THE ANNALS OF "DUNAREA DE JOS" UNIVERSITY OF GALATI
FASCICLE IX. METALLURGY AND MATERIALS SCIENCE
N°. 1 - 2015, ISSN 1453 – 083X

# ELECTRICAL PROPERTIES OF ULTRA-SONICATED EPOXY RESIN

**Iulia GRAUR[a,b], Vasile BRIA[a], Adrian CÎRCIUMARU[a,b*], Iulian-Gabriel BÎRSAN[a]**
[a]"Dunărea de Jos" University of Galati, Border Faculty of Humanities, Economics and Engineering Sciences
[b] Diagnose and Measurement Group, Galați
e-mail: adrian.circiumaru@ugal.ro

## ABSTRACT

*As ultra-sonication is a mostly used method to disperse the particles into the polymer matrix, the aim of this study is to point out the effect of epoxy's exposure to ultra-sounds over the electrical properties, especially the electrical conductivity. As the epoxy resins are insulators, the method is based on the determination of electrical conductivity via the measurement of electrical insulation resistance. The polymer solution was exposed for 45 minutes to ultra-sounds produced by one, two, three and four generators at different values of ultra-sounds frequency.*

KEYWORDS: *electrical properties, ultra-sonicated, epoxy resin*

## 1. Introduction

The rapid spread of polymeric materials to new markets requires not only innovation in technological processes but equally the up-grade of the existing polymer processing technologies that allow them to expand into new areas of social life [1]. In recent years, considerable efforts have been made to improve the quality and reliability of thermosetting polymer materials [2, 3]. Thermosetting materials obtaining methods have the following advantages: easy processing in the initial stage because the resin systems are in the liquid phase, fibers or particles thermoset moistening is very easy, so that gaps, porosity and dry areas in composites are avoided, and affordable price of the required processing systems [4]. Epoxy resins have high mechanical strength, resistance to solvents, to heat, to moisture and show a very high internal cohesive force [5-7]. Strengthened, they have a good dimensional stability and outstanding electrical characteristics [8, 9]. The determination of electrical resistivity is recommended as a control measure for temperature processing and to establish the compliance with the specifications of particular strength where needed. Improved mechanical properties of conducting polymers increase their potential commercial applications. One of the immediate applications of conducting polymers is electrostatic and electromagnetic protection [10]. Although the search for new polymers with improved properties continues to attract great research interest, the economic drivers in the polymer industry demand the continual improvement of existing materials. This has led to a large effort aimed at modifying the existing polymers [11]. The number of papers describing electrical and other properties of nano-composites is increasing rapidly. The main results are summarized in recently published reviews [12-14]. The propagation of ultra-sonic waves in polymers depends on their viscoelastic behavior and density, resulting in polymers significantly affected by phase transitions occurring when changing temperature and pressure or during chemical reactions [15, 16]. Ultra-sound treatment is a way to improve the polymer material qualities, in particular during components matching phase of a composite and the formation of its structure, i.e. while the mixture is passing to its gel phase [17]. Under the action of ultra-sound, there is a change in homogenization, viscosity, relaxation time, and strengthening [18]. During formation, thermo-setting polymer resins undergo changes in their physical characteristics from a viscous liquid to a gel and finally to a hardened solid [19]. Therefore, in the present paper we intend to analyse the electrical conductivity of polymer materials exposed to ultra-sounds during the gelling phase.

## 2. Experimental

Four air-generators were used as ultra-sounds sources. The use of this type of generators had required a difficult work to identify their possible frequencies by maneuvering parts 4, 5, 7 and 8 in Fig. 1, together with fine variations of air pressure. In the end, four frequency values were found common for all the generators, namely 24 kHz, 26 kHz, 30k Hz and 42 kHz.

