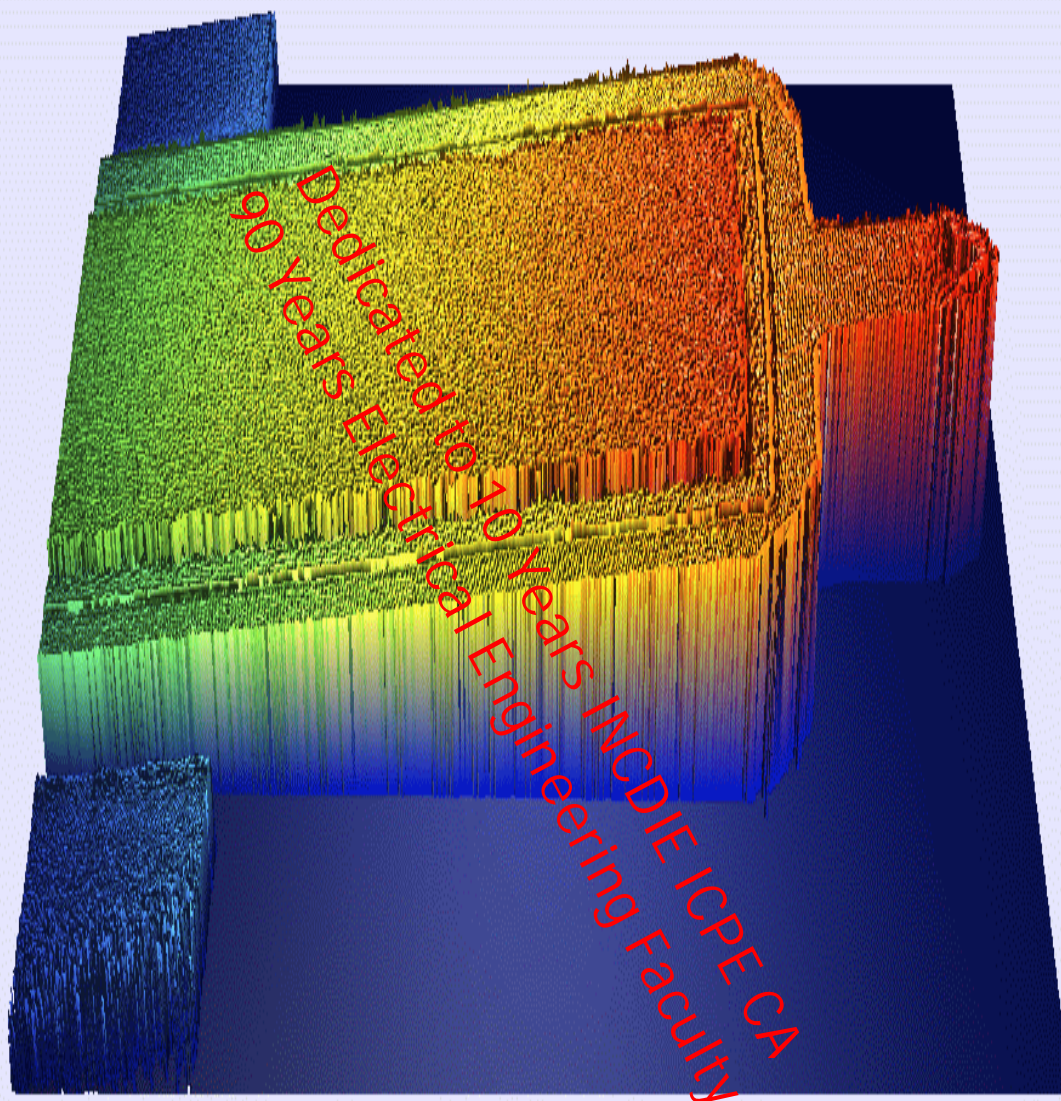


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In 2011 the Romanian electrical engineering community celebrates 90 year from the commencement of the Electrical Engineering (Electrotechnic) Faculty of Bucharest Polytechnic University and 10 year from the debut of the National Institute for Research and Development in Electrical Engineering – Advanced Research (INCDIE ICPE–CA), Bucharest.

Many remarkable teachers and scientific personalities through whom we remember Constantin Budeanu, Vasilescu Karpen, Constantin Bușilă, I. S. Gheorghiu, Alexandru Popescu, Remus Răduleț, Constantin Mocanu as emeritus professors at Electrical Engineering Faculty. On the recent period, Gheorghe Hortopan, Constantin Bălă, Alexandru Timotin, Andrei Țugulea, Alexandru Fransua, Augustin Moraru, Cezar Fluerașu, who represent a part of the prestigious teaching staff of this faculty, must not be forget.

INCDIE ICPE-CA represents a young and developing research institution which promotes new outlook by young scientific paradigm strategy. The new generation of Romanian specialists must to continue the fruitful tradition of our electrical engineering school.

This issue of Bulletin of Micro and Nanoelectrotehnology, is dedicated to these two definite events, which are milestones for the progress on electrical engineering.

*Editor in chief
Mircea Ignat*

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A New Challenge - the Nanoreliability

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Abstract – First, the terms used in the nano world are defined, in order to provide a comprehensible definition of nanoreliability. Then, the tasks that have to be fulfilled by nanoreliability are described, together with the possible tools. The domain is today on the main stream of the world researches in nanotechnologies, which is proved by the multitude of the important involved research groups and events dedicated to this subject.

Index Terms – failure, modelling, nanomaterials, nanotechnology, NEMS, reliability.

I. INTRODUCTION

The common sense says that in any definition of a new word, all the used terms must be previously defined. Consequently, trying to begin this paper with a definition of the reliability at nano level (in one word: *nanoreliability*), we have to solve a major issue. Yes, the reliability could be easily defined as the functionality and safety of any product or process during its whole intended life. But at nano level the definitions were not established yet. So, first, we have to describe (without claiming to provide the best definitions) the main terms.

Basically, the nanotechnology is aimed to produce objects with dimensions between 0.1 and 100 nm. In 1959, Richard Feynman delivered a conference at the American Physical Society, speaking about the use of integrated circuits (not yet invented!) in computers and about decrypting the DNA molecules. Among other subjects, Feynman predicted the development of individual atoms, idea materialized much later. For the first time, the term “nanotechnology” was used by Norio Taguchi in 1974, in a conference about improving the manufacturing accuracy. He said the “nanotechnology” will attain an accuracy of around 100 nm. In 1989, Don Eiger employed a Scanning Probe Electron Microscope (SPM) for writing the word “IBM” with xenon atoms. For the first time, somebody succeeded in depositing atoms in a given place.

In fact, in nanotechnology, there are two ways to act [1]:

a) The current approach is to diminish the dimensions of structures designed at higher scale, i.e. a “top-down” process, continuing the existing researches in microtechnologies, with some specific issues: increasing the number of transistors on chip (according to Moore’s Law), using in lithography light sources with a smaller wavelength than visible one, decreasing the dimensions of transistors (down to 70 nm), etc.

b) The reverse process, the “bottom-up” one, is close to Don Eiger attempt, meaning to assemble and synthesize by chemical processes (inspired from the biology) structural blocks at nano scale, building complex architectures, with new electron and optical properties. This process is also called Molecular Nanotechnology (MNT), and the structures are built atom-by-atom or molecule-by-molecule, the term “self-assembling” being also used. The main advantage of the “Bottom-up” approach is a reduction of the fabrication costs, but for the moment this is only a long term challenge. On medium term, a hybrid strategy, by combining the two approaches seems to be the best solution.

This document provides an example of the desired layout for a published MNE technical paper and can be used as a Microsoft Word template. It contains information regarding desktop publishing format, type sizes, and typefaces. Style rules are provided that explain how to handle equations, units, figures, tables, abbreviations, and acronyms. Sections are also devoted to the preparation of acknowledgments, references, and authors’ biographies.

II. TERMINOLOGY

Currently, there are three terms used by the nanoscale technologies, which must be correctly defined, in the aim to avoid confusions:

Nanotechnology is the assembling of individual atoms, molecules or groups of molecules in structures aimed to create materials or devices with new properties. Any

technological development at nanoscale that produces dimensional reduction or atoms / molecules manipulation is aimed. At this scale, the current rules of physics and chemistry are no longer valid. Such characteristics as colour, mechanical resistance, conductivity and reactivity may substantially differ at nano and microscale. For instance, the carbon nanotubes are 100 times more resistant as steel, but 6 times lighter. As we all know, the nanotechnologies are already used in many fields, such as: Information technology, Medicine, Material fabrication, Aeronautics, Environment protection, Agriculture, Biotechnology, etc.

Nanoscience is an extension of the existing science, for exploring the very small dimensions, by putting together many disciplines. The main applications are: nanomaterials, nanochemistry, nanobiology, nanophysics, etc.

Nanoengineering is an extension of the engineering fields at nanometric scale (nanofabrication, nanodevices).

The products of nanotechnologies are the *nanostuctures*, i.e. nanomaterials and nanodevices. The nanomaterials may enter in the composition of nanodevices (especially due to their specific properties, different to the properties of the same material at macroscopic scale). The nanodevices may be “classical” type of electronic devices (integrated circuits, transistors, etc.), or electro-mechanical nanosystems, called Nano Electro Mechanical Systems – NEMS (by analogy with MEMS – at micro level).

So, one may say that there are three main directions of nanotechnology development:

Nanomaterials – re-structured materials at nano level, with physical and chemical properties which are very often different (improved) versus the initial material; they are composed by nano-objects (having at least one dimension smaller than 100 nm) with specific properties: *i*) “classical” nanomaterials, such as: nanoparticles of black carbon, silica precipitate, silica gels or carbonates; they are already commercialized by many companies; *ii*) “new” nanomaterials (nanostructured by design): nanoparticles (aluminium oxide, colloidal silica, zinc oxide), carbon nanotubes, quantum dots, nanowires, nonporous materials.

The other two directions in nanotechnologies are linked to nanodevices, such as:

Nanoelectronics – which means creation of nanoelectronic architectures, with special design rules, taking into account also to the reliability of the future product; these are discrete and integrated devices with much smaller dimensions, where specific problems arise, different from those at the micro level; here the so-called quantum structures are included, which are in fact semiconductor devices with the electrons confined in all three dimensions;

Electromechanical systems at nano level (sensors + actuators + integrated circuits), the so-called *nano-MEMS* or *NEMS (Nano-Electro-Mechanical-Systems)*, similar to MEMS, but much smaller, which include also devices for microfluidics or bio-compatible ones, for biomedical applications [2]. In 2000, the first very large scale integrated (VLSI) NEMS device was demonstrated by researchers from IBM. Its premise was an array of Atomic Force Microscope (AFM) tips which can heat / sense a deformable substrate in order to function as a memory device. In 2007, the International Technical Roadmap for Semiconductors (ITRS) contains NEMS Memory as a new entry for the emerging research devices section; an indication that the semiconductor industry is actively considering the technology for implementation in the near (15 years) future [3].

Each of these new development directions at nano level have arisen specific issues about testing methodologies, reliability, metrology, etc. *The nanoreliability* is the discipline focused on identifying and solving the specific issues linked to the reliability of the products obtained by nanotechnologies.

III. ABOUT NANORELIABILITY

Reliability theory and physics of failure of macro- and micro-world are only partially applicable on the nanometre scale. Physical processes not scaling linearly with size and time dramatically change mass and heat diffusion, electrical conductivity, reaction kinetics, corrosion processes, etc. [4]. Fatigue, friction, damping, wear-out and repair mechanisms have a different physical meaning on atomic or molecular scales. Redundancy and correlation of quantum systems require quantum statistics of states and entanglement of wave functions. Certainly, the general reliability theory remains unchanged, but when it is used at nano level, some adjustments must be made.

The future nanotechnology products, i.e. products including nanoscopic components or nanoscale modified materials, have to withstand thermo-mechanical environmental and functional loading during life time. Also, sufficient mechanical reliability of non nanoscale, but microscale components can be reached only by better understanding and modifying of nanoscale driven processes of material deterioration, which finally lead to device failure. Because of the scaling, traditional methods for testing and simulative reliability assessment have to be refined or replaced by new, appropriate tools [5].

