ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 6, Issue 5 , May 2019

EPR – Spectroscopic Research of Structure of Soot Filled Polystyrene

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ABSTRACT. In article EPR – spectroscopic research of structure of soot filled polystyrene, also a research of characteristics of the strongest broadening of a signal of EPR is considered. Polystyrene EPR ranges about soot content in number of V_1=0,01 are presented in article; 0,04; 0,09 and 0,20.

KEYWORDS: polystyrene, EPR spectroscopy, soot, structure, signal, volume, polymer material, composite.

I. INTRODUCTION

As is well-known in the developed countries of the world in microelectronics and instrument making it is widely applied scarce and expensive semi-metals, metal glasses, magnetite's and other unified metals, sensors and various devices on their basis.

On today's day the modern equipment needs simple, rather cheap and not scarce sensors and devices of different function. For achievement of these purposes more and more popular is use of nonconventional materials with the operated properties. It is possible to carry the filled polymeric materials which essential role in manifestation of these or those properties belongs to interphase layers in a system polymer-filler to such materials. The attempts of establishment of a role of an interphase layer made still generally leaned on indirect methods of a research of structure of the filled polymeric materials. However further development of science about the filled polymeric materials not mentally without deep understanding of structure of these materials. In this plan carrying out in-depth studies of structure and a condition of interphase layers between filler and polymeric binding especially including in technological a stage of ultrasonic dispersion of particles of filler in binding environments is necessary.

Today in the world development of plain, not scarce, durable cheap semi-metals, metal glasses, magnetite's and others of the unified metals, sensors and various devices on their basis used in instrument making and microelectronics represents special value.

In this regard development of effective composite polymeric materials with special properties and establishments of optimum process parameters of production from them of sensors and devices of different function for instrument making and the microelectronic industry is relevant and demanded.

II. SIGNIFICANCE OF THE SYSTEM

In article EPR – spectroscopic research of structure of soot filled polystyrene, also a research of characteristics of the strongest broadening of a signal of EPR is considered. The study of literature survey is presented in section III, methodology is explained in section IV, section V covers the experimental results of the study, and section VI discusses the future study and conclusion.

III. LITERATURE SURVEY

Characteristic of works of scientists as – LipatovYu.S., Kolupayev B.S., Korsakov V.G., Plnheiro M., Theocaris F.S. was the fact that they defined between phase layer role generally by indirect methods of a research of structure. Research works of type [1, 2] where authors given by this or that direct structural method of the analysis of a role of between phase layer in composites did not gain mass character yet.

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Further we will analyses works the concerning researches of structure the soot filled of polymeric materials by means of EPR of spectroscopy [2, 3] and a possibility of this method for identification of special characteristics of the composites able to attract interest of materials scientists.

First of all, it is important to have information on existence of the paramagnetic centers their quantity, the nature, exchange interaction at components of composites. So, in works [4, 5] where besides soot of DG-100 brand used by us, are investigated also soot of other brands, it is supposed that in the soot's there are three types of the paramagnetic centers:

1. Mobile electrons of conductivity;
2. The localized electrons of defective sites;
3. The localized not coupled electrons of chemical groups of freely radical character.

Width of lines at DG-100 soot on air is equal 22 Ersted [3], and at pumping out of a sample it decreases approximately by 10 times.

If with DG-100 soot to let in air the vacuumed ampoule, then width of the resonant line during fractions of a second increases up to a reference value. it demonstrates how authors consider, to that, the item of m of c. DG-100 soot are easily available to molecules of oxygen and it is undoubted, caused by the fact that such centers are generally localized on a surface of black particles. In work [6] it is reported that the surface of serial DG-100 soot is covered considerable the number of functional oxygen containing groups: carboxyl, khinonic, phenolic, lactone and others. All these groups resulting the surface of soot in power heterogeneity disappear at heat treatment. As for polystyrene (PS), even if it is subjected to oxidizing destruction [8] nevertheless the essential paramagnetic does not appear and also as many polymers, it in general diamagnetic [7]. Spasmodic change of spectral characteristics of EPR at 0,4% of soot in polyester pitch Kozlowski K. [3] carries to emergence of communications soot polymer at a recombination of radicals. In other work [9] it, with authors, reports about confirmation of model of conductivity of the system (polyester pitch acetylene soot) considering emergence of connections between particles of soot and macromolecules of pitch during polymerization with the assistance of free radicals. In [10] the model according to which transfer of electrons between particles of soot is carried out on the aliphatic chains of polymer connecting them is offered and data of EPR confirm conclusions of this model. The researches directed to identification of a role of oxygen on an EPR line form are given in work [11]. In work [12] "PS existence of the spin both localized, and delocalized with certain concentrates is revealed in".

