

Obtaining and characterization of composite materials with polymeric matrix

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Abstract— Composite material has attracted increasingly remarked interest over the last few decades and set it apart in its own class due to its distinct properties. World annual production is over 10 million tonnes and the market has in recent years been growing at 5–10% per annum. The paper shows up the research results on processing and characterization of composite materials with polymeric matrix (silicone rubber). The synthetic polymeric matrix used at the obtaining composite is represented by a bicomponent silicone elastomer that strengthens itself at the room temperature by means of a poly condensation reaction. The materials obtained in laboratory contain metallized netting like reinforcement material and powdery nanocarbon and iron silicon as filling agent. Measurements were conducted to determine the transmission diminishing carried out and X-ray diffraction.

Keywords— filling additions, plated netting, polymeric composites, powdery nanocarbon and iron silicon

I. INTRODUCTION

Defining composites as an engineering material a progressive framework that starts with a generality and goes to specific is needed. In vast terms, the general definition of a composite is: “Two or more different materials that when are combined become more strong than individual materials” [1]. Composite materials are found in nature (different rocks solids). People have used them since ancient times, as the mixture of clay and reeds (adobe about 5000 years before Christ.), plywood (about 1500 years before Christ). After 1900, the polymer composites have been discovered. At the beginning of the World War II (1940) were first used in the manufacture of composite fuselage of a Spitfire fighter plane. These programmed materials with traditional properties, have entered the top high tech fields, such as aerospace technology, microelectronics, nuclear engineering, telecommunications, medical equipment impanturilor, automotive, marine vessels, chemicals, furniture, in construction, seismic protection of buildings, office equipment, sports materials industry and household products, but current efforts to save energy and raw materials were brought back to the present problem acuity.

Interest in multiphase composite materials has expanded lately as these advanced materials can be tailored to exhibit controlled mechanical, electrical and thermal properties. [2]

In principle and the materials conventional like metal alloys, are composite materials, but new composite material is characterized as follows:

- are made by humans;
- are made of at least two different elements, with a distinct interface separating the components;
- have properties that do not have any of the components are initially separated. [3]

Composite materials can include all types of materials consisting of two or more components. Constituents must have compatible structures, providing a link to interfacial resistance. Thermal diffusion should be linked so that the heating or cooling will not lead to cracking, cracking, breaking any of the components, or connections between them. This material composite must submit chemical stability during by the processing and operation. [4]

Making composite materials has become the basis for many technical and economic considerations, among which we mention: the need for materials with special properties, impossible to reach with traditional materials, the need to increase safety and reliability in operation of various machinery and equipment, need reducing consumption of scarce materials, because are expensive or precious, opportunity to reduce labor consumption and to reduce the time of manufacture.

Definition is too common to depict the specialized shapes of materials for that the composites industry endorses the respective names. It is necessary a definition that adequately segregates these structural materials from other the engineering ones.

Brent Strong, stress that “ the term implies that the composite material is macroscopically identifiable, meaning the materials are not only differentiated at the molecular level but have different proprieties and they are generally mechanically separated” [5].

In this case reinforcer may include fibers or particles and polymer matrix could be, ceramic or metal.

Particle reinforced composites emerged as viable alternatives to the classical materials due to their mechanical, electrical and thermal properties that can be tailored such as to fulfil a wide range or working conditions, from extreme temperatures

to normal ones, in applications such as force/pressure sensors, electro-magnetic shields, toothed wheels, etc. [6]

Definition is too common to depict the specialized shapes of materials for that the composites industry endorses the respective names. It is necessary a definition that adequately segregates these structural materials from other the engineering ones.

Considering that a composite is a combination of a reinforcement material with a matrix, becomes necessarily to defined the reinforcement and matrix terms.

Engineerically, one of function as reinforcer in a composite is to take over loading effort transferred through matrix. [7].

Generally, the particles have a low ratio of aspect (ratio length/diameter) and are almost spherical as shape. Handling as filling materials, the particles can be both organic materials and inorganic.

The presence of particles in matrix determine the isotropic properties. This means that the materials will have the same properties of tensile, shear and compression on all axes X, Y and Z.

Therefore, a matrix reinforced with particles will be homogeneous similarly to metals. Having a low geometric aspect (almost spherical-shaped), the particles cannot effectively transfer loading from particle to particle to result a homogeneous structure.

The function of matrix in a composite is to ensure a relative rigid medium that is able to transfer the effort to fibrous compounds of material.

Matrix embeds the reinforcer making synergy of physical properties between the two materials. To make composites, an important aspect of combination matrix and reinforcer is that between a chemical bond can be made.