Also, the use of destructive testing continues [6], but the non-destructive testing receives new valences at nano level. In NEMS metrology, the problem of reconstructing 3D images at nano level must be solved. At nano level, the modelling of materials and structures must be made on new grounds. For nano devices used in bio-medical applications, the reliability requirements are extremely severe. In one word, to pass from MEMS to NEMS implies to solve complex issues about the degradation phenomena. If for MEMS these issues are known in principle, for NEMS new phenomena arise, due to:

- Modifying of the physical and chemical properties for nanostructured materials (modelling of these material properties from the nano scale to the final macroscopic form must be done);

- Transitory faults arising due to the diminishing of the noise tolerance at much smaller levels of working current and voltage;

- Defects produced by the ageing in using molecular techniques for creating nano devices;

- Manufacturing faults, which become, at nano level, much more significant.

Being at the crossing of many fields, (chemistry, physics, biology, electrical engineering, computer science, material science), the nanotechnologies are based on the deep understanding and control of the structure at atomic, molecular and supra-molecular level, where the materials and systems may have new and improved physical and chemical properties. The main current directions in the research on the reliability of nanotechnologies are:

- Introduction of concepts and technical terms of reliability to nanotechnology in an early state [7];

- Reliability of nanostructured materials (identifying physical failure mechanisms);

- Design of system reliability at nano scale, including design for reliability of nanodevices;

- Determination of quality parameters of nanodevices, failure modes, and failure analysis including reliability testing procedures, and instrumentation to localize nanodefects [4];

- Reliability tests and analysis of failure modes (during fabrication process and operation [7]) for nano-devices and nanosystems;

- Modelling ageing, degradation, failure rates for nanodevices and nanosystems;

- Issues about the quality and reliability of nanoproduct manufacturing,

- Reliability standards for products and systems at nano scale,

- Reliability predictions for various manufacturers of nanoproducts.

The nanoreliability becomes more and more a favourite subject for scientific conferences, mainly after 2003. Some examples are given in the following [1]:

- On August 17-19, 2004, The National Institute of Standards and Technology (agency of the U.S. Department of Commerce's Technology Administration) and the University of Colorado Department of Mechanical Engineering have organized a Workshop on Reliability Issues in Nanomaterials;

- The Conference IFORS (International Federation of Operational Research Society), held in Hawaii, between July 11 and 15, 2005, had a section entitled Quality, Reliability and Statistics in Nanotechnology;

- The reliability at nano level is one of the subjects of the conference IEEE Nano 2006, July 16-20, Cincinnati, Ohio, SUA, which was organized by IEEE Nanotechnology Council;

- SPIE's 13th Annual International Symposium on Smart Structures and Materials, which was held between 26 February and 2 March 2006, has had a section entitled Testing, Reliability, and Application of Micro- and Nano-material Systems.

As far as the importance given at world level to the nanotechnology field is concerned, the best situation is in USA. At the symposium organized on June 17-18, 2004, in Alexandria, Virginia, USA, and led by Dr. Mihail Roco, he mentioned that the American investments in nanotechnologies exceeded, in 2004, 3.5 billions USD. An example about how to spend this funds

also for nanoreliability: Dr. Michael Orshansky, from the University of Texas (Austin), declared receiving a 400,000 USD grant for developing software for designing reliable chips at nano scale. At these dimensions, said Prof. Orshansky, the electrical behaviour of the transistors could be obtained based on the probabilistic theory.

Talking about the European researches in nanotechnologies, one may say that the 6th Framework Program of the European Union, initiated in 2003, already promoted a series of Network of Excellence (NoE) in micro- and nanotechnologies. Among these, we may found the NoE Design for Micro and Nano Manufacture - Patent-DfMM (FP6 project / 2004-2008), with 24 research groups from 14 European countries. Romania is represented by IMT-Bucharest, which was the leader of the Reliability & Characterisation Cluster of the NoE, being directly involved in the European researches in nanoreliability.

On the other side, in Switzerland, Eidgenössische Materialprüfungs- und Forschungsanstalt (EMPA), from Zurich (Switzerland), built in 2002 a network called Nano Reliability Network. Among network subjects are: failure physics for nanostructured materials and devices, reliability assessment and failure rate of nanosystems (based on stochastic modelling), procedures for reliability testing and failure analysis, quality assurance and standardization of nanodevices, design approaches for reliable nanosystems.

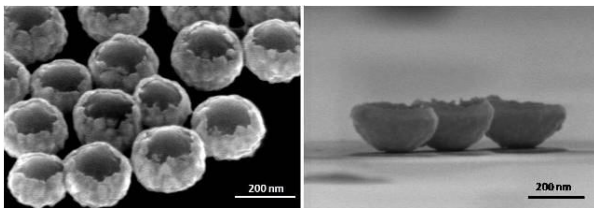


Fig. 1. SEM image of Au semishells (left) and hollow gold nanobowls (right), both fabricated by IMEC [8].

Also, IMEC, the well-known research institute from Leuven (Belgium), has notable results in nanotechnologies. Various nanostructures with at least one dimension between 1 and 100nm including nanospheres, nanowires, nanotubes, nanoholes, nanostars, nanoshells, and nanorods have been fabricated for different applications due to their unique optical, electrical, mechanical, catalytic, and magnetic

properties. Recently, IMEC focused on the fabrication of different Au nanoparticles. Recently, a method involving ion milling has been established to fabricate reduced-symmetrical Au semishells P17488. Furthermore, a versatile method was reported, aiming to fabricate hollow gold nanobowls and complex gold nanobowls (with a core) based on ion milling and vapour HF etching P17487 (see Figure 1). In IMEC, the reliability of nanostructures became a research subject for the Reliability Group led by Dr. Ingrid De Wolf.

IV. CONCLUSIONS

Today, the nanoreliability, a new and interesting subject, represents a major challenge for the scientific world: many papers are written on this subject, many conferences are organized and many special issues of prestigious journals are published. Also, international networks of excellence cover this field.

We think that the next several decades will see unprecedented levels of integration of emerging nanomaterials, nanoelectronic architectures, and nano-MEMS platforms. This will pose severe challenges for testing, reliability, and metrology techniques required to support such development. The integration of various materials and technology approaches has, and will continue to result in the combination of heretofore field-specific testing, reliability, and metrology methodologies.

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VI. BIOGRAPHIES

Marius Bazu was born in Ploiesti, on October 12, 1948. He graduated in 1971 and received Ph.D. degrees in electronics from Bucharest Politehnica University in 1994. He is Head of Reliability Laboratory, author of three books published by J. Wiley & Sons (2011), Artech House (2009) and Springer Verlag (1999) and of more than 130 papers in international journals and contributions to international conferences. His recent research interests include methods for building, assessing & predicting the reliability for microsystems and nanostructures. Leader of many national projects, he was leading Reliability & Characterisation Cluster of the NoE "Patent-DfMM" and he is in the Board of the network "European Microsystems Reliability" (EUMIREL). Author of papers and referent of the journals: *IEEE Trans. on Reliability*, *Sensors*, *IEEE Electron Device Letters*, *IEEE Trans. on Components & Packaging* and *Microelectronic Reliability*.

Virgil Ilian was born in Cluj, on November 17, 1947. He graduated in 1971 from Bucharest Politehnica University. He is Senior Researcher in the Reliability Laboratory, with recent research interests in electrical characterization of materials and devices, testing methods, failure physics for microsystems & microelectronic devices. He designed and developed equipment for electric characterization and reliability testing for integrated circuits. Author of more than 50 publications, he is also an active part in the field of standardization, as the president of National (Romanian) Committees TC 47 "Semiconductor Devices" and TC 91 "Electronics Assembly Techniques" of CNR CEI.

Dr. Lucian Galateanu was born in Iassy, on November 20, 1947. He graduated in 1970 and received Ph.D. degrees in electronics from Bucharest Politehnica University in 1993. He is Senior Researcher with recent research interests in testing methods, reliability physics, reliability evaluation and improvement for microsystems. Leader of national projects on Selective acceleration of semiconductor chips ageing by laser irradiation, Building-in Reliability methods, reliability evaluation of electronic components, etc. Author of more than 80 publications in journals and conferences: *Microelectronics and Reliability*, *ESREF*, *ICONE*, etc.

Tuning of controllers for processes based on identification results with application to the micro welding

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Abstract - In this publication are described identification methods specific to industrial processes which can be theoretical or experimental highlighting the models of order 1[2] or two for modeling of distinct known processes. For the identification of processes are known distinct methods, in the publication is highlighted Matlab as the main source for identifying the processes [3]. Starting from the identification methods of processes in the publication are described methods for tuning controllers based on identification results highlighting a method for determining the process parameters based on experimental identification results. According to normalized lag time and the normalized amplification factor a tuning method is chosen specific to the process. Using these methods for tuning controllers the problem of tuning the controller is solved for a process of microwelding determination with laser fascicle starting from identification results experimentally determined.

Index Terms – automation process, identification methods, laser , process control, tuning of controllers, welding.

I. INTRODUCTION

Identification methods based on step response are the most used methods for system identification, specially for industrial processes (Gustavsson, 1973), [2]. The idea of step response was introduced by Kupfmuller(1928) [2] which proposed also the first method for estimating the parameters of a system of order 1 with time lag starting from step response. This graphical technique , described by Oldenbourg and Sartorius (1948) and later by Rake (1980) and Unbehauen and Rao (1987), implies the drawing of a tangent at the inflexion point of the response curve and formed the base for a number of similar methods for models of order 1 or 2. The models of order 1 or 2 are described by the following two equations:

$$G(s) = \frac{K}{1 + Ts} e^{-\tau s} \quad (1)$$

or

$$G(s) = \frac{K}{(1 + T_1s)(1 + T_2s)} e^{-\tau s} \quad (2)$$

where K is the gain inherent associated with the process, τ is the lag time, T, T_1, T_2 are time constants.

The models characterized by the transfer function 1, are typical for pressure, level or flow control applications; also the method can be used at heating processes. The models of type 2 are specific to temperature processes with more elements for storing the energy Strejc (1959) proposed an improvement of Kupfmuller method in which the parameters are estimated based on two points well chosen on either side of the inflexion points. A big number of such graphical methods are available in the literature and they were used effectively in real applications [2].

The industrial processes are technical systems oriented on objects which have the task to change the inputs (raw materials, energies, information) in desired outputs (in general goods).

For obtaining the desired performances (quality, quantity) it is necessary to accomplish the transformation process using the automated control or monitoring.

For this goal it is necessary an equipment attached to the process (called automation equipment) composed from a number of structural elements, which solve the following problems: acquisition of data from the process, takes the good decision regarding the goal and acquisitioned dates, accomplishes the interventions which has influence on the process until the desired results are obtained.

The choice and adoption of the automation equipment necessitates to know the properties of the respective process.

To the present moment the properties of the industrial process necessary for choosing the controller type can be found using mathematical models. Usually these are differential equations (with variable or constant coefficients), transfer functions which group the process input/output parameters showing the static and dynamic behavior.

The operation from which the mathematical model is chosen is called identification [1].

The identification can be [1-3]:

- **Theoretical:** the mathematic model is obtained applying the governing laws from the analyzed process; method which combines the input/output parameters of the process.
- **Experimental:** on the basis of experimental tests obtained in the process time and acquisitioned data from the process, the proper mathematical model is obtained.

