides
(powders, fluids and composites). High frequency characterisation
and biomagnetic applications.**

R E P O R T

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1. Executive summary and 2. Scientific content of the event

The ESF Exploratory Workshop entitled “Microwave and optical behaviour of nanogranular ferroxides (powders, fluids and composites). High frequency characterisation and biomagnetic applications” was held from 21 to 25 September 2003 near Sofia, Bulgaria.

The event was organized by the Institute of Electronics at the Bulgarian Academy of Sciences and was financed by the European Science Foundation. It was convened by Philippe Tailhades from “Paul Sabatier” University, Toulouse, France, and Ivan Nedkov, Institute of Electronics, Bulgarian Academy of Sciences, Sofia, Bulgaria. The European Science Foundation was represented by Prof. Massimo Martinelli of the University of Pisa, Italy, who opened the meeting with a short overview of the ESF and its activities. The opening session was chaired by Prof. H. Hofmann of the Federal Institute of Technology, Lausanne, Switzerland, Chairman of the European Programme COST 523 “Nanotechnology”.

The total number of participants was 29, of whom three from Belgium (incl. one lecturer), one from Germany, one from Ireland, one from Russia, one from Slovenia, two from France, one from Croatia, one from the Czech Republic, one from Switzerland and 13 from Bulgaria (incl. eight lecturers and four technical staff). In addition, there were two lecturers from Japan and one from the United States.

During the Workshop the Bulgarian Publishing House Heron Press organized an exhibition of scientific literature.

The Program of the Workshop was focused on the modern trends in the development of measurement techniques used in the characterization of nanosized magnetic materials in the microwave (incl. THz) and optical spectra and dynamic behavior of the nanosized magnetic objects as well as the progress in the protection of biological objects, the latest achievements in the techniques for preparation of nanogranular ferroxides (powders, fluids and composites) for bioapplications.

The 24 lectures delivered were in three main topics – Characterization and modeling, Nanotechnological approaches and Bio-magnetism and applications topically separated in 6 sessions, namely:

1. Nanostructured magnetic oxides (monodomain, particles and clusters);
2. Ferrofluids and composites;
3. Biomagnetism and nanostructured magnetic oxides;
4. Characterization of the properties of these materials;
5. Modeling;
6. Applications.

A large proportion of the lectures and discussions was devoted to the microwave and optical methods for studying the dynamic behavior. The lectures of Prof. P. Fannin of Trinity College, Dublin, Ireland (*“Measurements and analysis of the complex susceptibility of ferrofluids”*) and of Prof. S. Nikitov, Corresponding Member of the Russian Academy of Sciences, Institute of Radioelectronics, RAS (*“Image formation by frequency method in magnetic (atomic) force microscopy”*, and *“Magneto-photonic and magnonic crystals”*), which dealt with microwave techniques for the analysis of the magnetic properties of fluids and the behavior of the superparamagnetic particles as magneto-photonic and magnonic crystals, as well as the modern optical methods for their characterization, were met with particular interest. An interesting addition to this topic was the report of *a novel all-optical technique for precise measurement of very low magnetic fields*, developed by a joint Bulgarian-Italian research team (Prof. S. Kartaleva and co-workers at the Institute of Electronics, Bulgarian Academy of Sciences, and Prof. S. Gozzini and Prof. V. Biancalana, CNR, UNISI, Pisa, Italy). Dr. K. Zadro of the University of Zagreb, Croatia, presented the capabilities of the classical methods in a DC field (SQUID) for investigating *the magnetic relaxation processes in small particles*.

In his lecture *“Advances in nanomaterials characterization”*, Prof. I. Petrov of the University of Illinois, Urbana-Champaign, USA, demonstrated some of the latest achievements in the scientific instrumentation and its potential in the structural studies of nanosized materials.