IV. METHODOLOGY

The variety of the methods applied in the analysis of absorbing ranges does not allow to carry out comprehensive discussion of possible mistakes. Let's stop only on three main sources of mistakes and we will specify ways as we eliminated them.

One of considerable sources of mistakes is the scattered radiation of the monochromatic as which understand a part of the radiation getting to the receiver due to dispersion of light in the device. In simple prismatic spectrometers the share of diffused light makes often 3%. But, in used by us modern "Specord 75 IR" a share of diffused light it is less than 1%. This size increases a little only in long-wave area. Referring to work [8] we did not use calibration curves or methods of the relative analysis for amending values of extent of transmission.

In many spectrometers the absolute error of measurement of transmission of T at a research of samples is defined on a gleam by noise of the receiver and quality of a bunch of comparison. To minimize mistakes of this sort, thickness of a layer of a sample needs to be chosen such that extent of transmission lay ranging from 20% up to 60%. Out of this area the mistake quickly grows. Doing very thin cuts for clean polystyrene we tried to obtain elimination of this kind of mistakes. However, for composites, because of their fragility, receiving very thin cuts was extremely difficult. On the other hand, increase in content of soot in them, of course, led to stronger absorption of IR of beams and by that increased percent of admissible mistakes.

At last, as for errors of the strips of absorption connected with search. Absorption strips usually should be looked for in tables in the range of ±10 cm^{-1} from the specified value, except specially stipulated cases. In the analysis of IR spectrums this essential reservation is considered also in deviation cases from the specified figure, the conclusions are not provided in an assertive form.

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V. EXPERIMENTAL RESULTS

Ranges of EPR polystyrene (PS) about soot content in number of V_1=0,01; 0,04; 0,09 and 0,20 are presented in figures 1, 2, 3 and 4 respectively. The repeatability of results of measurement is sufficient high (periodic check within several years) and therefore radicals responsible for these signals can be considered stable. However, it is easy to notice, that in all listed cases it is about the amazing scientific phenomenon – the strongest broadening of the line of a signal of EPR (ΔHpp, E) because the reality is that DG-100 soot on air gives a singlet signal with ΔHpp=22 E, and polystyrene is not signaled at all, i.e. by it diamantine. In this case it is reasonable to find the answer a question – whether broadening is connected with imposing of signals from the centers of localization of spin various by origin or it comes from super thin interactions of the electrons localized on the paramagnetic centers organic origin.

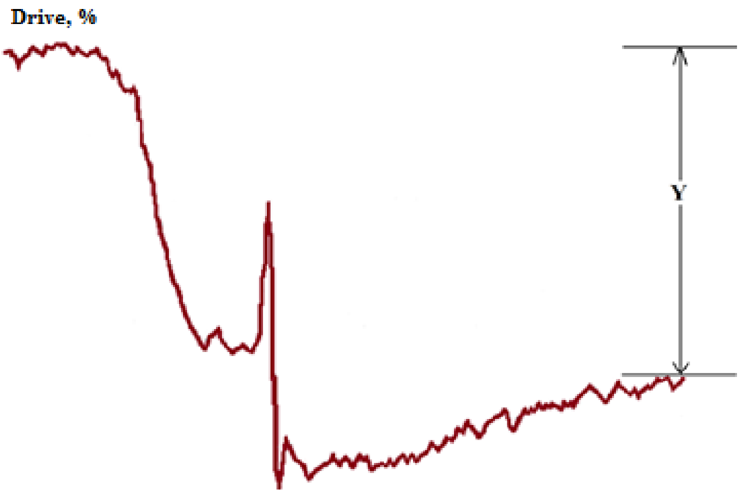

Figure 1. Spectrum EPR systems PS+soot (0,01)

