Warsaw, 31.10. 2004

Professor Marcin Leonowicz
Division of Structural and Functional Materials
Faculty of Materials Science and Engineering
Warsaw University of Technology
Woloska 141, 02-507 Warszawa, Poland.
Tel; (48 22) 660 8450, Fax (48 22) 660 8725
e-mail: mkl@inmat.pw.edu.pl
Award No FA8655-03-1-3042

European Office of Aerospace Research
and Development
223/231 OLD MARYLABONE RD
LONDON NW1 5TH

PERFORMANCE REPORT

Project title: **Functional polymer matrix fibers**
Period of performance: 1 November 2003 – 31 October 2004

Aim and scope of the project

The project is aimed at the processing of polymeric functional fibers containing magnetic and conductive fillers. For this purpose the production method of cellulosic fibers, by an ecological method, using organic solvent N-Methylmorphline-N-Oxide (NMNO) has been elaborated. The fibers were filled with soft and hard magnetic powders. Effect of volume fraction of the fillers on the physical, mechanical and chemical properties of the fibers was studied.

Introduction

XXI century technology requires new high performance materials with diverse properties, tailored to the particular applications. Nanostructured composites based on polymer materials appear to be promising, cost-effective candidates for development of new functional materials. This activity goes parallel with development of new ecological and economical processing methods of polymer materials such as carbon disulphide - free production of cellulose fibers. Soft, hard magnetic and conductive fibers can find applications as electromagnetic field shields, separators, filters, textiles and many others, especially in those fields where high mechanical properties are not decisive. Development of such materials requires studies of the processing - property relations and investigation of mechanical and physical properties.

Objectives

Systematic studies of the effect of the processing variables and structure on the physical properties of polymer fibers containing magnetic and electrically conductive modifiers.

Project description

The project comprises production of cellulose fibers, on a basis of the new and more environmentally friendly processing route elaborated at Lodz University. The equipment built enables production of fibers in a quantity of 1.5 - 2 kg / hour. The fibers were filled with magnetic additives such as: hard barium ferrite, nanocrystalline Nd-Fe-B alloy and soft ferrite powder. Structure, effect of volume fraction of fillers on the magnetic properties and chemical properties of the fibers were studied.

Ecological processing of cellulose fibers

An original method of the production of cellulose fibers, by an ecological method, using organic solvent N-Methylmorphline-N-Oxide has been developed. The equipment for this processing has been designed and built at the Department of the Man-Made Fibers, Technical University of Lodz. The process does not release gases to the atmosphere nor discharges effluents and waste products. The process is about ten times shorter that the traditional viscose method and the fibers are high quality. The properties of the cellulose fibers are as follows: tensile strength: 35-42 cN/tex (0.5-0,6 MPa), modulus of elasticity (wet) - 7-30 cN/tex, max. working temperature 150 °C. Functional fibers based on the cellulose can be applied in those areas where high mechanical properties are not of particular importance.

AQ F05-08-1991

Research tasks planned for the year 2004:

1. Study of the powder preparation conditions as well as the effect of particle size and volume fraction of fillers on the properties of the fibers.

2. Investigation of the method of introduction of sub-micron powder particles into cellulose solutions and formation of composite fibers exhibiting useful properties.

3. Elaboration of methods of the magnetic measurements of thin fibers.

5. Seeking application fields for the new materials.

Task sharing

The Polish side will produce the material using the new processing route elaborated at the Department of the Man-Made Fibers, Technical University of Lodz. The fibers will contain magnetic particles. Structure and the physical properties will be studied jointly at the Faculty of Materials Science and Engineering, Warsaw University of Technology and at Wright - Patterson Air Force Laboratory in Dayton.