Now, considering the technical aspects of the characterization of composite materials, their definition can be made more precise: "composites are a combination of fiber reinforcement in a polymer matrix, which is a report of the appearance reinforcer able to take transfer of effort between the fiber and the fibers are chemically bonded to polymer matrix [8]

The latest evolutions from different fields of science and technique demonstrated the importance obtaining of some materials with high performances that can meet all applications, but mainly actions some different physical and/or chemical external factors. The composite materials class responds better all requires and the most used materials for obtaining matrixes are polymers.

II. MATERIALS COMPOSITE FOR SHIELDING

At present, all over the world, concerns in absorbents field of electromagnetic waves are directed to directon of new materials with low cost and maximum efficiency both by designing of shieldind means as **diverse** and also for **specific** applications.

Therefore, the Materials Research Laboratory of Building of US Army has **ongoing** a complex program of research with the following goals:

- development of new materials: conductive polymers and covering for the shielding componnets; The materials comprise amorphous materials and inserted graphite;

- shielding of standard building materials: foils of aluminium, copper, netting of metallic threads and ceilings of metallic layers.

The first papers and practical achievement of reflectants and absorbents of electromagnetic field before the World War II, following diferently insurance of military security. USA, Germany and England give an important volume of work and big funds to vary these materials.

The United States of America tries to obtain light absorbent materials with low cost usable both at marine vessels and airships. The most recent achievements are:

- HARP Cloth Material made by MIT Radiation laboratory on the basis of conductive sparkles (aluminium, copper or ferromagnetic materials) scattered in a polymeric binder (rubber/plastic) sprayed on surfaces, especially used to reduce the electromagnetic interface;

- "Salisbury Screen material", as multilayer consisting in a layer of glass fibres impregnated with graphite, a foam layer and a conductive counterface.

England was also active in the absorbent field. Since 1947, British Radar, Signal Research Establishment have sponsored research and experiments about these materials and British Navy used the first materials resulted in the field of resonant absorbents to reduce the false echoes and perturbations made at the reflection of radar energy of masts. The recent results refer to materials on the basis of composite rubber with ferrite and carbonyl iron, carrying out absorbents of 20-30dB in a large range of frequency (1-18 GHz).

The composite materials exhibiting electromagnetic properties have different applications in electronics. Electronic composites, whose properties can be controlled by thermal or electromagnetic means, play an important role in micro- and nano-electromechanical systems (MEMS/NEMS) such as sensors, actuators, filters and switches. [9]

The goal of present paper consits in the realization a composit material with polymeric matrix. The synthetic polymeric matrix used at the obtaining composite is represented by a bicomponent silicone elastomer that strengthens itself at the room temperature by means of a poly condensation reaction.

III. STRUCTURAL CHARACTERIZATION OF COMPOSITE MATERIALS

A. Polymer matrix

To carry out composite materials with polymeric matrix silicone rubber (siloxanic) as polymeric matrix of RHODOSIL RTV 3325 type that has a **beige-colored** viscous aspect have been used.

The best reticular is achieved at a temperature of 23⁰C in humidity conditions of about 50%. Catalyst 60 is on base of C₆H₁₂N₂O₄ (dinytrohexan), this hurries the polymerization reaction (strengthening) of polymer. A ratio of (between catalyst and silicone rubber) = 1:20 PARTS is used.

B. Reinforcing agent

As reinforcer, it is used plated netting (PN).

The metallized texture are obtained by metallic coating/plating (galvanizing coating) of fibers or polymers textures, basalt, glass, silica, graphite, etc.

The plated netting helps to improve screening against the electric, magnetic, electromagnetic fields, infrared emissions and the biologic protection against their negative effects on living creatures.

C. The filling materials

Agents filling are nanocarbon the dust and iron powder silicon. Using materials as particles has known a great extension since performs some important advantage such as:

- Low cost;
- Simple technologies of introduction particles into matrix;
- Possibility to obtain isotropic materials.

The filling materials are used to induce some properties, such as increasing capacity of electromagnetic shielding different as their nature and configuration.

The preparing the raw materials of filling in essence consists in the eliminating hygroscopic humidity. This is carried out by drying the raw materials. The operation is very important as from the bad dried raw materials cannot be obtained products of quality.

Particle reinforced metal matrix composites are attractive materials for many engineering applications. The apparent deformation behavior of composite is primarily determined by the respected properties of constituents. However, the architecture of microstructure, i.e. volume fraction, size, shape, orientation and arrangement geometries of constituents affect the stress and strain portioning of constituents.[10]

IV.METHOD OF WORK

Were processed several types of materials with different compositions. The materials were processed in the laboratory and contain the reinforced metallic mesh, which has been impregnated with a mixture of nanocarbon the dust and iron powder silicon, based siloxanic rubber with containing filling additives.