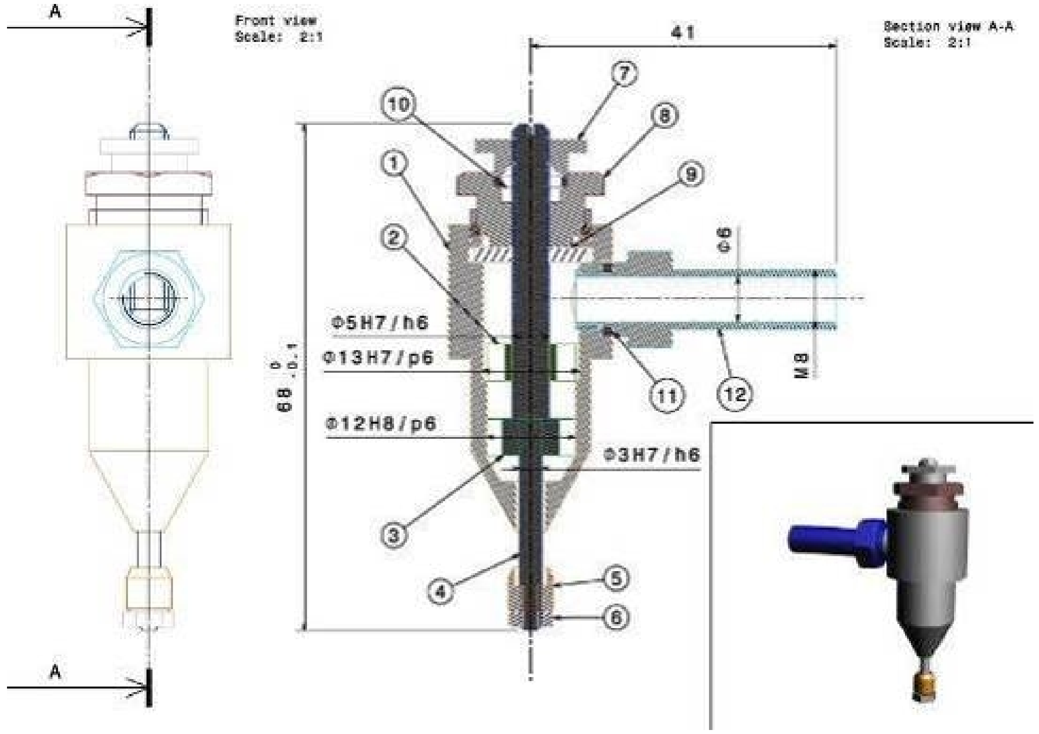


***Fig. 1.** Ultra-sound source: 1 - nozzle; 2, 3 - cross sleeves; 4 - rod; 5 - resonator; 6 - screw nut; 7 - locknut; 8 - cover; 9, 11 - washers; 10 - gasket; 12 - nozzle*

It was possible to set the configuration (Fig. 2) which allows for samples exposure to the ultra-sounds generated by one, two, three or four air generators. In fact, using the same frequency for all the generators, the energy of the exposure is a multiple of the energy of one generator. The pre-polymer solution was poured into cylindrical molds and exposed for 45 minutes to ultra-sounds.

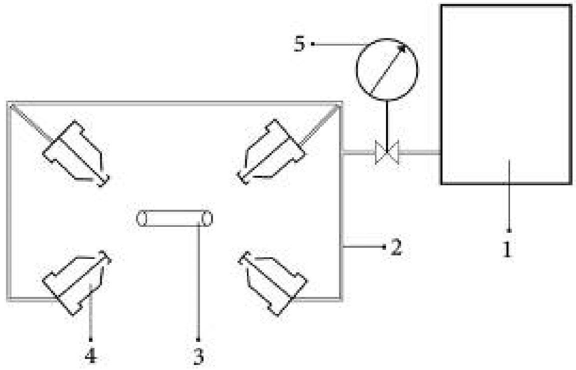


***Fig. 2.** Configuration of ultra-sound sources: 1 - air compressor; 2 - supply hose; 3 - test sample; 4 - ultra-sound source; 5 - pressure gauge*

Noise measurements were performed according to Standard noise emission SR EN ISO 3744/2009. The molds were placed at 1 m distance from each ultra-sound air generator (Fig. 3) [20]. Generators calibration was performed using an UltraMic 200k model microphone with Sea Wave software (Fig. 4).