Collaborators

1. Dr Richard Vaia, Air Force Research Laboratory, Materials and Manufacturing Directoriate, 2941 P Street, Suite 1, Wright-Patterson AFB, OH 45433-7750, E-mail:richard.vaia@wpafb.af.mil
2. Professor Marcin Leonowicz, Faculty of Materials Science and Engineering, Warsaw University of Technology, Woloska 141, 02-507 Warszawa, E-mail: mkl@inmat.pw.edu.pl
3. Professor Bogumil Laszkiewicz, Department of the Man-Made Fibers, Technical University of Lodz, S. Zeromskiego 116, 90-543 Lodz, E-mail: blaszkie@ck-sg.p.lodz.pl

Funds granted

25 000 USD

PROGRESS ACHIEVED

Task 1: Processing of soft magnetic cellulose fibers

Processing method of ferromagnetic cellulose matrix fibers on a basis of a novel, ecological method of cellulose production, using organic solvent N-oxide-N-methylmorphine and containing soft ferrite has been developed. Such composites can be used as soft magnetic materials, magnetic field shields, magnetic sensors etc., especially in those applications where high mechanical properties of fibers are not crucial (pure cellulose fiber exhibits tensile strength 0.5 MPa, modulus of elasticity 15.7 MPa and max working temp 150 °C). Effect of volume fraction of the ferrite powder on the microstructure, mechanical and magnetic properties has been studied. It has been found that the powder (7 μm mean particle size) is homogenously distributed within the fibers volume. The mechanical properties decrease with increasing powder content. Saturation magnetization increases proportionally to the ferrite content and for 5- wt% (23.8 vol %) of the powder contents the composite fiber attains a value of 0.1 T. However, apparently do to extended ball milling the coercivity of powder substantially increased up to 3.5 kA/m.
As the next step a special soft magnetic powder, used by the FerroxCube for shielding tiles, was prepared by ball milling and used for fibers preparation. In spite of the application of such material the coercivity did not decrease. This behavior we attribute to hindered domain wall movement in small, well isolated ferrite particles.

The results of this research were presented at the ICAMT'2004 - 3rd International Conference Advanced Manufacturing Technology, 11-13 May, Kuala Lumpur. The copy of the paper and specimens of fibers have been sent to AFRL/ML Dayton OH.

Task 2: Processing of hard magnetic fibers

Processing method of hard magnetic composite cellulosic fibers was developed. Such materials can be applied as flexible magnets, magnetic mats, magnetic filters, permanent magnet mats and textiles. This method applies ecologically friendly N-oxide-N-methylmorpholine (NMMO) as a cellulose solvent. The NMMO process, due to its versatility and stability, enables introduction up to 50 wt.% of a modifier to the spinning solution. In this study barium ferrite was used as the modifier. The powder exhibited homogeneous distribution within the fiber volume. The coercivity of the composite fibers did not depend on the powder content, whereas the remanence grew linearly with the powder content. This evidences that the modifier is stable in the NMMO solution. The thermal analysis (DSC and TGA) of the fibers did not show negative effect of the modifier on the thermal stability of the fibers. Small decrease of the mechanical properties of the fibers was the only drawback of the modifier addition. Generally, the fibers exhibit satisfactory magnetic and mechanical properties, which predispose them for various applications. This method has also the potential for further improvement by optimization of the powder parameters; both mechanical and magnetic properties of the composite material can be improved.

The results of this research were presented at the conference Composites 2004, April 21-23, Ustron, Poland (in Polish) and VIth SYMPOSIUM EL-TEX 2004, Electrostatic and electromagnetic fields - new materials and technologies, November 25-26, 2004, Lodz, Poland. The copy of the paper and specimens of fibers have been sent to AFRL/ML Dayton OH.

Task 3: Processing of conductive fibers – studies in progress

Conductive fibers are prospective materials for the production of lightweight, fabrics for shielding of electromagnetic radiation. A lot of effort has been aimed at the development of cellulose based conductive fibers, containing carbon nanofibers. The attempts of producing such composite materials have, however, been unsuccessful because the highly active surface of the carbon nanofibers led to the deterioration of the polymeric cellulose structure. Extensive research on the surface treatment of carbon nanofibers appears did not result in satisfactory improvement. Thus, recently we have been started a new research on conductive mats. Such materials are in a form of unwoven fabrics (Fig. 1). The fabrics are covered by polyester paste made from a mixture various types of acrylic resin and carbon nanofibers. The effect of the resin type, fibers content and structure of the composite material on the longitudinal and through resistivity will be studied in the light of application as shields of the electromagnetic field. The preliminary results showed longitudinal and through resistivity $3 \cdot 10^4$ and $4 \cdot 10^4$ Ωm, respectively. These studies will be carried out in 2005 y and will be mainly aimed at improvement of the electrical properties by optimization of the composition and processing variables.