A natural continuation on the characterization of nanosized magnetic objects was the discussion of the modern approaches to the modeling of their properties. Important aspects of the bio-magnetic interactions on the nanometer scale were considered in the lectures of Prof. A. Proykova of St. K. Ohridski University of Sofia *on advanced simulation techniques for nanosized systems*; of Prof. G. Munteanu of Institute of Physico- Chemistry, Romanian Academy of Sciences, Bucharest, Romania *on modeling of nanocomposite materials*, and Dr. E. Balabanova, Institute of Electronics, BAS *on modeling of nanoparticles synthesis in aerosol*, who turned the audience’s attention to the potential of the Monte Carlo and molecular dynamics techniques in modeling the magnetic properties of nanosized systems.

Another important part of the lectures and discussions was dedicated to the advances in the techniques for preparation of nanosized ferroxides. Attempts to specify the properties that determine the possibilities for bio-application of superparamagnetic particles were made in the lectures of Prof. H. Hofmann of the Federal Institute of Technology, Lausanne, Switzerland, *on Nanostructured biomagnetic materials: from Physics to Applications*, Prof. Ph. Tailhades, University Paul Sabatier, Toulouse, France *on the Different ways to tailor the magnetic properties and size of ferromagnetic oxide particles*, Prof. G. Vissokov, Institute of Electronics, BAS *on the Plasma chemical preparation of ferroxide nanoparticles* and of Prof. I. Nedkov, IE, BAS, Sofia,

Bulgaria on the *Magnetization reversal and anisotropy in ferroxide nanoparticles*. The ferroxides of the type Fe_3O_4 and $\gamma\text{-Fe}_2\text{O}_3$ were pointed out as the best choices for the applications considered.

Another fruitful discussion focused the attention of the participants on the problems related with the surface oxidation of the magnetic ferroxide particle. The ferroxides with hexagonal structure were defined as promising in the development of novel materials for protection of biological objects from MW radiation in the lectures of Prof. J. Takada of Yokohama University, Japan, whose talk was on *Microwave absorbers based on the ferroxide structures* and of Dr. D. Lisjak of “Jozef Stefan” Institute, Ljubljana, Slovenia on *U-type hexaferrites*. The hexaferrite materials with large anisotropy prepared by them open up new possibilities for a wide range of applications.

In his lecture “*Making magnets by bacteria: the biomineralization of magnetic nanoparticles*” Dr. D. Schueler of the Max Plank Institute of Marine Biology in Bremen, Germany, gave a vivid account of the nanosized ferroxide particles existing in living organisms (magnetotactic bacteria) and analyzed comprehensively the processes of mineralization in living matter; the properties of nanoparticles thus produced were compared with those obtained by means of modern nanotechnologies. The following discussions concentrated on their role in the natural balance in living organisms and the possible bio-applications.

Aspects of the physics of living matter were also discussed by Prof. A. Petrov, Regular Member of the Bulgarian Academy of Sciences, director of the Institute of Solid-State Physics, BAS, as he presented his monograph “*The Lyotropic State of Matter – Molecular Physics and Living Matter Physics*”. The author dwelled specifically on new data concerning membrane effects in the living cell and the interaction nanoparticle-living cell.

A fundamental problem related with bio-applications of ferroxide materials, namely, their encapsulation by bio-compatible polymers, was discussed by Prof. J. Bibette of University Paris-Sud, France in the lecture “*Colloidal assembling for biological diagnostics*”, and Dr. Ch. Jerome, of the University of Liege, Belgium on *Functionalized magnetic nanoparticles produced in a copolymer micelles*. Prof. Bibette reported on the effects of self-organization in a water emulsion of 10-nm $\gamma\text{-Fe}_2\text{O}_3$ particles encapsulated in an organic film, as used in biological diagnostics. Dr. Ch. Jerome shared results on the preparation and possibilities for medical applications of magnetic fluids and gels.

Promising experimental results and ideas on the possible applications of the nanostructure ferroxide magnetic materials were present in practically all lectures, but in this respect the program was completed by the encyclopedic lecture on the *Magnetic nano- and microparticles in bioscience and biotechnology* of Dr. I. Safarik of the Institute of Landscape Ecology, Prague, the Czech Republic, centered on the topics of known natural phenomena related to superparamagnetic

particles and the possible bio-applications of ferroxide powders, fluids and composites, and the lecture “*Using sub-millimeter radiation for the study of the living body*”, by Prof. T. Idehara of Fukui University, Japan, presented by Dr. S. Sabchevski of the Institute of Electronics, BAS. The latter lecture made a particularly strong impression. The new sub-mm generator described and its possible applications in the study and treatment of the living body are rated among the latest top achievements in the field and will certainly stimulate further microwave research and development in the bio-world.