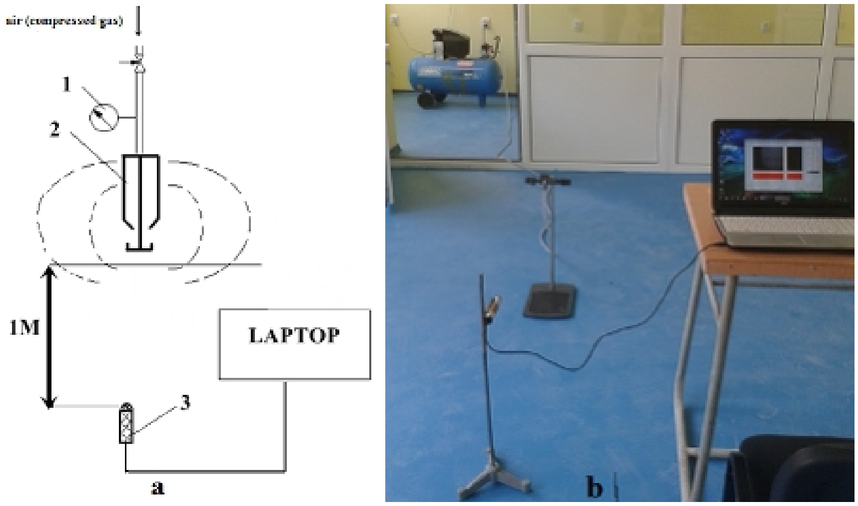
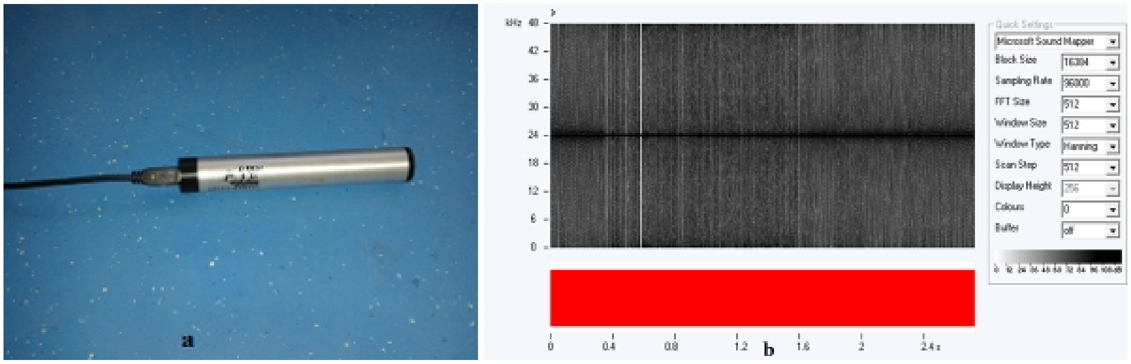


***Fig. 3.*** *Acoustic measurements: a - scheme; b - picture; 1 - pressure gauge; 2 - experimental ultra-sound generator; 3 - microphone*



Microsoft Sound Mapper
Block Size 16384
Sampling Rate 96000
FFT Size 512
Window Size 512
Window Type Hanning
Scan Step 512
Display Height 256
Colours 0
Buffer off

***Fig. 4.*** *a - Ultramic 200 k microphone; b - ultra-sound generator calibration at 24 kHz*

Due to the energy carried by ultra-sounds, it is possible to appear some changes in the polymer structure. These changes could be observable in the final properties of the polymer. This study is oriented towards the identification of some changes in polymer electric conductivity. As any polymer is an electric insulator, the electric conductivity was evaluated via electric insulation resistance measurement using an insulation analyzer (Terra Ohm Meter) (Fig. 5).

***Fig. 5.*** *Electric insulation resistance measurements*

## 3. Results

The electrical conductivity of the analyzed samples was evaluated on the basis of the well-known formula:

$$\sigma=\rho^{-1} \quad (1)$$

where: ρ is the electrical resistivity derived from the measured electric insulation resistance R as:

$$\rho=RSl^{-1} \quad (2)$$

where: S is a cross-section area of the sample and l is its length.

The electrical conductivity of epoxy resin is plotted as a function of the generators number (as the frequency is the same for all the generators which means that the electrical conductivity is plotted as a function of ultra-sonic energy) and at different values of frequency (cs-control sample, 1G - ultra-sonicated epoxy resin with one generator, 2G - ultra-sonicated epoxy resin with two generators, 3G - ultra-sonicated epoxy resin with three generators and 4G - ultra-sonicated epoxy resin with four generators).