II. TECHNICALWORK PREPARATION

A. Methods for controller tuning based on identification results [8, 9].

Practical methods exist for tuning of optimal accord parameters based on $\frac{\tau}{T_p}$ rapport characteristic for a process with lag time τ and a time constant T_p :

$$H_p(s) = \frac{K_p e^{-\tau s}}{T_p s + 1} \quad (3)$$

For determining the parameters which characterize the process behaviour at a step command signal the following stages must be completed:

- a) a step from u_0 to u_{st} is applied at t_0 moment at the process input which is in the stationary regime characterized by u_0, y_0 , in open loop ,which can be between (10-20%) of the whole scale;
- b) the new value of stationary regime for y_{st} output is computed and the process response is registered at $\Delta u = u_{st} - u_0$ input;

c) the parameters of mathematical model are computed with the following relations(Fig.1):

$$K_p = \frac{y_{st} - y_0}{u_{st} - u_0}, \tau = t_1 - t_0, T_p = t_2 - t_1 \quad (4)$$

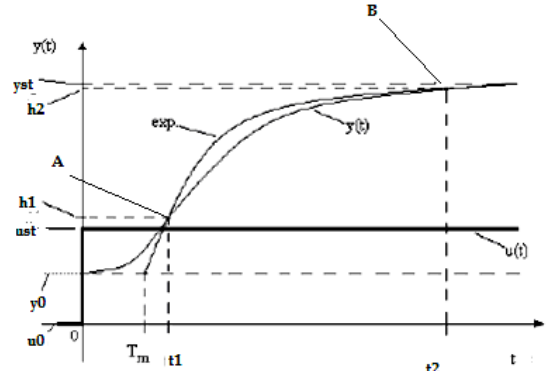


Fig. 1. First order process identification: u_0, y_0 - input/output initial values, u_{st}, y_{st} - input/output final values $h_2 = 0.9 * (y_{st} - y_0)$. A –inflexion point of the experimental curve obtained t_1, t_2 - times of points A and B.

With the parameter values K_p, T_p, τ obtained experimentally the optimal parameters of PID controller are computed using different relations.

The most known relations for computing the optimal PID parameters of the controller are[5]:

- Ziegler-Nichols.
- W. Oppelt.
- Kappelovici.
- Chien, Hrones, Reswich.

B. Tuning of controllers based on identification results with application to an integrated system for welding determination with laser fascicle of polymeric materials, of monitoring and control in real time of welding process.

The micro welding process structure, on which the identification and tuning is done based on identification results contains the following functional blocks:

- Microwelding module with laser fascicle.
- Translation unit on ox axis, translation unit on oy axis.
- Micro sensor for temperature reading using non contact method.
- Data acquisition module.
- Laptop with specialized software.

- Equipment for editing the measuring bulletin.

From the functional point of view, the role of this controller is to control the power of an laser equipment with the help of a command and control system with specialized software (fig. 2), having the task to maintain a relatively constant temperature in the impact zone with laser fascicle in time period.

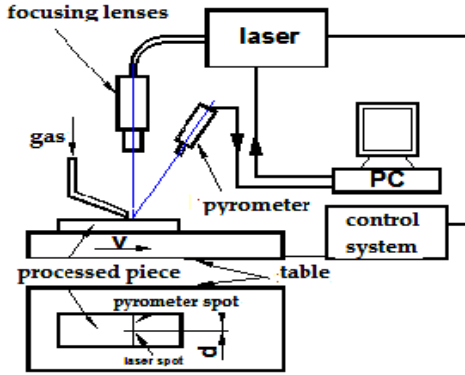


Fig. 2. Functional schema of the process.

All the modern systems for command and control of processes, projected in the last years in the European Union countries are provided with non contact transducers and measuring equipment, data acquisition and processing having as base modern computing systems [8].

Using the theoretical elements presented above the process and controller parameters can be determined starting from identification results. Below is presented the identification and tuning of the welding process.

$$y_0 = 120, u_0 = 0.1, u_{st} = 1, t_0 = 0.1$$

$$t_1 = 2.1s \quad t_2 = 7.5s$$

K_p is determined as follows:

$$K_p = \frac{y_{st} - y_0}{u_{st} - u_0} = \frac{400 - 120}{1 - 0.1} = \frac{280}{0.9} = 311.11 \quad (5)$$

τ is determined as follows:

$$\tau = t_1 - t_0 = 2.1 - 0.1 = 2s \quad (6)$$

T_p is determined as follows:

$$T_p = t_2 - t_1 = 5.4s \quad (7)$$

Where K_p is the gain, τ - lag time of the process, T_p - time constant of the process, u_0 - initial input value of the process, u_{st} - stationary value applied at the process input, y_0 - initial value of

the process output, y_{st} - stationary value of process output

From here the transfer function of the controlled process is:

$$H_p(s) = \frac{K_p e^{-\tau s}}{T_p s + 1} = \frac{311.11 e^{-2s}}{5.4s + 1} \quad (8)$$

Using the trapeziums method $e^{Ts} \approx \frac{1+s\frac{T}{2}}{1-s\frac{T}{2}}$ (9) of

approximation results:

$$e^{-2s} \approx \frac{1-s}{1+s} \quad (10)$$

The process transfer function becomes:

$$H_p(s) = \frac{311.11 \frac{1-s}{1+s}}{5.4s + 1} = \frac{311.11 - 311.11s}{5.4s^2 + 6.4s + 1} \quad (4)$$

The Nyquist diagram of the system is presented bellow (Fig.3).

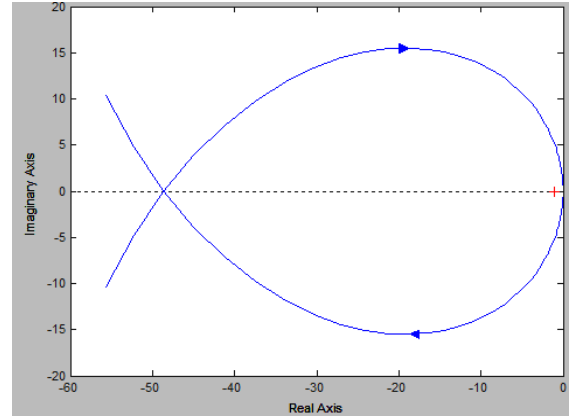


Fig.3 Nyquist diagram of the micro welding process.

The normalized lag time is:

$$\theta_1 = \frac{\tau}{T_p} = \frac{2}{5.4} = 0.37 \quad (11)$$

Using the Nyquist diagram (figure 3) is found the point in which the Nyquist diagram cuts the real axis:

$$H_{1\tau}(j\omega_\tau) \approx -47 \quad (12)$$

The normalized amplification factor is:

$$K_1 = K_p K_{R0} = \frac{H_{1\tau}(0)}{|H_{1\tau}(j\omega_\tau)|} = \frac{311.11}{|-47|} = 6.61 \quad (13)$$

Using the Ziegler – Nichols methods for tuning the controller the following controllers can be found:

- **For a P controller:**

$$K_{Ropt} = \frac{1}{K_p} \frac{T_p}{\tau} = \frac{1}{311.11} \frac{5.4}{2} = 0.008 \quad (14)$$

- **For a PI controller:**

$$K_{Ropt} = \frac{0.9}{K_p} \frac{T_p}{\tau} = \frac{0.9}{311.11} \frac{5.4}{2} = 0.0078 \quad (15)$$

$$T_{iopt} = 3.3\tau = 3.3 * 2 = 6.6$$

- **For a PID controller:**

$$K_{Ropt} = \frac{1.5}{K_p} \frac{T_p}{\tau} = \frac{1.5}{311.11} \frac{5.4}{2} = 0.013$$

$$T_{iopt} = 2.5\tau = 2.5 * 2 = 5 \quad (16)$$

$$T_{dopt} = 0.5\tau = 0.5 * 2 = 1$$

where K_{Ropt} is the optimal constant for the proportional component of the process controller, T_{iopt} - is the optimal constant of the integral component of the controller and T_{dopt} is the optimal constant of the derivative component of the process.

IV. CONCLUSIONS

Industrial process identification starting from the step input is a popular method and very often used. However there are still challenges in applying this methods in real implementations.

In the first part of the publication the principal methods of identification were described which can be theoretical and experimental or a combination of both methods.

The transfer functions of order 1 and 2 were presented specific to different industrial processes, highlighting that the identification methods that use mathematical models are hard to use and in this case the process identification using Matlab or Labview is recommended. In the last part of the paper tuning methods of control based on identification results are presented, with a numeric example for an integrated system of micro welding determination with laser fascicle. According to the theory using the normalized lag time θ_1 and normalized amplification factor K_1 , the Ziegler –Nichols method for tuning the micro

welding process was chosen, highlighting the optimal parameters for P, PI, PID controllers.

V. ACKNOWLEDGEMENT

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Electric Induction Micromotor - Torque Estimation

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Abstract – An analytical method for torque estimation of an electric induction micromotor is presented. The actuation is based on the interaction between 3-phase rotating field generated by the stator electrodes and rotating field generated by the electric charges induced into the rotor poles. After calculation of the interaction energy-angular displacement curve, the torque is then found by using the principle of virtual work, where in the torque is given by the partial derivative of the stored energy vs. angular displacement. The average torque over one electrical period is finite only when the stator and rotor fields are synchronized. The proposed method is applied to estimate the torque of a microactuator simulated with a FEM approach; there is a good agreement.

First, the terms used in the nano world are defined, in order to provide a comprehensible definition of nanoreliability. Then, the tasks that have to be fulfilled by nanoreliability are described, together with the possible tools. The domain is today on the main stream of the world researches in nanotechnologies, which is proved by the multitude of the important involved research groups and events dedicated to this subject.

Index Terms – electrostatic actuators, MEMS, torque computation.

I. INTRODUCTION

The greatest promise of microelectromechanical systems (MEMS) lies in their ability to produce mechanical motion on a very small scale. A variety of techniques for achieving microactuation have been developed, including electrostatic, electromagnetic, ultrasonic, hydraulic and thermal method. The electrostatic microactuator is less affected by scaling and from the fabrication point of view is compatible with integrated circuits fabrication processes.

Two types of electrostatic micromotors have already been studied and built: variable capacitance micromotors and electric induction micromotors (MESI).

MESI is based on the interaction between 3-phase rotating field generated by the stator electrodes and electric charges induced into the rotor poles.

With traditional numerical techniques like FEM and BEM are computed forces and torques by means Maxwell's stress tensor or virtual work method [1]. The use FEM or BEM in MEMS analysis has a significant drawback: the need of constructing volume meshes that increase the computational cost.

In [2] the rotor is modeled as a perfect conductor that leads to a considerable reduction of the mesh size. Once the field solution has been obtained, the driving torque on the rotor is evaluated by means of the so called equivalent charge method. When compared to simulations obtained with a commercial FEM electromagnetic code using a classical high-permittivity formulation, this method proves to be accurate and exhibits an overall reduction of computational time.

In this paper, an analytical method for torque estimation of MESI is presented. With this method is estimated the torque of MESI presented in [2]. There is a good agreement between two results.

II. Estimation of MESI driving torque

The bidimensional model of a MESI is depicted in Fig. 1. The stator includes a 3-phase system of electrodes with $2p_1$ poles under 3-phase voltage with r.m.s. value U and pulsation ω_1 .

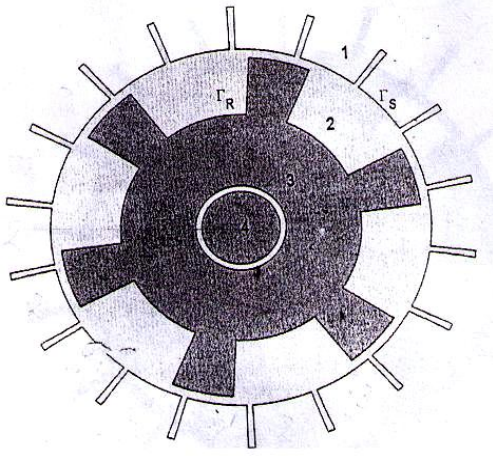


Fig. 1. MESI's bidimensional model. The four regions in the figure are: stator electrodes (1); air gap (2); the rotor (3); rotor shaft (4), [2].

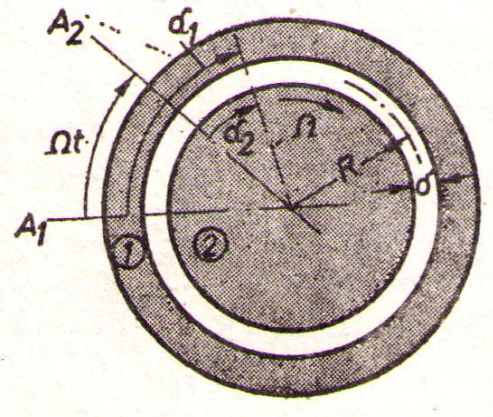


Fig. 2. Explanatory for reference axis [3].