Fig.1. Conductive unwoven fabric.

Publications

1. M. Leonowicz, M. Rubacha, P. Kulpiński, B. Łaszkiewicz, W. Kaszuwara, Z. Liu, H.A. Davies: *„Processing and Properties of Soft Magnetic Cellulose Fibers"*, Proc. International Conference on Advanced Materials and Processing Technologies, ICAMT 2004, Kuala Lumpur, Malaysia, 11-13 May 2004. p. 938-940.
2. W. Kaszuwara, M. Leonowicz, M. Rubacha, P. Kulpiński, B. Łaszkiewicz, P. Pawlik, Z. Liu, H.A. Davies: *„Composite Cellulosic fibers exhibiting magnetic properties"*, Kompozyty 2004, 21 – 23 April, 2004, Ustron, Poland (in Polish).
3. M. Rubacha, M. Leonowicz, P. Pawlik, B. Łaszkiewicz & P. Kulpiński, *„Cellulose matrix hard magnetic fibers"*, proc. VIth SYMPOSIUM EL-TEX 2004, Electrostatic and electromagnetic fields - new materials and technologies, November 25-26, 2004, Lodz, Poland – in print.

List of specimens delivered to Dr Richard Vaia, Air Force Research Laboratory

1. Samples of soft magnetic composite cellulosic fibers containing 10, 20, 30, 40 and 50 wt% of Mn-Zn ferrite.
2. Samples of hard magnetic composite cellulosic fibers containing 20, 30, 40 and 50 wt% of barium ferrite.
3. Three samples of unwoven fabrics, containing constant amount of carbon nanofibers and different types of acrylic resin.

Research tasks planned for 2005 y

Systematic studies of the composite variables and carbon fibers content on the electrical and shielding properties of unwoven polyester fabrics.
The properties will be studied versus:

- Fibers content
- Type of the resin applied

Measurements will comprise:

- Electrical resistivity
- Shielding properties for electromagnetic fields of various frequency
- Structure
- Mechanical properties

Funds claimed
25 000 USD

Professor Marcin Leonowicz

Marcin Leonowicz

Enclosures
Copies of publications

REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*	2. REPORT TYPE	3. DATES COVERED *(From – To)*
13-12-2004	Final Report	1 November 2003 - 14-Mar-05

4. TITLE AND SUBTITLE

Functional Polymer Matrix Fibres

5a. CONTRACT NUMBER
FA8655-03-1-3042

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)

Professor Marcin Kazimierz Leonowicz

5d. PROJECT NUMBER

5d. TASK NUMBER

5e. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Warsaw University of Technology
Division of Structural and Functional Materials
Woloska 141
Warszawa 02-507
Poland

8. PERFORMING ORGANIZATION REPORT NUMBER

N/A

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

EOARD
PSC 802 BOX 14
FPO 09499-0014

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
SPC 03-3042

12. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited. (approval given by local Public Affairs Office)

13. SUPPLEMENTARY NOTES

14. ABSTRACT

This report results from a contract tasking Warsaw University of Technology as follows: The Grantee will investigate incorporation of magnetic and conductive nanoparticles into polymer fibers to maximize magnetic susceptibility, electrical conductivity, and physical flexibility.

15. SUBJECT TERMS
EOARD, Conducting Polymers, Carbon nanomaterials, Nanoparticles

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18, NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			MATTHEW MORGAN, Lt Col, USAF
UNCLAS	UNCLAS	UNCLAS	UL	4	19b. TELEPHONE NUMBER *(Include area code)* +44 (0)20 7514 4505