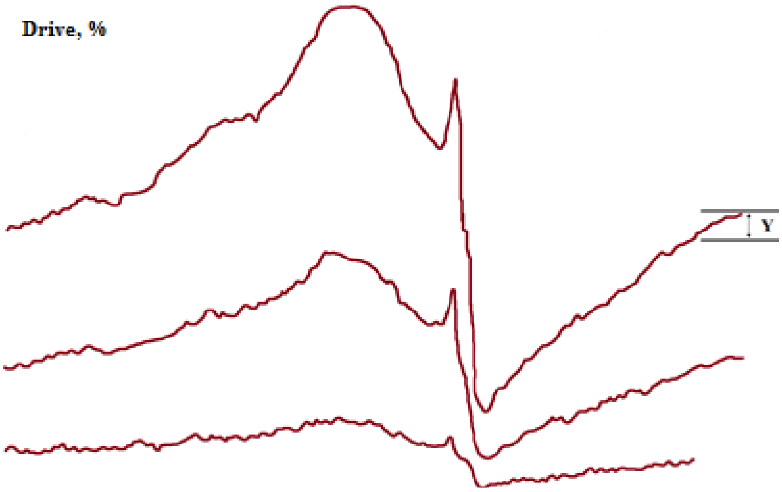

Figure 2. Dependence of intensity on the power of the microwave oven of the field of a big signal of PS+soot (0,04)

According to factory data DG-100 soot has the following structure: 94,8% of carbon (C), 0,9% of hydrogen (N), 4,3% of oxygen (O), less than 0,1% are gray, it is less than 0,1% of the mineral rest. In spite of the fact that the main part of composition of soot consists of elements of organic origin, nevertheless existence in it guards though slightest quantity (0,1%) of the mineral rest because even existence of the smallest quantities of some ions of transitional metals agrees to rather strongly established facts [13] can lead to strong broadening of width of the EPR

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line. To receive well-founded belief concerning the fact that the broadening of width of the EPR line observed by us in experiments is not connected with any elements of inorganic origin we did the following works:

First of all, we used services of the X-ray fluorescent analysis (analysis RFA) for more detailed analysis of structure of soot (you look at fig. 5). Apparently in figure 5 such elements as Kr, Nb, Ru, In and In, Cs and Cs, and in a quantitative sense all these elements are allocated, combined do not exceed 0,1% of the general composition of soot and apparently will be coordinated with factory data of filler. Secondly, we used services IR – spectroscopy ("Specord 75 IR") for definition in the studied objects of such elements as Fe, Ni, Co, Mn, Mg (you look at fig. 1-5). We did not manage to receive good IR spectrums for pure soot that, probably, is connected by strong absorption of IK of beams soot particles. However, in PS system IR spectrums soot (0,01) apparently from figure 2 in the range of lengths of waves from 400 cm^{-1} to 4000 cm^{-1} characteristic strips of absorption of Fe, Ni, Co, Mn, Mg or connections on their basis are not shown. The facts established by means of IR – and the X-ray fluorescent analysis, stubborn deny a possibility of origin of so wide signal of EPR (you look at fig. 1-4) from the elements Fe, Ni, Co, Mn, Mg.