Thus, we started from the mesh fabric metallic, PN.

The powdery fillings (nanocarbon, - N and iron powder silicon – SI) have been dispersed into polymeric matrix – silicone rubber, abbreviation SN , and SI . Mixing has been made at the room temperature. Carrying out composites has been made by the laying mixtures on the plated netting (PN) by means of doctor blade technique. The following abbreviations/acronyms, such as PNSN means polymeric mixture of silicone and nanocarbon laid on the plated netting and SN is polymeric mixture with silicone rubber and nanocarbon without plated netting will be used, such as PNSI means polymeric mixture of silicone and iron powder silicon laid on the plated netting and SI is polymeric mixture with silicone rubber and iron powder silicon without plated netting will be used.

The composite materials have been polymerized in open atmosphere at a temperature of about 22 – 25°C, for 24 hours. Materials with dimensions of 350 x 350 x 3 mm.

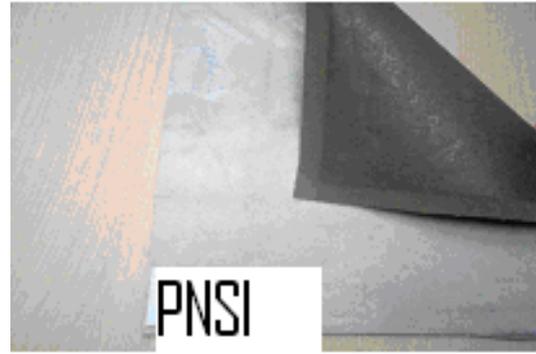


Fig. 1 Settling down mixture of siloxanic rubber – iron-silicon on only face of plated netting (PNSI)



Fig. 2 Laying of siloxanic rubber and iron-silicon mixture without plated netting (SI)

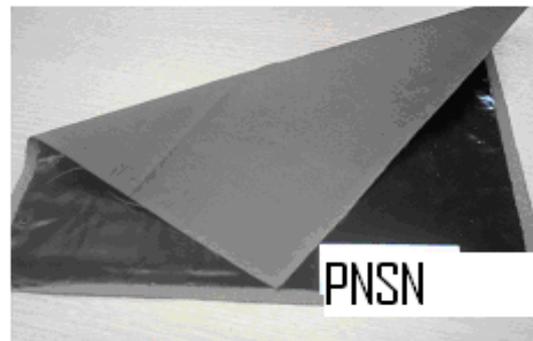


Fig. 3 Settling down mixture of siloxanic rubber – nanocarbon on only face of plated netting (PNSN)

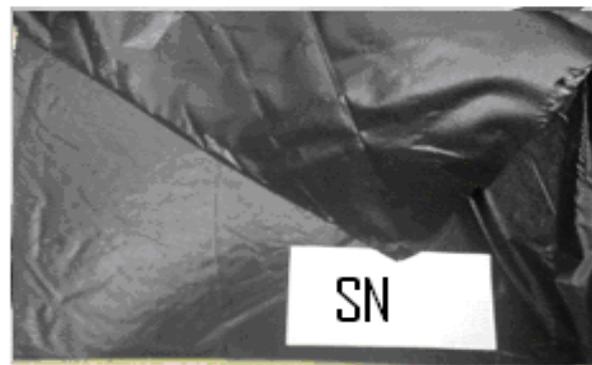


Fig.4 Laying of siloxanic rubber and nanocarbon mixture without plated netting (SN)

IV. RESULTS AND INTERPRETATIONS

A. X-ray diffraction

X-ray diffraction is a determination method of crystalline materials structure. The physical principle is based on the interaction of monochromatic X-ray with atoms electrons in structure. These atoms **scatter** X-ray that further **interwork** between them coming out diffraction maxim at a certain angle against the propagation direction of incident ray.

The position diffraction peaks corresponds to interplanar distances from crystalline structure. Therefore, any crystalline structure will be represented in the diffraction spectrum by a peaks set corresponding to each a certain crystallization direction and will be suitable identified be means of data base (ICDD – International Center of Diffraction Data).

The molibdenum tube (Mo) is used for sample that contain cobalt (Co) or iron (Fe₀, because the radiation of copper tube (Cu) performs the maxim absorbent for theses elements and consequently it is lost from signal.

For analysis:

- Diffractometer D8 ADVANCE of AXS Bruker type has been used;
- anodic tube used was of molybdenum;
- pass of 0.04°;
- scanning speed 1 sec/pass;
- scanning has been achieved between 2-50° (2θ).