The hot topics were subjects of long discussions during the workshop; to summarize them, one should mention that the modern nano-technological techniques proved their potential in the preparation of nanosized ferroxide materials, with the specificity that categorizes them as a distinct state of matter. The superparamagnetic state of these materials is not well understood, because of the complicated magnetic structure and superexchange interactions. The possibilities for bio-applications of superparamagnetic ferroxide particles are still not well explored, with the main obstacle being the lack of non-destructive techniques for monitoring and controlling the stoichiometry, crystal structure and shape of the grains, as well as their magnetic properties at the interface magnetic particle / organic matrix (protective encapsulating film, liquid (fluid) or solid composite). The discussions that took place confirmed that small deviations in the magnetic particle shape (from spherical to needle-like), which cannot be avoided in 100 % by the modern nanotechnologies, can turn the harmless particle into a hazard for the living organism.

The relaxed and friendly atmosphere established enabled the participants in the Workshop to exchange opinions and ideas on future scientific contacts and joint research projects (ESF Scientific Network). Particularly productive along these lines was the official meeting of Prof. M. Martinelli - ESF’s representative at the Workshop, and Prof. N. Yakimoff, Scientific Secretary General of the Bulgarian Academy of Sciences, and the other participants in the event, where, among other important topics, the possibilities were discussed for ESF Scientific Network creation. The natural result of this was the participants’ uniting behind the idea of preparing an ESF research project with the draft title of *Superparamagnetic Particles in Biotechnology and Medicine: Physical Properties, Colloidal Behavior, Applications* (the draft of the project is presented below).

To conclude, it is the organizers’ belief that the Workshop was very successful and lived up to the expectations for fulfilling its aims.

3. Assessment of the results, contribution to the future direction in the field

ESF Scientific Network Project: SUPERPARAMAGNETIC COMPLEX MATERIALS IN LIFE SCIENCES AND BIOTECHNOLOGY: Synergic behavior, Biology and Chemistry

Acronym: BIOSUPERMAG

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Motivation:

The project is a natural continuation of the discussions that took place during the ESF Exploratory Workshop “Microwave and optical behaviour of nanogranular ferroxides (powders, fluids and composites). High frequency characterization and biomagnetic applications”, Sofia, 21-25 September 2003. The earliest version was submitted to the ESF representative at the Workshop.

The project envisages combining the efforts of a group of scientists from 11 European countries ESF Members (**Belgium, Bulgaria, Croatia, Czech Republic, France, Germany, Ireland, Italy, Romania, Slovenia, Switzerland**) and preliminary application of 3 non ESF Members (**Russia, USA and Japan**), who have accumulated a critical mass of experience and knowledge in the fields of synergic behaviour, biology, physics and chemistry of nano-sized superparamagnetic materials. Its aim is to create new knowledge concerning the superparamagnetic complexes (ferroxide core / organic shell) and their applications in life sciences and biotechnology. These materials are still not well studied, so that the project will contribute to:

- The development of sufficiently stable technologies for preparation of superparamagnetic complexes (SPC) and their introduction in fluids and composite structures. Clarification of the processes of self-organization, observed in SPC in a liquid matrix, the magnetic interactions and

the membrane effects during the SPC penetration in living cells. Study of the possibilities for the organic shell to fix medicinal substances for drug delivery applications.

- The investigation of the physics and chemistry of the interface superparamagnetic particle / organic shell, incl. magnetic interactions.
- The clarification of the process of mineralization and production of nano-sized ferroxide particles (e.g. Fe₃O₄) in living organisms and their role in the natural balance of the living organisms.

The project will particularly emphasize the development of novel techniques for characterization of SPC-fluids and composites that are based on their magneto-optical properties and on resonance effects in the microwave range; this will contribute to the better understanding of the SPC dynamic behaviour, incl. the self-organization processes, the latter still being a “white field” in the fundamental knowledge concerning these materials.