The stator generates a tangential electric field which has a rotating spatial distribution:

$$e_1 = E_{m1} \sin(\omega_1 t - p_1 \alpha_1 - \varphi_1) \quad (1)$$

The stator wave induces an electric charge (electrostatic induction) on the surface of the salient-pole rotor. The surface-charges generate a tangential electric field which has a rotating spatial distribution.

$$e_2 = E_{m2} \sin(\omega_2 t - p_2 \alpha_2 - \varphi_2) \quad (2)$$

Between spatial coordinates (α_1, α_2) and angular displacement of the waves (φ) there are following relationships:

$$\alpha_1 = \alpha_2 + \Omega t; \varphi = \varphi_1 - \varphi_2 \quad (3)$$

where Ω is the relative angular speed.

The instantaneous electrostatic torque (m) is found by using the principle of virtual work

$$m = \left(\frac{\partial W_e}{\partial \phi} \right) \Big|_{E_{m1}; E_{m2} = \text{const}}; \quad (4)$$

$$W_e = \frac{LR}{2\epsilon_0} \int_0^{2\pi} (e_1 + e_2)^2 d\alpha$$

where W_e is the electrostatic energy, R is the average radius, L is the length of armature.

It's easy to demonstrate that:

- the machine can develop an instantaneous torque only when

$$p_1 = p_2 = p \quad (5)$$

- the average torque (M) over one electrical period (T)

$$M = \frac{1}{T} \int_0^T m dt \quad (6)$$

is finite only when the waves are synchronized

$$\omega_1 = \omega_2 + p\Omega \quad (7)$$

and this value is

$$m = M = -\pi\epsilon_0 \delta R L E_{m1} E_{m2} \sin \varphi \quad (8)$$

Remark: the electrostatic forces acting on the rotor are exerted in the three major direction: axial, radial and tangential. Because of the cylindrical geometry only tangential forces generate mechanical torque.

III. THE AMPLITUDES OF THE ELECTRIC WAVES

The structure of the armature electrodes is electrostatically equivalent with a capacitance grid by capacitance coefficients C_{ij} .

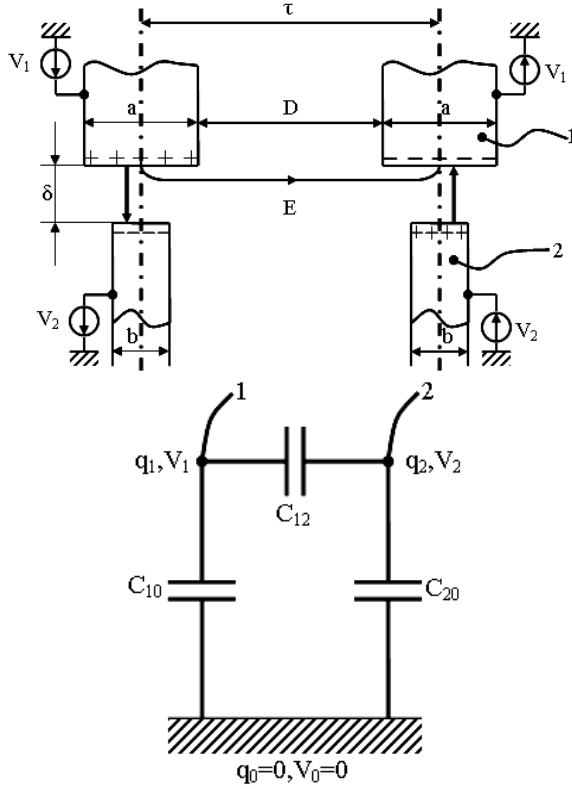


Fig. 3. a) MESI's unidimensional model; b) Equivalent capacitance grid.

C_{12} is the capacitance of a plane capacitor and it's given by

$$C_{12} = \frac{\epsilon_0 b L}{\delta} = C_{21} \quad (9)$$

C_{10} and C_{20} are the capacitances of a plane capacitor with coplanar armatures. Approximating field lines with arcs and segments, under assumption $\pi a \ll D$, they are given by

$$C_{10} = \frac{\epsilon_0 L a}{\tau}, C_{20} = \frac{\epsilon_0 L b}{\tau} \quad (10)$$

The problem is to calculate the capacitance coefficients C_{sij} in the service s:

$$s : q_1 + q_2 = 0 \quad (11)$$

Maxwell's relationships for three capacitances system with $V_0 = 0$ and $q_0 = -(q_1 + q_2) = 0$ (complete system) are

$$\begin{cases} q_1 = C_{12}(V_1 - V_2) + C_{10}V_1 \\ q_2 = C_{21}(V_2 - V_1) + C_{20}V_2 \end{cases} \quad (12)$$

which lead to

$$\begin{cases} \left(\frac{V_1}{V_2}\right)_s = -\frac{C_{20}}{C_{10}} \\ C_{s12} = \left(\frac{q_1}{V_1 - V_2}\right)_s = C_{12} + \frac{C_{10}C_{20}}{C_{10} + C_{20}} = C_{s21} \\ C_{s10} = \left(\frac{q_1}{V_1}\right)_s = C_{10} + C_{12} \frac{C_{10} + C_{20}}{C_{20}} \\ C_{s20} = \left(\frac{q_2}{V_2}\right)_s = C_{20} + C_{12} \frac{C_{10} + C_{20}}{C_{10}} \end{cases} \quad (13)$$

so that the amplitudes of the electric waves are given by

$$E_{m1} = 1,5 \cdot \sqrt{2} \cdot V_{1s} / \tau, E_{m2} = 1,5 \cdot \sqrt{2} \cdot V_{2s} / \tau \quad (14)$$

Factor 1,5 arises from the phase character of the applied voltage of the stator electrodes.

IV. ANALYSIS MODEL

A FEM method for the numerical electromagnetic analysis of a MESI is presented in the paper [2]. The geometry is: $R = 75 \mu m$, $\tau = 234 \mu m$, $a = 78 \mu m$, $b = 26 \mu m$, $D = 156 \mu m$, $L = 250 \mu m$, $\delta = 3 \mu m$. The electrodes are supplied by means of a 3-phase square-wave voltage at a level of $U = V_{1s} = 100V$. The maximum computed value of the torque is

$$M_{\max} = 3,3 \cdot 10^{-12} Nm \quad (15)$$

For the shown geometry, by using formula from §3, it's deduced:

$$\begin{aligned} C_{12} / L &= 7,7 \cdot 10^{-11} F / m, \\ C_{10} / L &= 0,3 \cdot 10^{-11} F / m, \\ C_{20} / L &= 0,1 \cdot 10^{-11} F / m, \\ C_{s12} / L &= 7,8 \cdot 10^{-11} F / m, \\ C_{s10} / L &= 31,1 \cdot 10^{-11} F / m, \\ C_{s20} / L &= 10,4 \cdot 10^{-11} F / m \end{aligned}$$

which lead to

$$V_{2s} = -300V_{ef},$$

$$q_{1s} = -q_{2s} = 30 \cdot 10^{-9} C / m,$$

$$E_{m1} = 9,1 \cdot 10^5 V / m,$$

$$E_{m2} = 27 \cdot 10^5 V / m.$$

By means (8) it's deduced $M_{\max} = \pi \varepsilon_0 \delta R L E_{m1} E_{m2} = 3,8 \cdot 10^{-12} Nm$, a result in agreement with [2].

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Electrothermic Carbon Microactuator

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Abstract - This paper provides a preliminary study regarding electrothermic behavior of a flaky layered carbon material. Some electrothermic microactuator functional structures are described and as a new approach theoretical aspects about designing of thermal microactuators are discussed.

Index Terms - Actuators, electrothermic, displacements, carbon, layered compounds.

I. INTRODUCTION

Electrothermic micro-actuators are considered as a new class of actuators [1, 2, 3, 4] which can provide both large displacements and forces.

This paper highlights the potential of flaky layered carbon materials to act as electrothermic actuators. These devices are easy to manufacture and could be easily integrated into various electromechanical systems. These layered carbons should be applicable potentially in technical fields such as electronics, catalytic or hydrogen-storage systems. In this study will be presented some experimental features of flaky layered carbons designed as electrothermic actuators (displacements vs. applied current fields, time of response etc). In a first attempt, are also included few theoretical aspects regarding thermal modelling of microactuators (electrothermic circuit, diagram of thermal flux propagation, thermic impedance, critical current etc)

Studied layered carbon consists in a formic acid-graphite intercalation (formic acid-GIC) compound. Formic acid-GIC has been successfully synthesized by an electrochemical process whereby formic acid solution serves as both the electrolyte and the intercalate source. Next, the synthesized compounds have been exfoliated by rapid heating to a relatively low temperature ($\sim 400^{\circ}\text{C}$). The resulted flaky compound was pressed into circular plates being studied and modeled in this shape as thermal micro-actuator. During experiments, the carbon plates were connected to an electrical supply and

as a result an expansion effect of the plates that actuate the carbon plates has been obtained.

II. EXPERIMENTAL

II.1. MATERIALS

As raw material for layered carbon compound was used natural graphite flakes with average flake diameter of 0.4 mm and carbon content of 99.3% (Shanghai Chemical Corp. - China). HCOOH (98%) was purchased from Sigma Aldrich

Electrochemical procedures were performed by using an galvanostat (EG and G Model 273).

The stage structure of reacted graphite was characterized by powder X-ray diffraction with $\text{CuK}\alpha$. The microstructure and morphology of flaky layered carbon compounds were examined using a scanning electron microscope (FSEM-FIB AURIGA ZEISS). The specific surface area of the layered carbon was measured by nitrogen gas adsorption and Brunauer –Emmett-Teller (BET) calculation in a surface analyzer (AUTOSORB 1-TCD Quantachrome).

Actuation measurements have been performed by using a CORREX microdinamometer for microforce: $0 \div 5\text{N}$, a precision microcomparator for microactuation (microdisplacement); $0, 2 \div 50\mu\text{m}$ and a FLUKE multimeter.

II.2. SYNTHESIS OF FLAKY LAYERED CARBON MATERIAL

As raw material for flaky layered carbon materials was synthesized an acid formic-GIC. Thus, natural graphite flakes with average flake diameter of 0.4 mm with carbon content of 99.3% were pressed into a disk of 6 cm in diameter. The disk of graphite serving as the working electrode was placed into a platinum gauze. Two platinum plates were employed as the counter electrodes.

The $\text{Hg}/\text{Hg}_2\text{SO}_4$ 0.615V versus the standard hydrogen electrode (SHE) served as the reference electrode. 100ml of pure HCOOH (98%) was used as electrolyte in the electrochemical process by using a galvanostat (EG and G Model 273)

was applied an anodic current and recorded the reaction potential change with time. An anodic oxidation occurred under a constant current density with a range of $1\text{-}20\text{mA cm}^{-2}$ and the intercalation reaction lasted for 5-10 hours. After reaction, the graphite was dried in air at 70°C for one hour.

For exfoliation a graduated quartz glass beaker was heated in a furnace to desired temperature. The HCOOH-GIC decomposed and expanded immediately. After 10s the beaker was removed from the furnace.

II.3. CHARACTERIZATION OF SYNTHESIZED GICS - BASED CARBON MATERIALS

Formation of formic acid-graphite intercalated graphite by an electrochemical reaction is demonstrated by XRD spectra. The (00l) X-ray diffraction patterns (fig. 1) show that reacted graphite exhibits the stage structure 3 and 4 with the repeat spacing along the c-axis (I_c) of 14.41 \AA and 17.75 \AA respectively. The average thickness of the intercalate layer (d_l) is identified as 7.71 \AA .

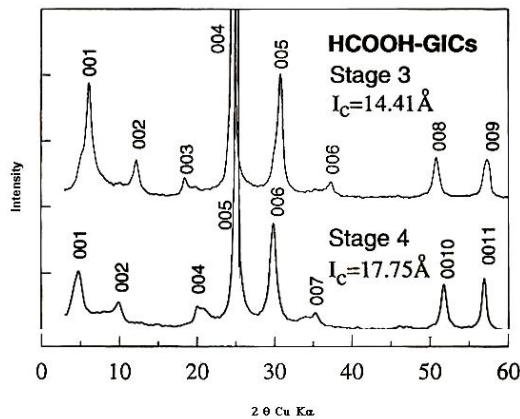


Fig. 1. X-ray diffraction patterns of HCOOH-GICs (non-exfoliated carbon) with stage structure 3 to 4.