Method is in accordance with SR EN 13925/2003.

Uncertainty of measurement positions of diffraction lines was 0.005%.

I made a calculation for interplanar distance for all the angles wich appear on diffractometry, to see if Bragg's law is applicable to diffractogramelor presented.

Following calculations interplanar distances, d, with Bragg's law indicates that the calculated values are very close to the values that appear in the index files.

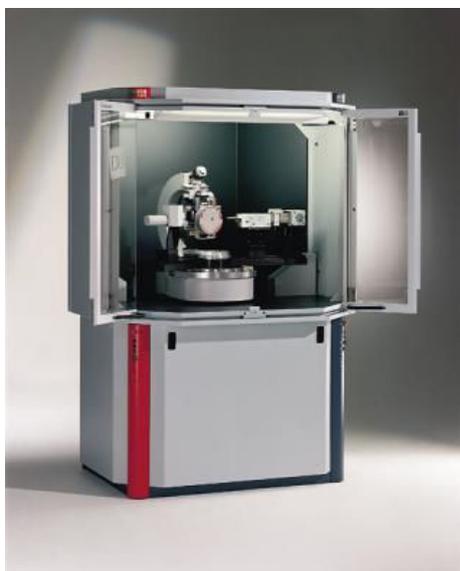


Fig. 5 Diffractometer D8 ADVANCE of AXS Bruker type

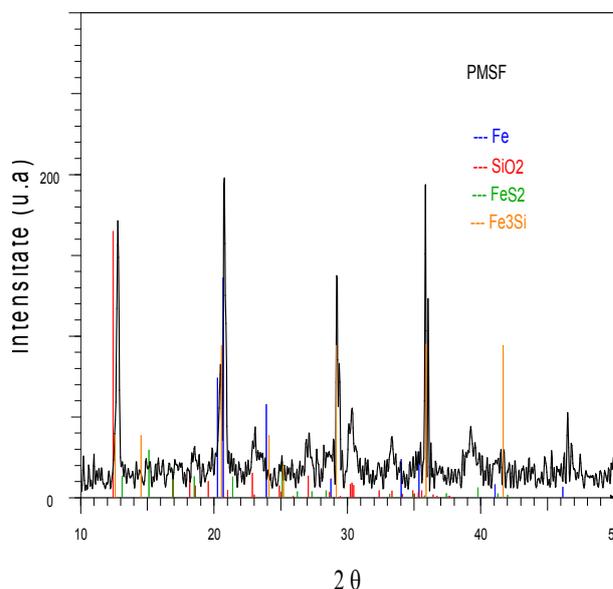


Fig.6. PNSI diffractogram

According diffractometry 6, peaks have maximum intensity for silicon oxide 12,34°, 16,77° for FeS₂, iron at 20,11°, and for Fe₃Si the maximum diffraction peak is at the angle of 35,78°. According to records of indexes files, FeS₂ Fe₃Si crystallizes in cubic system, and silicon oxide crystallizes in the tetragonal system.

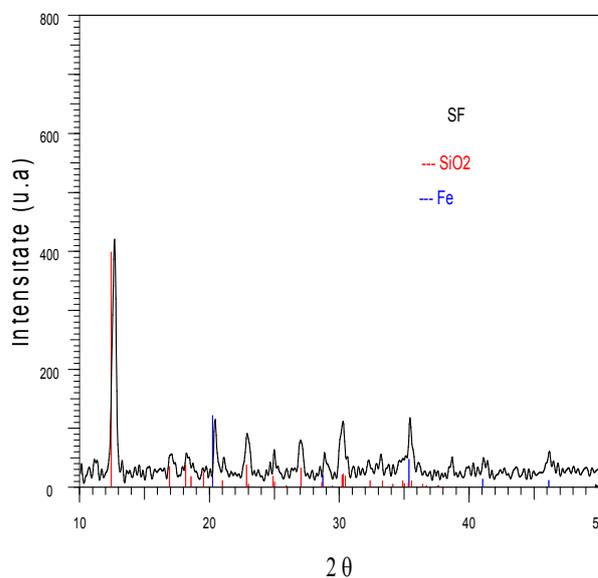


Fig.7. SI diffractogram

Diffractometry 7 phase shows that is a majority proportion of silicon oxide and a the minority phase is represented by iron. According to index files, note that the maximum of diffraction peaks for iron oxide is at an angle of 10,12° compound wich crystallize in hexagonal system and iron has the maximum

peak value of the angle is $19,88^{\circ}$ and the iron crystallize in the cubic system.