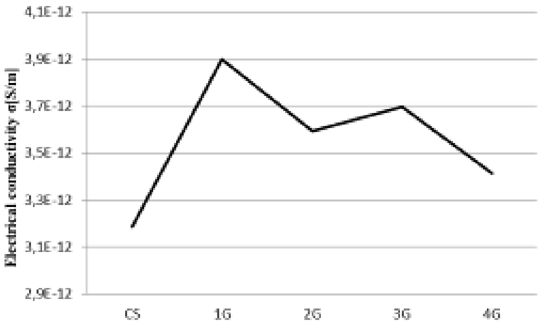


***Fig. 6.*** *Electrical conductivity of 24 kHz ultra-sonicated epoxy*

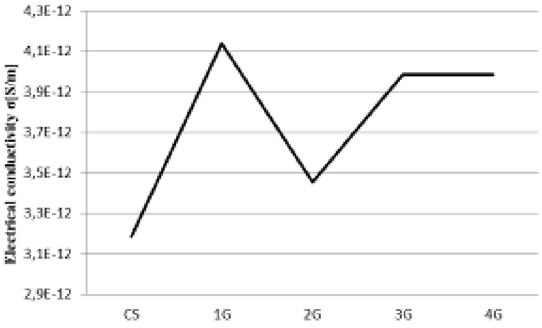


***Fig. 7.*** *Electrical conductivity of 26 kHz ultra-sonicated epoxy*

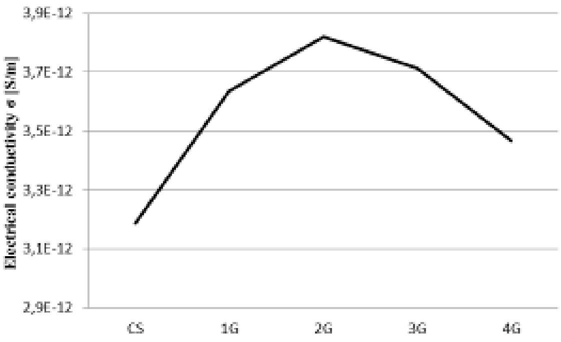


***Fig.*** *8. Electrical conductivity of 30 kHz ultra-sonicated epoxy*

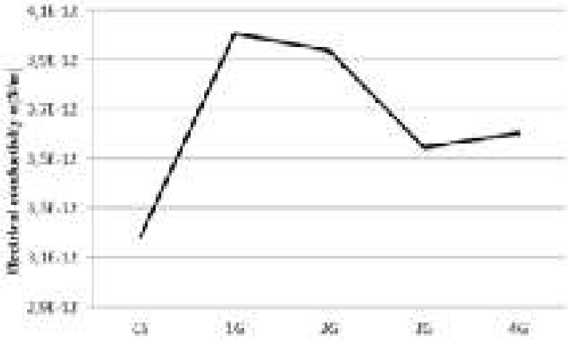


***Fig. 9.*** *Electrical conductivity of 42 kHz ultra-sonicated epoxy*

The results, as it can easily be noticed in the four graphs above (Fig. 6-9), show that practically the electrical conductivity of the polymer depends neither on the frequency of ultra-sounds nor on the total energy of ultra-sounds. That means that from the electrical point of view, the ultra-sound exposure of epoxy resin does not change its electrical properties. From the electrical properties point of view, the use of ultra-sounds to disperse fillers into the polymer matrix is an advantageous method since the ultra-sound exposure does not change the basic electric properties of the polymer. Small variations of the evaluated parameter could be associated with gaseous intrusions or small changes of polymer induced by the environmental conditions (temperature, humidity, atmospheric pressure) that are, at this moment, uncontrollable.

## 4. Conclusions

The paper presents the electrical properties of ultra-sonicated EPIPHEN epoxy resin cylindrical samples treated by different numbers of ultra-sonic air-jet generators for 45 minutes at different frequencies. Based on the above presented results, the use of air-jet generators treatment during epoxy resin curing was implemented. This investigation was focused on the possibility to eliminate the physical contact between the ultra-sonic device and the resin. This study has to be followed by others regarding the mechanical properties (tensile, compression, bending), tribological properties and thermal properties to completely characterize the effect of ultra-sound exposure of epoxy resin as intended matrix for some reinforced or filled composites. Since the modifications induced by ultra-sonic exposure on the electrical properties of polymer are negligible, the presented method can be used in a double way – first to disperse the particles of a potential filler or additive, and second to avoid the gaseous intrusions determined by the chemical reactions developed during the forming process.

## Acknowledgement

The work of Vasile BRIA was supported by Contract nr. 138963/2014, Cod proiect: POSDRU/159/1.5/S/138963.

The work of Iulia Graur and Adrian Cîrciumaru had been supported by the Project 12 P01 024 21 (C11) /31.08.2012 (code SMIS 50414).

## References

**[1]. Avila-Orta C., Espinoza-Gonzalez C., Martinez-Colunga G., Bueno-Baques D., Maffezzoli A., Lionetto F.**, *An Overview of Progress and Current Challenges in Ultrasonic Treatment of Polymer Melts*, Advances in Polymer Technology, Vol. 32, No. S1, p. 582-602, 2013.