Table 1

Scientific fields	Research topics
Physics	Relaxation behavior, resonance, spin glass, high M_S , high K , non-linear phenomena, diffusion of magnetic particles, magnetic separation from large volumes
Chemistry	Synthesis of highly magnetic ferrofluids, beyond γ -Fe ₂ O ₃ , synthesis of highly superparamagnetic colloids based on the above- fragmentation/growth, polymerization, surface chemistry – colloidal stability, biocompatibility, grafting of biologically active molecules, composites – magnetic + organic matrix, quantum dots, oxides, dyes
Biotechnology	Separation technologies, immobilization techniques
Medicine	Particle-cell interaction, toxicity, biocompatibility

The project is essentially multi-disciplinary; it is expected that it will generate fundamental knowledge in the fields of biology, physics and chemistry, etc. (see Table 1). Besides the purely scientific goals, the project will seek the practical implementation of the knowledge obtained. Table 2 below summarises the potential fields of application of the expected results.

Table 2

Potential applications	Phenomena/Properties
1. Separation (Proteins, Cells, DNA, RNA)	Transport, magnetic field gradient, high saturation magnetization (M_S), different materials possible, incl. core-shell, blocking temperature, alignment of particles in a magnetic field.
2. Hyperthermia, heat generation with external AC magnetic field	Néel relaxation, Brownian relaxation, high magnetic anisotropy (K), resonance effects, core-shell, different materials possible, if not medical application
3. Drug delivery	High M_S in iron oxide, core-shell to minimize the unordered surface layer, alignment of particles in a magnetic field, drug binding, drug release
4. Self-organization and formation of arrays	Alignment of particles in a magnetic field, particle-particle interaction (magnetic and colloidal), stabilization of particle alignment,
5. Diagnostics	Highly sensitive one-step multi-criterial techniques

The project duration is **3 years**. For the first year (2004) we envisage the organization of one meeting and the 11 short-term exchange visits for exchange of experience. The appropriation of the subsidy will be decided collectively, based on the contribution of the respective participants, especially the teams with the most promising results.

ESF Member Country Participants:

1. Switzerland

2. France

3. Ireland

4. Belgium

5. Germany

6. Italy

7. Romania

8. Bulgaria

9. Slovenia

10. Czech Republic

11. Croatia

3 years - Preliminary financial program

1. Workshops – 3 years x 15 000 Euro = 45 000 Euro

2. Short-term visits – 90 000 Euro

Total: - 135 000 Euro

4. Final programme SCIENTIFIC PROGRAM

Saturday, 20 September

10:00 – 18:00 - Arrival and Registration

Sunday, 21 September 2003

10:00 – 18:00 - Arrival and Registration

10:00 – 11:20:00 – I. Nedkov (Bulgaria) Presentation of Institute of Electronics and Bulgarian Academy of Sciences

14:00 – 18:00 - Opening of the Exposition of “HERON” Press. Editor

Monday, 22 September

Opening Ceremony

9:30 - M. Martinelli (Italy) - Representative of ESF

9:45 - H. Hofmann, (Switzerland) – Chairman of the European Programme COST 523 “Nanotechnology”

Session - Nanostructured magnetic oxides (single domain, particles, and clusters) – **Chairs:**

Ph. Tailhades, University “Paul Sabatier”, Toulouse, (France) and I. Petrov, Center of Microanalysis of Materials, University of Illinois at Urbana-Champaign, (USA)

10:00- *Magneto-Photonic and Magnonic Crystals.* - S. Nikitov, (Russia)

11:00 Coffee break

11:15 - *Advances in Nanomaterials Characterization* – I. Petrov, (USA)

12:15 - *Magnetisation Reversal and Anisotropy in Ferrosinzel Nanoparticles* - I. Nedkov, (Bulgaria)

13:30 Lunch break

Session - Ferrofluids and Composites

Chairs: P. Fannin, Trinity College, Dublin, (Ireland) and A. Petrov, Institute of Solid State Physics, Bulgarian Academy of Sciences.