The formation of HCOOH-GIC is also demonstrated by the exfoliation of reacted graphite by rapidly heating at 400°C . Exfoliation of HCOOH-GIC flakes produce flaky layered structures as is shown in fig. 2.

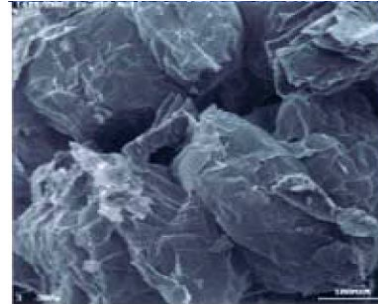


Fig. 2. SEM image of exfoliated HCOOH-GICs.

The BET surface area of expanded HCOOH-GIC was of $50\text{m}^2 / \text{g}$.

II.4. ELECTRICAL CHARACTERIZATION OF LAYERED FLAKY CARBON

After thermal expansion of the HCOOH-GIC described at II.3., resulted compound was pressed into circular plates being studied and modelled in this shape as thermal micro-actuator.

In electromechanics applications a necessary characteristics is the linear or the nelinear behaviour [9]. From the experimental results (as is shown in fig. 3) this material can be electrical characterised as having a nonlinear behaviour.

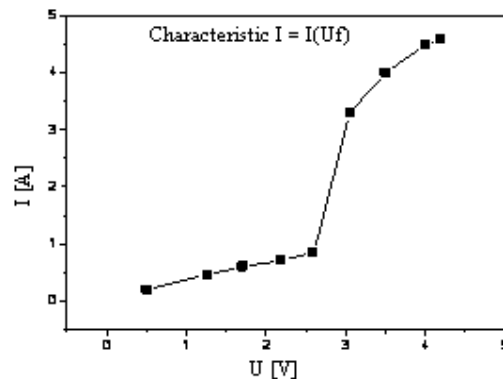


Fig. 3. Electric nonlinearity characteristic $I(U)$ for carbon material.

II.5. ELECTROTHERMIC MICROACTUATOR

II.5.1. THEORETICAL CONSIDERATIONS

Taking into consideration a thermo inhomogeneous system [6, 7, 8] with two elements (considering for the each active electrothermic carbon element as is described in fig. 4):

I.(c) – connecting cooper strap and

II.(elth) - electrothermic carbon element characterised by the following parameters:

$$I\{S_1, s_1, J_1, \rho_1, \lambda_1, \alpha_1, a_1, b_1\} \text{ and}$$

$$II\{S_2, s_2, J_2, \rho_2, \lambda_2, \alpha_2, a_2, b_2\} \text{ where:}$$

$S_{1(2)}$ the transverse section, $s_{1(2)}$ - the transverse perimeter, $\rho_{1(2)}$ - the electric resistivity, $\lambda_{1(2)}$ - the thermal conductivity, $\alpha_{1(2)}$ - the thermal coefficient of the resistivity, $a_{1(2)}$ and $b_{1(2)}$ - the electrothermic ratios, which can be expressed as:

$$a_1 = \sqrt{\frac{\alpha_1 s_1}{\lambda_1 S_1}}, a_2 = \sqrt{\frac{\alpha_2 s_2}{\lambda_2 S_2}} [m^{-1}] \quad (1)$$

$$b_1 = \sqrt{a_1 \theta_a + \frac{J_1^2 \rho_1}{\lambda_1}} [grad^{0.5} m^{-1}]$$

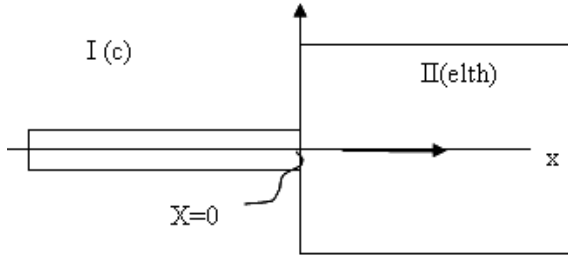


Fig. 4. Modelling of electrothermal microactuator system.

$$b_2 = \sqrt{a_2 \theta_a + \frac{J_2^2 \rho_2}{\lambda_2}} [grad^{0.5} m^{-1}] \quad (2)$$

with θ_a - room temperature.

To each system element the thermal stress is defined by the recursive equations [6, 7].

$$\frac{d^2 \theta_1}{dx^2} - a_1^2 \theta_1 + b_1^2 = 0, \quad \frac{d^2 \theta_2}{dx^2} - a_2^2 \theta_2 + b_2^2 = 0 \quad (3)$$

The solution for the equation 3 are the following:

$$\theta_1(x) = A_1 e^{a_1 x} + B_1 e^{-a_1 x} + \theta_{s1} \quad (4)$$

$$\theta_2(x) = A_2 e^{a_2 x} + B_2 e^{-a_2 x} + \theta_{s2} \quad (5)$$

and:

$$\theta_{s1} = \frac{b_1^2}{a_1^2} = \theta_{a1} + \frac{J_1^2 \rho_1 S_1}{\alpha_1 s_1} \quad (6)$$

$$\theta_{s2} = \frac{b_2^2}{a_2^2} = \theta_{a2} + \frac{J_2^2 \rho_2 S_2}{\alpha_2 s_2} \quad (7)$$

These solutions represent the stationary temperature state on the electrothermic electric current effect in the absence of the axial heat change.

For the determination of the A_1, B_1, A_2, B_2 constants we consider the frontier conditions:

$$x = -\infty, \theta_1(-\infty) = \theta_{s1}, \left(\frac{d\theta_1}{dx} \right)_{x=-\infty} = 0 \quad (8)$$

$$x = 0, \theta_1(0) = \theta_2(0) = \theta_d, \left(\frac{d\theta_1}{dx} \right)_{x=0} = \left(\frac{d\theta_2}{dx} \right)_{x=0} \quad (9)$$

$$x = +\infty, \theta_2(+\infty) = \theta_{s2}, \left(\frac{d\theta_2}{dx} \right)_{x=+\infty} = 0 \quad (10)$$

By the condition (8), $B_1 = 0$ and the solution (4) is:

$$\theta_1(x) = A_1 e^{a_1 x} + \theta_{s1} \quad (11)$$

From (9) in section between subdomains I and II (see Fig. 6):

$$\theta_d = A_1 + \theta_{s1} \quad (12)$$

And

$$A_1 = \theta_d - \theta_{s1} \quad (13)$$

Thus results:

$$\theta_1(x) = (\theta_d - \theta_{s1}) e^{a_1 x} + \theta_{s1} \quad (14)$$

Similar

$$\theta_2(x) = (\theta_d - \theta_{s2}) e^{a_2 x} + \theta_{s2} \quad (15)$$

From (9) to $x = 0$ is possible to determinate θ_d :

$$a_1(\theta_d - \theta_{s1}) = -a_2(\theta_d - \theta_{s2}) \quad (16)$$

$$\theta_d = \frac{a_1 \theta_{s1} + a_2 \theta_{s2}}{a_1 + a_2} \quad (17)$$

In table 1 is presented a numerical cover example (specific for the microstructure of electrothermic actuator presented in fig. 5).

Table 1. Dimensions of the electrothermic actuator.

$L[mm]$	$l_c[mm]$	$d_c[mm]$	$d[mm]$	$g_c[mm]$	$g_s[mm]$
49	13	5	12	0,5	1,16
100	25	5	10	0,5	1
320	4	4	4	0,5	1

III. MICROSTRUCTURES OF THE ELECTROTHERMIC MICROACTUATORS

In fig. 5 is presented an electrothermal experimental microactuator structure where the active carbon element (with grey color) is fixed on a glass support. In table no. 1 are listed three experimental variants, where g_c - represented the thickness of the carbon element and g_s - the thickness of glass support with square geometry

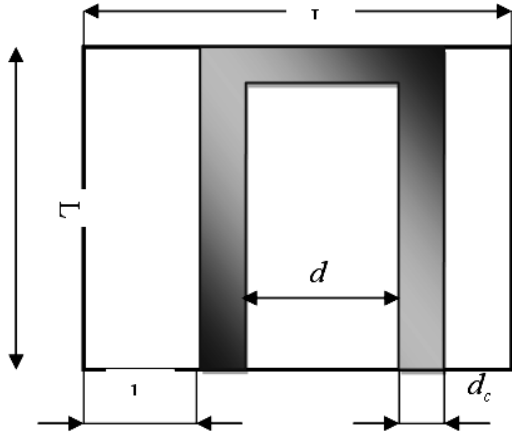


Fig. 5 .The microstructure of an electrothermic actuator

Table 2. Electrothermic material parameters.

Parameters	I(c) Subdomain microactuator cooper strap	II(elth) sub-domain micro-actuator carbon element
Transverse section $S_1 [m^2]$	$S_1 = 0,1 \cdot 10^{-3} \cdot 2 \cdot 10^{-3} = 2 \cdot 10^{-6}$	$S_2 = 0,5 \cdot 10^{-3} \cdot 2,5 \cdot 10^{-6}$
Transverse perimeter $s_1 [m]$	$s_1 = 4,2 \cdot 10^{-3}$	$s_2 = 11 \cdot 10^{-3}$
Resistivity $\rho [\Omega m]$	$\rho_1 = 0,00158 \cdot 10^{-6}$	$\rho_2 \cong 14 \cdot 10^{-6}$
Thermic conductivity $\lambda [W \cdot m^{-1} \cdot grd^{-1}]$	$\lambda_1 = 390$	$\lambda_2 \cong 120$
Thermic coefficient of the resistivity $\alpha [grd^{-1}]$	$\alpha_1 = 0,0043$	$\alpha_2 = 0,0013$

Electric current density $J [A/m^2]$	$J_1 = 225 \cdot 10^{-3}$ ($toI = 45 \cdot 10^{-3} [A]$)	$J_2 = 18 \cdot 10^{-3}$
Electrothermic ratio $a [m^{-1}]$	$a_1 = 0,481175$	$a_2 = 0,2183$
Electrothermic ratio $b [grd^{0,5} m^{-1}]$	$b_1 = 390$	$b_2 = 2,089$
Environment temperature $\theta_a [^{\circ}C]$	$\theta_a = 20$	$\theta_a = 20$
Particular solutions to extreme heat stress $\theta_s [^{\circ}C]$	$\theta_{s1} = 41,5$	$\theta_{s2} = 91,57$
Temperature to the $x = 0$ $\theta_d [^{\circ}C]$	$\theta_d = 57,1$	$\theta_d = 57,1$

Other type of carbon actuator microstructure is presented in fig. 6 where active carbon element (2) of the electrothermal microactuator is mounted on a glass support (1), the connection between the carbon element is realized with a copper strap (3) (width $2mm$, thickness $0,1mm$) and one external connection (4). The carbon elements have a square geometry with $5mm$ side and glass support has also a square geometry with $20mm$. The carbon element has a thickness of $0,5mm$ and width of $5mm$. The thickness of glass support is of $1mm$. In fig. 6 is presented an microactuator with a spiral geometry of the carbon element (breadth $2mm$, thickness $0,5mm$ distance between the sides of spiral $1mm$).

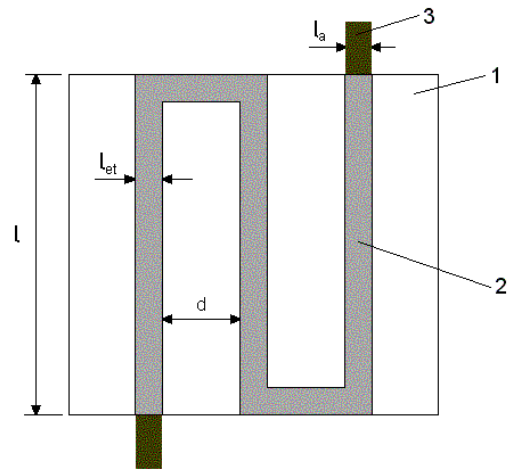


Fig. 6. An electrothermic actuator spiral structure.