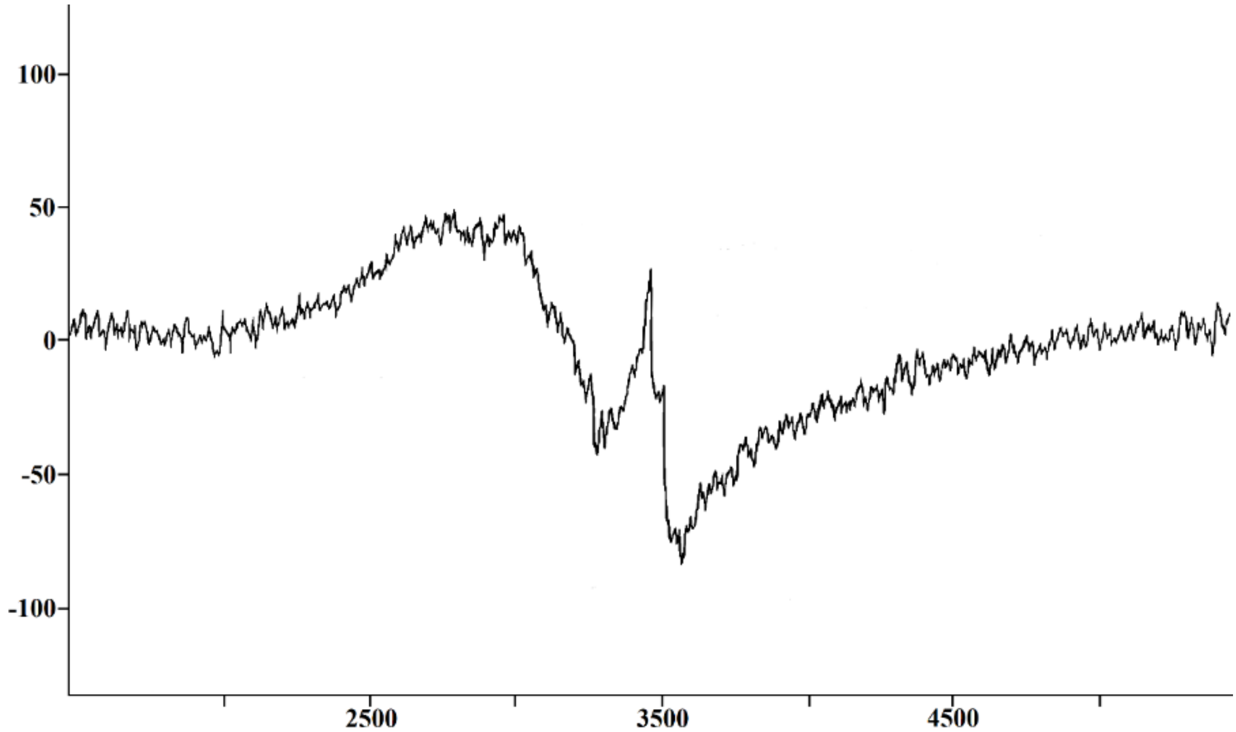

Figure 3.Spectrum EPR systemsPS+soot (0,09)

ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
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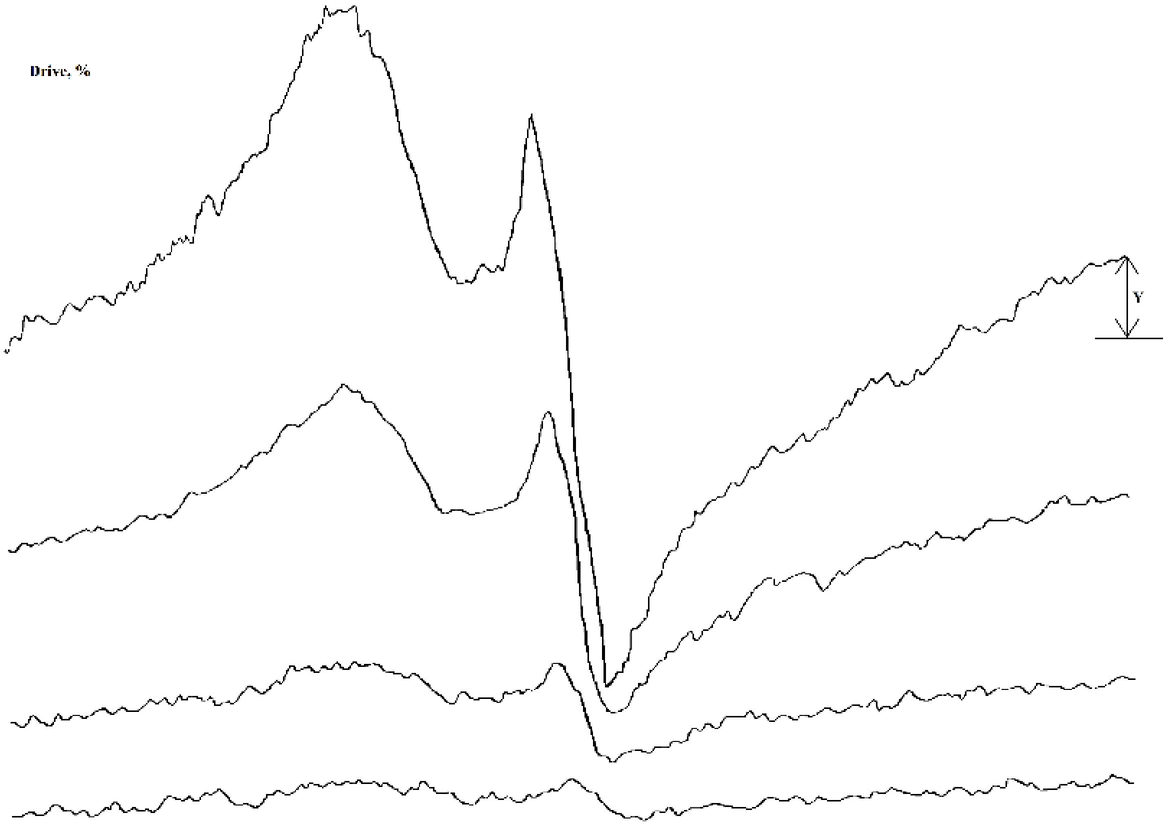