15:30 – *Measurement and Analysis of the Complex Susceptibility of Ferrofluids* - P.C.Fannin, (Ireland)

16:30 – *The Lyotropic State of the Matter – Molecular Physics and Living Matter Physics*- A. Petrov, (Bulgaria)

17:30 Coffee break

18:00 – *Crystals. Image Formation by Frequency Method in Magnetic (Atomic) Force Microscopy. Magneto-optic Visualisation of Small Scale Scattering Fields Created by Dynamic and Static Objects.* - S. Nikitov, (Russia)

Tuesday, 23 September

Session - Biomagnetism and nanostructured magnetic oxides

Chairs: H. Hofmann, Technical University of Lausanne (Switzerland) and I. Safarik, Laboratory of Biochemistry and Microbiology, Institute of Landscape Ecology (Czech Republic)

9:00 - *Nanostructured Biomagnetic materials: From Physics to Application* – H. Hofmann, (Switzerland)

10:00 - *Making magnets by bacteria: The biomineralisation of magnetic nanoparticles* - D. Schueler, (Germany)

11:00 - Coffee break

11:30 - *Colloidal Assembling for Biological Diagnostics* – J. Bibette, (France)

12:30 - *Functionalized Magnetic Nanoparticles Produced by a "in situ" reduction process in a copolymer micelles* – Chr. Jerome, (Belgium)

13:30 - Lunch break

Session - Characterisation

Chairs: J. Delwish, University of Liege, (Belgium) and I. Nedkov, Institute of Electronics, Bulgarian Academy of Sciences.

15:30 - *Different Ways to Tailor the Magnetic Properties and the Size of Ferrimagnetic Oxide Particles* - Ph. Tailhades, (France)

16:30 – *All Optical Methods for Precise Measurement of Magnetic Fields* - S. Cartaleva, (Bulgaria), presented by Ch. Andreeva

17:30 Coffee break

18:00 – *Magnetic Relaxation in Small Particles* – K. Zadro, (Croatia)

Wednesday, 24 September

Session - Modelling

Chair: G. Munteanu, Romanian Academy of Sciences and A. Proykova, University of Sofia (Bulgaria)

9:00 - *Simulation Techniques (Monte Carlo and Molecular Dynamics) for Nanosized Systems* - A. Proykova (Bulgaria)

9:45- *Modelling of Magnetic Properties of Nanocomposite Materials by Monte-Carlo Simulation*, G. Munteanu (Romania)

10:30 - Coffee break

11.00 – *Modelling of the Processes of Nanoparticles Synthesis in Aerosol Reactors* – E. Balabanova (Bulgaria).

11:30 - *Plasma Chemical Preparation of Nanodispersed Feroxide Powders* - G. Vissokov (Bulgaria)

13:00 Lunch break

Session - Applications

Chairs: J. Takada, Okayhama University (Japan) and S. Sabchevski, Bulgarian Academy of Sciences

14:30 - *Magnetic Nano- and Microparticles in Bioscience and Biotechnology* – I. Safarik, (Czech Republic)

15:30 - *Microwave Absorbers Based on the Feroxide Structures for Protection of Biological Objects* - J. Takada (Japan)

16:30 - Coffee break

17:00 - *Influence of Composition and Morphology on the Magnetic Properties of Nanostructured U-type Hexaferrites* – D. Lisjak, (Slovenia)

18.00 – *Using sub-millimetre radiation for the study of the living body* – T. Idehara (Japan) presented by S. Sabchevski

19:00 – Closing

Thursday, 25 September 2003

Departure

5. Final list of participants (including full, address, tel, fax, e-mail)

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6. Statistical information on participants (ages structure, countries of origin)

The total number of participants was 29 from 14 countries:

Belgium -	3	Romania -	1
Bulgaria -	13	Russia -	1
Croatia -	1	Slovenia -	1
Czech Rep.-	1	Switzerland -	1
France -	2	USA -	1
Germany -	1	Japan -	1
Ireland -	1		
Italy -	1		

The average age of participants was 47, the youngest one was 25 (L. Slavov, Bulgaria). There were 22 invited speakers, the youngest speaker was 29 (Ch. Andreeva, Bulgaria), the oldest one- 64 (G. Visokov, Bulgaria).

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