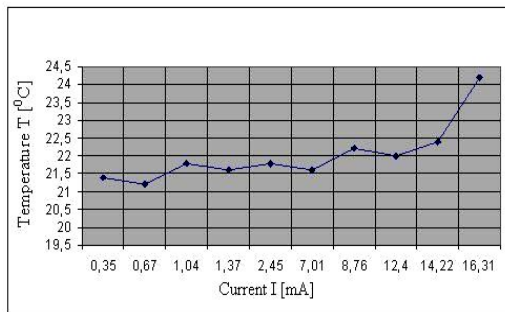
The carbon spiral element is mounted on a glass support with 10mm side.

IV. EXPERIMENTAL SET-UP FOR THERMAL ACTUATION EVALUATION

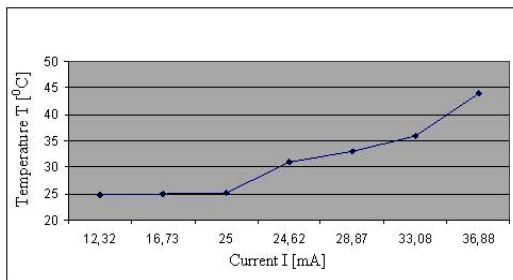
Two heating characteristics (shown in fig. 7) for the electric current of the microactuator described in fig. 6, has two domains: a) $0 \div 17mA$ and b) $12 \div 37mA$; these represents the functional field of electrothermal actuation. In characteristics b) between $25mA$ and $37mA$, became obvious a distinct field with a gradient $\sim 2,083 [^{\circ}C / mA]$.

Specific experimental electromechanic features such as displacement function to electrical current is presented in fig. 8 with a displacement gradient of $1,666 [\mu m / mA]$ in subdomain $25 \div 37mA$, which can be considered the optimum functional subdomain.

An experimental characteristic of the responding time indicates 125sec (see fig. 9). The measurements were realized to a constant force of $2,5N$.



a)



b)

Fig. 7. The experimental dependence temperature vs. applied electric current.

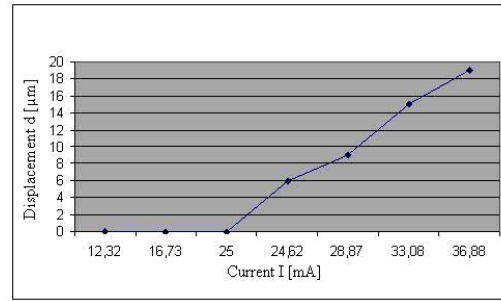


Fig. 8. Displacement characteristic of the microactuator.

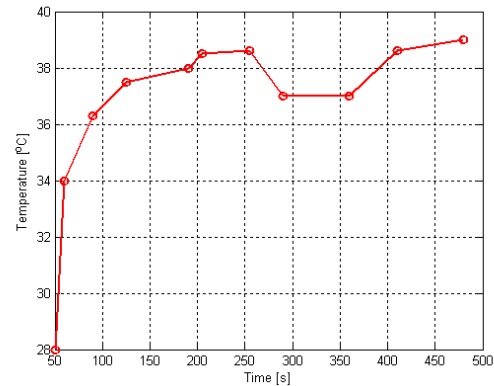


Fig. 9. The experimental feature of the responding time.

V. CONCLUSION

Electrothermal actuators are a category of thermal actuators rarely approached in present. In this paper are described some electrothermal microactuators based on a flaky layered carbon material. This type of micro actuator has a very low temperature field $25 \div 40^{\circ}C$ (which can not influence the microactuation application problems) and very little field of electric current (the supplying of microactuator): max. $38mA$. Another important parameter for this type of applications is the gradient or the sensibility. The identified value for carbon material in present case is $1,66 [\mu m / mA]$ which represents a distinct value.

In conclusion, layered carbon compounds could represent a potential important starting material for designing of new electrothermal actuators.

VI. ACKNOWLEDGMENT

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• VI. BIOGRAPHIES

Mircea Ignat was born in Bucharest on March 4, 1953. He graduated at 1977 and he received Ph.D. degrees in electrical engineering from Bucharest Polytechnic University in 1999. His employment experience included the National Research Electrical Engineering Institute, Dep. of Electrical Micromachines Research and he is the head of Electromechanics Department. The research preoccupation include: the synchronous generators and the high speed electric machines. He is member of IEEE.

Hristea Gabriela: Phd (2002) in Chemical engineering, *Politehnica* University Bucharest with : *Graphite Intercalation Compounds. Synthesis and Characterization*; Over 15 years of solid experience conducting research of chemical compounds: synthesis, characterization, analysis and tests of different inorganic compounds (especially carbon based materials) to identify properties for development and improvement of new and existing products/materials; in-depth knowledge of synthesis (*powder metallurgy, sol-gel, wet chemistry*) and testing/characterization of carbon materials on small and medium scale; Knowledge of a range of analyzing techniques such as: *Pore and Surface area* characterization-nitrogen adsorption, and *Small angle neutron scattering*.

Polyethylene electrical insulation materials under accelerated degradation

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Abstract- The structure on dielectric materials is one of the main important parameter, which determines the durability of products. The degradation process runs differently in various types of polyethylene, because the macromolecules present different level of branching, different crystallinity grades, different chemical resistance. The dissimilarities between the resistance on ageing of different sorts of polyethylene are depicted by FTIR analysis, chemiluminescence determinations and the measurement of electrical resistivity. The consequences on the long term service under γ -irradiation degradation are discussed.

Index Terms--: Polyethylene materials, accelerated degradation, FTIR, chemiluminescence, electrical properties.

I. INTRODUCTION

Polyethylene is an essential plastic whose manufacture places it on the first position through engineering plastic materials. The several areas of applications (cables and wires, automotive, medical wear, packaging, pipes, miscellaneous) consume enormous amounts of polyethylene due to its excellent features like flexibility, toughness, barrier for diffusion, easy of fabrication. The polyethylene type determines the peculiar use. The dissimilarities between their structural characteristics (branching, crystallinity, physical properties, chemical resistance) are the criteria by which the praxis of polyethylene is based.

A large amount of work can be found in literature devoted to the depiction of material behavior under certain working conditions [1-6]. Polyethylene blends have received special attention due to the alternative possibility of suitable formulation for certain applications [7-11].

The accelerated degradation promoted by ionizing radiation is an appropriate procedure for characterization of the effects induced by the long term utilization under oxidative environments. The qualification of materials performances

directs the customers on the correct way of implementation.

The concomitant reactions occurred during endurance testing run to the modifications in the chemical state of material, which determines the warranty period for hazardous applications like nuclear power stations, aircrafts, automotives, electrical cables, gaskets, and many other key items.

The present investigation presents a comparative study on the polyethylene answer to the degradative action of γ -radiation as the carrier and donor of energy.

II. EXPERIMENTAL

Three kinds of polyethylene (two types of HDPE (named HDPE 1 and HDPE 2) and one sort of LDPE) were selected for the characterization of resistance on the hard conditions of usage. These materials were investigated as received products supplied by ARPECHIM Pitești (Romania). Table 1 presents the main features of raw materials.

TABLE 1. Material input data

Property	HDPE 1	HDPE 2	LDPE
Density @ 23 ⁰ C (g.cm ⁻³)	0.964	0.963	0.925
Melting flow rate (190 ⁰ C/2.16 kg) (g/10 min)	5.5	0.33	1.63
Crystallinity (%)	68.60	65.07	48.75
Nr. CH ₃ /100 C	1.17	0.91	3.55

Each polyethylene sheet (thickness: 0.3 mm) was obtained in an electrical heated press at 150 atm for 10 min. The polyethylene films were also processed by pressing under similar conditions.

The exposure in high energy radiation field was done using an irradiator GAMMATOR M-38-2 (USA) provided with ¹³⁷Cs source in air at room temperature. Dose rate was 0.4 kGy/h. This

low dose rate simulates the accidental conditions that are really met when electrical and thermal overcharges are attended.

For the characterization of chemical modifications two sensitive procedures were applied: chemiluminescence (equipment: LUMIPOL 3 – SAS, Slovakia). The procedure and data interpretation for chemiluminescence results have been previously reported [12]. FTIR and UV-Viz spectrometric records were carried out on JASCO 4200 with 20 scans and 4 cm⁻¹ resolution and JASCO V 570 Japan, respectively. Electrical measurements were assured by Keithley 7600A (USA) multimeter coupled with a resistivity test chamber 8009 (Agilent, USA).

Carbonyl and hydroxyl indexes were calculated as the ratios of the absorptions at 1720 cm⁻¹ and 3350 cm⁻¹, respectively, and the absorption at 1475 cm⁻¹ (reference) [13]. The values for number of CH₃/100 carbon atoms were calculated according with ASTM 2238-68.

The samples were subjected to investigations immediately after the end of irradiations.

III. RESULTS AND DISCUSSION

The polymers exposed to the action of ionizing radiation are profoundly modified as the consequence of energy transfer onto macromolecules. The random scissions of weaker bonds of macromolecules creating free radicals are the primary chemical events [10,14]. The subsequent reactions in which free radicals are involved become the competitive processes. During γ -exposure to applied low dose rate oxidative degradation is the main process that depletes free radicals. The mechanism of radiation induced oxidation of polymers may be found elsewhere [15,16].

The dissimilar susceptibility of tested polyethylenes to oxidation is described by the evolution in UV-Viz spectra (Fig. 1).

The maxima placed at 220 and 270 nm in the UV spectra of LDPE are ascribed to the presence of ketonic carbonyl groups and conjugated double bonds in polyene structures, respectively [17]. These peaks are shifted towards higher wavelengths in tested HDPEs. This difference arises from the discrepancy in the polymer matrixes. The augmentation in the absorption of carbonyl components is faster than the enhance in the absorption depicting the accumulation of double bonds. The former oxygenated function is the result of the reactions

of free radicals with the molecular oxygen diffused from the outer layers of polypropylene and the later peak absorption for unsaturation increases due to the disproportionation reactions. The advance in the – C = C – absorption is smoother than the accumulation of carbonyl moieties because double bonds are consumed by addition or oxidation.

The comparison of UV recorded spectra for the three polyethylenes reveals the advance in the degradation process faster in LDPE than it occurs in HDPE. The explanation is the branching level of each material, which is directly by the higher number of tertiary carbon atoms in low density polyethylene. The early start of oxidation in LDPE is related either by the direct attack of molecular oxygen on the tertiary carbon positions, or by the formation of peroxy radicals, the promoters of chain process of oxidation, as the intermediates resulting from the reactions R[•] + O₂. The present results are in a good agreement with other previous information [1, 18-20]. The exposure to high energy radiation accelerates the formation of free radicals by the formation of *trans*-vinylene structures [21].

The influence of branching on the evolution in the amounts of oxidation products can be explained by the different values in Nr. CH₃/100 C. The highest figure for this property (3.55) for LDPE illustrates the material tendency to oxidation, because the probability of the scission or oxygen attack on tertiary carbon positions is higher in comparison with HDPEs. The radiation stability order of tested polyethylenes:



is the opposite sequence of the increase in the number of CH₃ per 100 carbon atoms. The relative increases in the absorbance at 220 nm, (A_{D=100}/A_{D=0}) are 0.62, 0.75 and 3.4 for HDPE 1, HDPE 2 and LDPE, respectively. It defines the corresponding augmentation in the oxidation availability on polyethylene macromolecules.

The susceptibility of polyethylene to the generation of unsaturation (absorption at 270 nm) follows the same order as the sequence of oxidation instability. The lower – branched polyethylene withstands better on the low irradiation dose range, which may be assimilated with the condition of accident. Because 1 kGy is equivalent to 10³ J/kg, the radiation – induced modifications occurred for each 1 kGy involves high amount of energy transferred onto material

on a short time interval. This energy is enough for the induction of degradation during the encroaching technological limits.

FTIR spectra (Fig. 2) describe more detailed the structural modifications occurred in irradiated polyethylenes. The absorption of several peaks is modified displaying the contribution of various reactions of free radicals during the competition between the formation and the decay of these reactive intermediates.