**[2]. Rupprecht L.**, Conductive Polymers and Plastics, ISBN: 978-1-884207-77-8, 1999.

**[3]. Cusano A., Breglio G., Giordano M., Calabro A., Cutolo A., Nicolais L.**, *An optoelectronic sensor for cure monitoring in thermoset-based composites*, Sensors and Actuators, 84, p. 270-275, 2000.

**[4]. Cîrciumaru A.**, *Proiectarea, formarea şi caracterizarea materialelor compozite cu matrice polimerică (Note de curs)*, Ed. Europlus, Galați, ISBN:978-606-628-060-0, 2013.

**[5]. Wallace P. D.**, *Thermosetting resins*, Borden Chemical, p. 1-16, 2000.

**[6]. Chikhi N., Fellahi S., Bakar M.**, *Modification of epoxy resin using reactive liquid (ATBN) rubber*, European Polymer Journal, 38, p. 251-264, 2002.

**[7]. Mamunya Ye. P., Davydenko V. V., Pissis P., Lebedev E. V.**, *Electrical and thermal conductivity of polymers filled with metal powders*, European Polymer Journal, Vol. 38, p. 1887-1897, 2002.

**[8]. Mihalcu M.**, *Materiale plastice armate*, Ed. Tehnică, București, 1973.

**[9]. Zhang M. Q., Xu J. R., Zeng H. M., Huo Q., Zhang Z. Y., Yun F. C., Friedrich K.**, *Fractal approach to the critical filler volume fraction of an electrically conductive polymer composite*, Journal of Materials Science, Vol. 30, Issue 17, p. 4226-4232, 1995.

**[10]. Krevelen D. W. V., Nijenhuis K. T.**, *Properties of Polymers*, ISBN: 978-0-08-054819-7, 2009.

**[11]. Kenneth S. Suslick, Gareth J. Price**, *Applications of ultrasound to materials chemistry*, Annu. Rev. Mater. Sci. 29, 1999.

**[12]. Vitaliy G. Shevchenko, Sergey V. Polschikov, Polina M. Nedorezova, Alla N. Klyamkina, Alexander N. Shchegolikhin, Alexander M. Aladyshev, Vyacheslav E. Muradyan**, *In situ polymerized poly(propylene)/graphene nanoplatelets* *nanocomposites: Dielectric and microwave properties*, Polymer, 53, 2012.

**[13]. Masao Sumita, Kazuya Sakata, Shigeo Asai, Keizo Miyasaka, Hideaki Nakagawa**, *Dispersion of fillers and the electrical conductivity of polymer blends filled with carbon black*, Polymer Bulletin, Vol. 25, Issue 2, 1991.

**[14]. François Carmona, Jérôme Ravier**, *Electrical properties and mesostructure of carbon black-filled polymers*, Carbon Vol. 40, Issue 2, Third International Conference on Carbon Black, 2002.

**[15]. Francesca Lionetto, Francesco Montagna, Alfonso Maffezzoli**, *Ultrasonic Dynamic Mechanical Analysis of Polymers*, Appl. Rheol., 15:5, p. 326-335, 2005.

**[16]. Kuiming Liang, Clyde Oakley, Wayne Huebner, Hal Kunkel**, *Acoustic characterization of ultrasonic transducer materials II. The effect of curing agent in binary epoxy blends on acoustic properties*, Ultrasonics, 37, 1999.

**[17]. A. R. Horrocks, B. Kandola, G. J. Milnes, A. Sitpalan, R. L. Hadimani**, *The potential for ultrasound to improve nanoparticle dispersion and increase flame resistance in fibre-forming polymers*, Polymer Degradation and Stability, 97, 2012.

**[18]. Kolosov A. E., Sakharov A. S., Sivetskii V. I., Sidorov D. E., Sokolskii A. L.**, *Substantiation of the efficiency of using ultrasonic modification as a basis of a production cycle for preparing reinforced objects of epoxy polymer composition*, Chemical and Petroleum Engineering, Vol. 48, Nos. 5-6, Sept., 2012.

**[19]. Albert M. Lindrose**, *Ultrasonic wave and moduli changes in a curing epoxy resin*, Experimental Mechanics, Vol. 18, Issue 6, 1978.

**[20]. *****, *SR EN ISO 3744/2009*.