Figure 4. Spectrum of a big signal of EPR PS+soot (0,20) in dependence on field microwave oven power

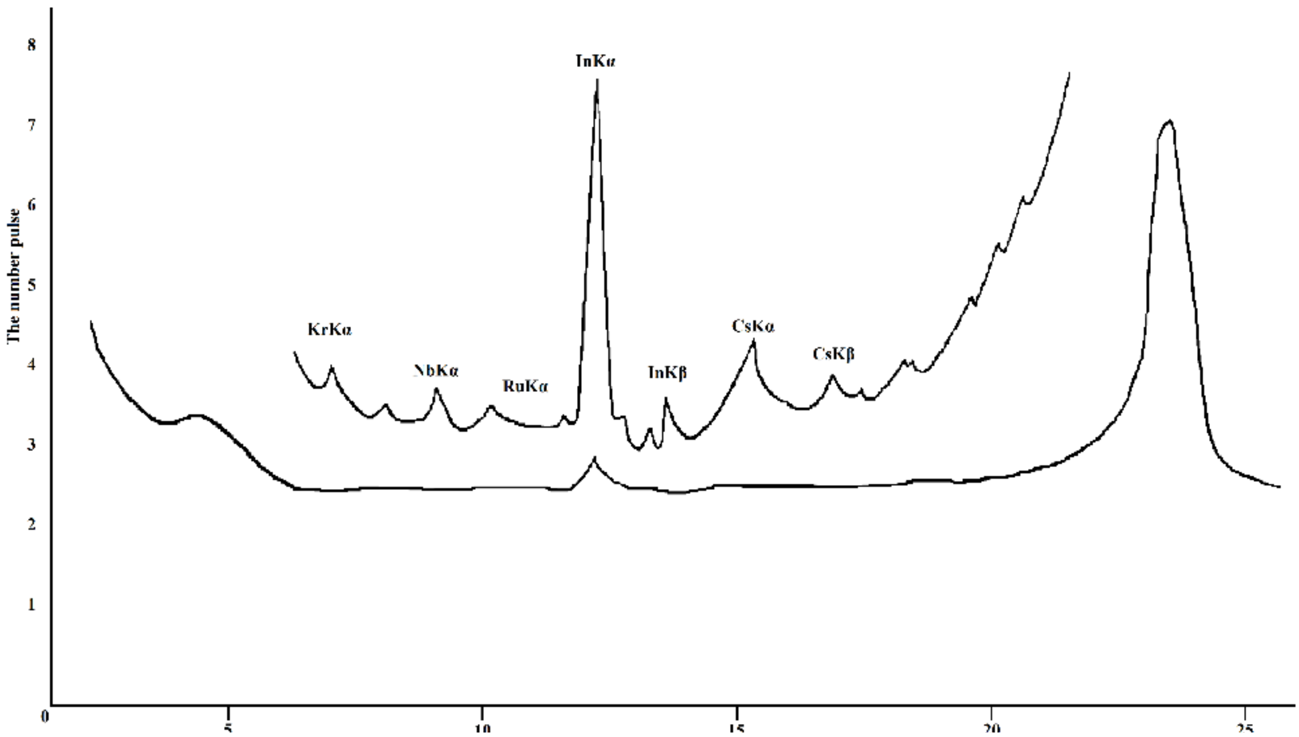

Figure 5. X-ray fluorescent analysis of structure of soot

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VI. CONCLUSION AND FUTURE WORK

Our further efforts on identification of the reason of broadening will be connected with use of opportunities of the spectroscopy of EPR. One of ways of establishment of the nature of broadening of a signal of EPR is studying of growth rate of intensity of components of a signal of EPR depending on power given super high-frequency of the field. In case broadening happens for the account imposing of signals from various items of parametric center, then usually their rates of change of intensity each other do not correspond [3, 13]. On the contrary, when the widened signal of EPR represents result of superfine interaction, their rates of change of intensity correspond. Apparently in figures 1-4, in our case the last case is implemented. In this connection it is represented to us not possible origins of components of the general signal of EPR from various centers by the nature in localization of free radicals.

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