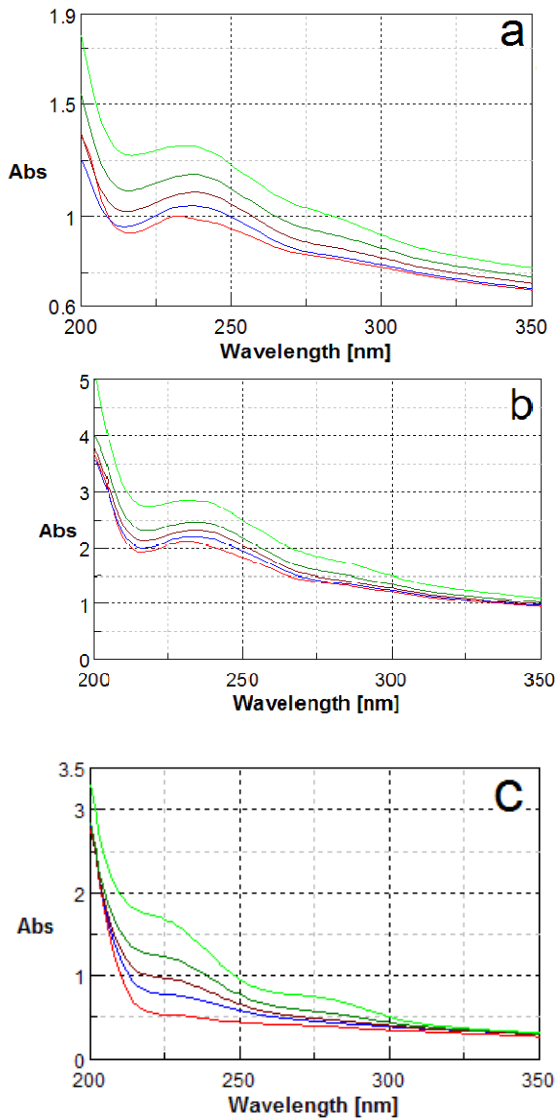


Fig. 1. UV-Viz spectra of different kinds of polyethylene: (a) HDPE 1; (b) HDPE 2; (c) LDPE.

(-) 0 kGy; (-) 20 kGy; (-) 40 kGy; (-) 70 kGy; (-) 100 kGy.

There are three important spectral regions: below 1000 cm^{-1} , where several unsaturated structures like *trans*-vinylene (965 cm^{-1}), vinyl

(909 cm^{-1}) and vinylidene (888 cm^{-1}) appear [22]; the peak around 1720 cm^{-1} (1716 cm^{-1} for acids, 1722 cm^{-1} for ketones, 1735 cm^{-1} for aldehydes [23] and 1746 cm^{-1} for esters [24]) and the last range around 3350 cm^{-1} , where the hydroperoxides can be determined. All these functions appear as the result of degradation mechanism through which free radicals react with oxygen forming peroxy intermediates further subjected to intramolecular rearrangements or with macromolecules abstracting proton. These reactions occur predominantly in the amorphous zones, where the movement of reacting entities is much less restricted than it is inside the ordered phase (crystalline component).

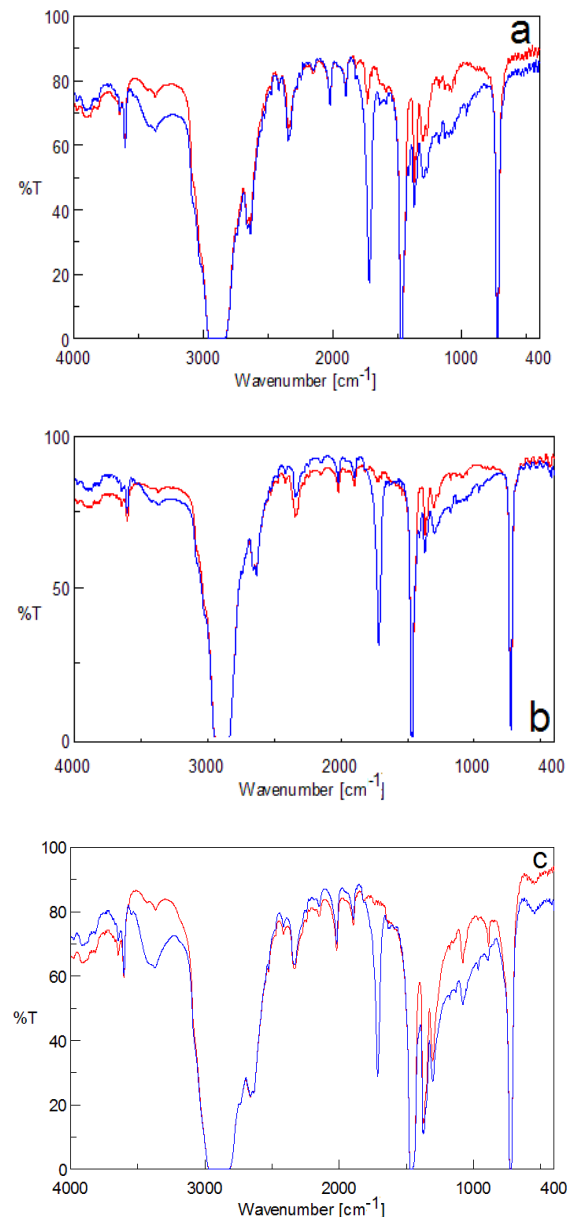


Fig. 2. FTIR spectra of different kinds of polyethylene: (a) HDPE 1; (b) HDPE 2; (c) LDPE. (-) 0 kGy; (-) 100 kGy.

There are three important spectral regions: below 1000 cm^{-1} , where several unsaturated structures like *trans*-vinylene (965 cm^{-1}), vinyl (909 cm^{-1}) and vinylidene (888 cm^{-1}) appear [22]; the peak around 1720 cm^{-1} (1716 cm^{-1} for acids, 1722 cm^{-1} for ketones, 1735 cm^{-1} for aldehydes [23] and 1746 cm^{-1} for esters [24]) and the last range around 3350 cm^{-1} , where the hydroperoxides can be determined. All these functions appear as the result of degradation mechanism through which free radicals react with oxygen forming peroxy intermediates further subjected to intramolecular rearrangements or with macromolecules abstracting proton. These reactions occur predominantly in the amorphous zones, where the movement of reacting entities is much less restricted than it is inside the ordered phase (crystalline component).

The progress in the oxidative degradation of different sorts of polyethylene is well depicted as a self catalyzed process. The most important kinetic characteristics are oxidation induction time and rate of oxidation. The chemiluminescence investigation for the qualification of the resistance to oxidation has revealed the

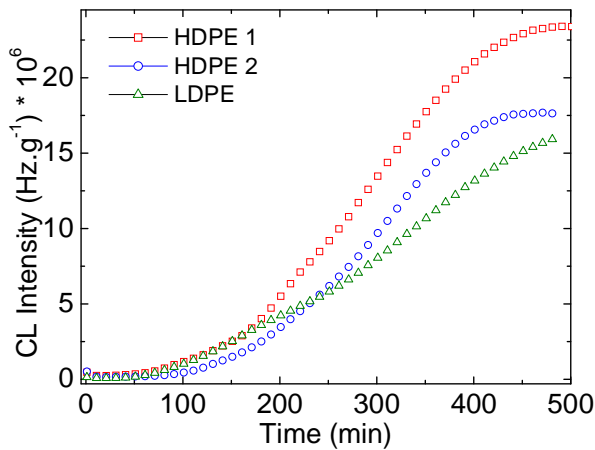


Fig. 3. Dependences of CL intensity on time for studied polyethylenes evaluated at 170°C .

The oxidation induction time is 52 min. for LDPE, while the same parameter attends 154 min. and 188 min. for HDPE 1 and HDPE 2, respectively. Due to the high percentage of crystalline phase, the propagation of oxidation takes place somewhat slower than it was expected for a branched molecule materials.

The advanced degradation obtained in γ -radiation field brought about significant cropping

of oxidation induction time (Fig. 4) anticipated by the higher amount of free radicals formed during irradiation. The medallion inserted in Fig. 4 depicts the tendency for oxidation in the case of low density polyethylene. The same order of radiation resistance is pointed out:

$$\text{HDPE 1} > \text{HDPE 2} > \text{LDPE}$$

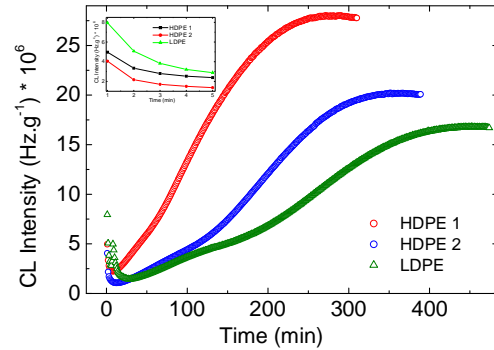


Fig. 4. The CL curves for polyethylenes irradiated at 100 kGy (measurement temperature: 170°C).

Fig. 5 illustrates the results of the competition between degradation and crosslinking. If at the first applied dose (25 kGy) a sharp alteration in the oxidation resistance can be noticed. For higher doses ($50, 75$ and 100 kGy) a slight amelioration in the oxidation rate may be obtained, but the thermal stability of all irradiated samples is far from the behavior of pristine material. The higher concentration of free radicals generated by radiolysis in the higher dose irradiated polyethylenes allows the recombination of radicals in a larger extent than in the same material subjected to low irradiation dose.

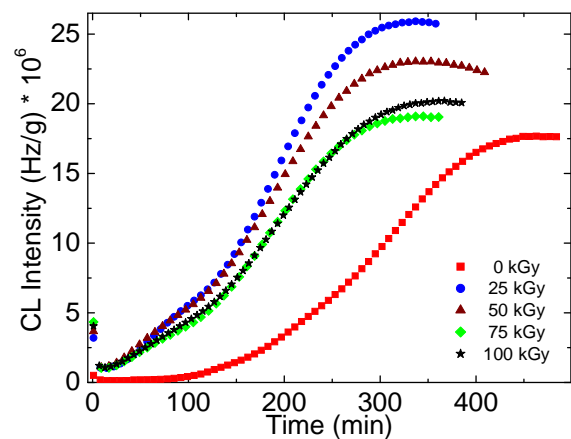


Fig. 5. CL curves recorded for HDPE 1 sample irradiated at various doses.

This comportment was observed for all three types of polyethylene. However, there is difference between the intake levels of recombination.

The effect of oxidative degradation can be described by the evaluation in electrical properties, because the oxygenated products act as electrical dipoles. If the high energy radiation passes the testing polymer samples, δ electrons appear. These charge carriers may be trapped onto electronegative sites like oxygen atoms, double bonds, or even some molecular defects acting as gaps in entanglement configurations [25].

The excellent electrical insulation properties of polyethylene are the result of the

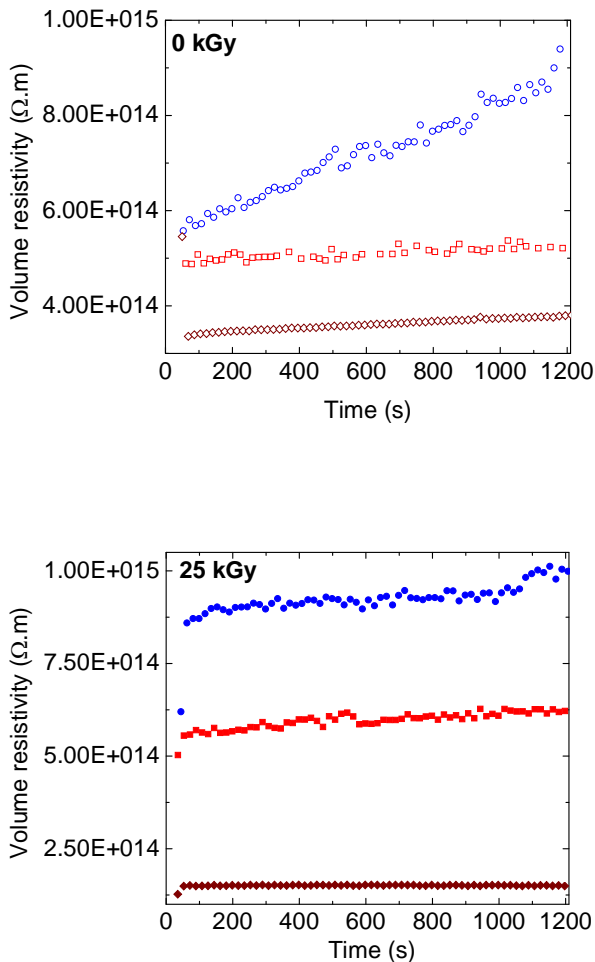


Fig. 5. Evolution of volume resistivity on time (applied tension 50 V)

(□, ■) HDPE 1; (○, ●) HDPE 2; (◇, ◆) LDPE

constitution of polyethylene molecule exclusively by non-polar atoms (carbon and hydrogen). The residual conduction measured on pristine samples

is the consequence of random scissions which take naturally place by the attack of moderate stressors during handling.

In Fig. 5a (dose 0 kGy) it may be noticed some definable remarks:

- the different values for volume resistivity are obtained characterizing the peculiarity of each type of polyethylene; the most conductive type is low density polyethylene, which is the most susceptible to scission and oxidation; the order based on the increase in the conductivity:

$$\text{HDPE 2} < \text{HDPE 1} < \text{LDPE}$$

is the same sequence that defines the increase in the values of CH_3 number per 100 carbon atoms, i. e. the order of the increasing in branching level.

- HDPE 2 presents an ascendant manner in the resistivity values. It may be considered that the electron traps are successively released in the succession of increasing their depths.

In Fig. 5b (dose 25 kGy) the contribution of dipole structures simultaneously with the trapped electrons alters the insulation performances of polyethylenes. The largest difference in resistivity values is displayed by low density polyethylene, due to its noticeable availability for oxidation.

Different dipoles containing oxygen presenting their specific dipole moments are accumulated during γ -exposure and oxygen diffusion [26], whose presence leads to the depreciation of insulating features of PEs.

IV. CONCLUSION

The comparative study on the modification in chemical and electrical resistances of three sorts of polyethylene emphasizes the higher susceptibility of low density polyethylene to oxidation due to the lower content of crystalline phase and higher value of CH_3 number per 100 carbon atoms. The history of material plays an important role in the determination of material durability, so that it is very important to establish the contribution of molecular structure on the service behavior.

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• VI. BIOGRAPHIES

Traian Zaharescu was graduated on 1965 at the Faculty of Chemistry, University of Bucharest. The PhD thesis was presented on the area of thermodynamics of His career followed activities in nuclear fuel elements, chemistry of materials for electrical engineering, radiation chemistry for durability characterization, educational performances teaching on modification of polymers under high energy irradiation. His expertise covers complementary areas like modification induced by ionizing radiation, chemistry of electrical engineering, aeronautic and nuclear materials. The published several papers and books on the materials science branch are related to the degradation and stabilization of polymers, physical, chemical and electrical which depict on structural changes induced by polymerization, ageing and recycling.

Preparation of a Formatted Technical Paper for the Bulletin of Micro and Nanoelectrotechnologies

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Abstract - This document is itself an example of the desired layout (inclusive of this abstract) and can be used as a template. The document contains information regarding desktop publishing format, type sizes, and typefaces. Style rules are provided that explain how to handle equations, units, figures, tables, abbreviations, and acronyms. Sections are also devoted to the preparation of acknowledgments, references, and authors' biographies. The abstract is limited to 150 words and cannot contain equations, figures, tables, or references. It should concisely state what was done, how it was done, principal results, and their significance.

Index Terms - The author shall provide up to 10 keywords (in alphabetical order) to help identify the major topics of the paper and to be enough referenced.

I. INTRODUCTION

This document provides an example of the desired layout for a published MNE technical paper and can be used as a Microsoft Word template. It contains information regarding desktop publishing format, type sizes, and typefaces. Style rules are provided that explain how to handle equations, units, figures, tables, abbreviations, and acronyms. Sections are also devoted to the preparation of acknowledgments, references, and authors' biographies.

II. TECHNICALWORK PREPARATION

Please use automatic language check for your spelling. Additionally, be sure your sentences are complete and that there is continuity within your paragraphs. Check the numbering of your graphics (figures and tables) and make sure that all appropriate references are included.

A. Template

This document may be used as a template for preparing your technical paper. When you open the file, select "Page Layout" from the "View" menu (View | Page Layout), which allows you to see the footnotes. You may then type over sections of the document, cut and paste into it

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C. Typefaces and Sizes

Please use a Times New Roman font. (See your software's "Help" section if you do not know how to embed fonts.) Table I is a sample of the appropriate type sizes and styles to use.

TABLE I. Table name will be written in Times New Roman font.

Micromotor Code	b [mm]	a [mm]	h [mm]	Material
MPR33	33	25	20	PZT 5
MPR27	27	18	9	PZT 5
MPR15	16	10	10	PZT 5

D. Section Headings

A primary section heading is enumerated by a Roman numeral followed by a period and is centered above the text.

A primary heading should be in capital letters and bolded. The standard text format is considered times new roman 12.

The paper title should be in times new roman 20 uppercase and lowercase letters, not all uppercase.

Author name is set to times new roman 12, institution and contact address (E-mail) are set to times new roman 10.

Financial support should be acknowledged below the author name and institution. Example: This work was supported in part by the U.K. Department of Defence under Grant TX123.

A secondary section heading is enumerated by a capital letter followed by a period and is flush left above the section. The first letter of each important starting word is capitalized and the heading is bolded and italicized.

Tertiary and quaternary sections are accepted only in special cases, so usually must be avoided in order to keep a clear article structure. If required, a tertiary and quaternary section heading must be italicized and enumerated by adding an arabic numeral after each letter.

E. Figures and Tables

Figure axis labels are often a source of confusion. Try to use words rather than symbols. As an example, write the quantity "Torque," or "Torque, M ," not just " M ." Put units in parentheses. Do not label axes only with units. As in Fig. 1, write "Torque (cNm)" not just "(cNm)". Do not label axes with a ratio of quantities and units. For example, write "Current (A)," not "Current/A." Figure labels should be legible, approximately 10-point type.

Large figures and tables may span both columns, but may not extend into the page margins. Figure captions should be below the figures; table captions should be above the tables. Do not put captions in "text boxes" linked to the figures. Do not put borders around your figures.

All figures and tables must be in place in the text centered and written with times new roman 10. Use the abbreviation "Fig. 1" in sentence and for each figure name. Each table must be defined as „TABLE I”, with capital letters and roman numbers.

Digitize your tables and figures. To insert images in Word, use Insert | Picture | From File.

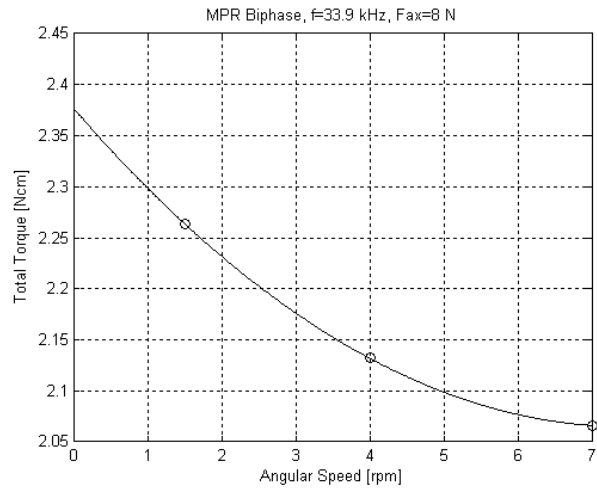


Fig. 1. Total torque function of angular speed. (Note that "Fig." is abbreviated and there is a space after the figure number.)

F. Numbering

Number reference citations consecutively in square brackets [1]. The sentence punctuation follows the brackets [2]. Multiple references [2], [3] are each numbered with separate brackets [1]-[3]. Refer simply to the reference number, as in [3]. Do not use "Ref. [3]" or "reference [3]" except at the beginning of a sentence: "Reference [3] shows....".

Number footnotes separately with superscripts (Insert | Footnote). Place the actual footnote at the bottom of the column in which it is cited. Do not put footnotes in the reference list. Use letters for table footnotes.

Check that all figures and tables are numbered correctly. Use Arabic numerals for figures and Roman numerals for tables.

Appendix figures and tables should be numbered consecutively with the figures and tables appearing in the rest of the paper. They should not have their own numbering system.

G. Units

Metric units are preferred in light of their global readership and the inherent convenience of these units in many fields. In particular, the use of the International System of Units ("Système International d'Unités" or SI Units) is advocated. This system includes a subsystem of units based on the meter, kilogram, second, and ampere (MKSA). British units may be used as secondary units (in parentheses). An exception is when British units are used as identifiers in trade, such as 3.5-inch disk drive.

H. Abbreviations and Acronyms

Define less common abbreviations and acronyms the first time they are used in the text, even after they have been defined in the abstract. Standard abbreviations such as SI, CGS, AC, DC, and *rms* do not have to be defined. Do not use abbreviations in the title unless they are unavoidable.

I. Math and Equations

Use either the Microsoft Equation Editor or the *MathType* commercial add-on for MS Word for all math objects in your paper (Insert | Object | Create New | Microsoft Equation *or* MathType Equation). "Float over text" should *not* be selected.

To make your equations more compact, you may use the solidus (/), the exp function, or appropriate exponents. Italicize symbols for quantities and variables. Use a long dash for a minus sign or after the definition of constants and variables. Use parentheses to avoid ambiguities in denominators.

The number of each equation must be consecutively added in parentheses and arranged at the right margin, as in (1). Be sure that the symbols in your equation have been defined before the equation appears or immediately following.

Don't use "Eq. (1)" abbreviation instead of "equation (1)," in a sentence.

$$L_m = \frac{m}{A^2} \tag{1}$$

With *m* – mechanical mass, *A* – force factor, *L_m* – Electromechanical inductance.

III. ACKNOWLEDGMENT

The following is an example of an acknowledgment.

The authors gratefully acknowledge the contributions of Mircea Ignat and Puflea Ioan for their work on the original version of this document.

IV. APPENDIX

Appendixes, if needed, appear before the acknowledgment.

V. References

References are important to the reader; therefore, each citation must be complete and correct. There is no editorial check on references, only the format times new roman 10 must be considered.

[1] Satanobu J., Lee D.K, Nakamura K., Ueha S., "Improvement of the Longitudinal Vibration System for the Hybrid Transducer Ultrasonic Motor", IEEE Trans. On Ultrasonic ferroelectrics and Frequency Control, vol. 47, no. 1, January 2000, pp. 216-220.

[2] Morita T., Yoshida R., Okamoto Y., Kurosawa M., "A Smooth Impact Rotation Motor Using a Multi-Layered Torsional Piezoelectric Actuator", IEEE Trans. On Ultrasonic ferroelectrics and Frequency Control, vol. 46, no. 6, November 1999, pp. 1439-1446.

• VI. BIOGRAPHIES

A technical biography for each author must be included. It should begin with the author's name (as it appears in the byline). Please do try to finish the two last columns on the last page at the same height. The following is an example of the text of a technical biography:

Mircea Ignat was born in Bucharest on March 4, 1953. He graduated at 1977 and he received Ph.D. degrees in electrical engineering from Bucharest Polytechnic University in 1999.

His employment experience included the National Research Electrical Engineering Institute, Dep. of Electrical Micromachines Research and he is the head of Electromechanics Department.

The research preoccupation include: the syncrone generators and the high speed electric machines. Is member of IEEE.

"We master an area of research if we meet the following three conditions:

- We know the primitive values, the main derived values and the domain laws*
- We know how to demonstrate from the laws the important theorems of the domain*
- We can deduce, using laws and theorems, the future, present or past evolution of any phenomenon from the field, depending of the real data conditions, that are related to the initial state of the physical system. These phenomena are generated in that state of that physical system, and its border.*

Initial conditions and borders conditions are sufficient for the determination of uniqueness phenomena "

Acad. Remus Radulet "Fundamentals electrodynamics" 1954