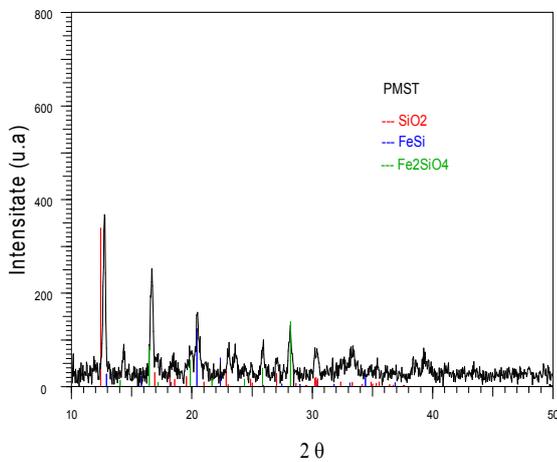


Fig.8. PNSN diffractogram

According diffractometry 8, shows that diffraction peak for silicon dioxide is the $12,19^{\circ}$ for Fe_2SiO_4 is the $16,14^{\circ}$ and for the FeSi is the $20,61^{\circ}$.

According to index files, silicon dioxide crystallizes into the hexagonal system, Fe_2SiO_4 crystallizes into the ortorombic system and silicate of iron in the cubic system.

We find that at the same angle of $20,61^{\circ}$ have upper limit for FeSi phase, but a somewhat lower proportion of silicon dioxide.

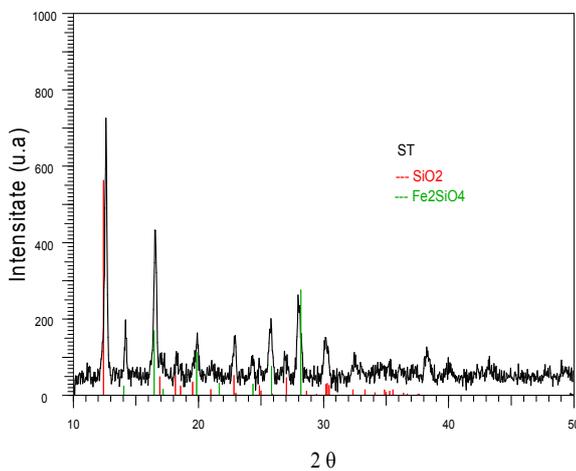


Fig.9.SN diffractogram

For diffractometry 9, it is noted that the diffraction lines correspond to the angle of $16,56^{\circ}$, both phases are present here (SiO_2 and Fe_2SiO_4). From the index files we found that the proportion of the major phase is (90.62%) and is allocated to silicon dioxide and the minority phase is in proportion (9.38%) represented by the Fe_2SiO_4 .

In the diffractograms presented and carried out X-ray tube with molybdenum anode, there is some shift of diffraction lines, which can occur either due to impurities in the compounds,

either due to sample preparation problems, or if a sample is present in very small quantities. We note that calculations made to determine the interplanar distance, are good values and have the same values, with tabulated values from the index files.

Interplanar distance calculation was done to highlight the Bragg's law, ie the relationship link that exists between diffraction angle and wavelength.

For all diffractogram can see that the majority phase is SiO_2 , which occurs at the angle of $12,2^{\circ}$. For diffractogram 6-7, besides SiO_2 is found Fe also to $21,07^{\circ}$. In cas of diffractogram 8-9 Fe_2SiO_4 phase is observed and occurs at $28,2^{\circ}$.

B. Measurements of transmission diminishing in frequencies range 1-18 GHz.

The most perturbing effects of electromagnetic fields occur in the frequency intervals between some tens of MHz and hundreds of MHz, an interval characteristic to many receiving and transmitting equipments. The directives and regulations issued by international organizations provide specific measures that must be taken in order to minimize the influence of the perturbing electromagnetic fields generated by a wide variety of equipments which are accordingly classed to harmonize with the requirements of electromagnetic compatibility.

Recent advances in science and technology have led to the development of a new class of materials – nano-materials, obtained by combining material particles having different electrical and magnetic properties. Due to their structure, nanomaterials present physical properties conferring them extreme values of electrical conductivity and magnetic permeability, which can be attained by controlling their internal structure and composition.[11]

The analysis goal is to determine the radio transmission diminishing in frequencies range 1-18GHz, with a view to establishing applicability field of composite materials with polymeric matrix obtained.

The method principle consists in the diminishing measurement of radio transmission chain in two situation: without material and with the material inserted on the transmission chain. The transmission chain can be free space or waves guide.

The (emission and receiving) antennas are set up in the shielded room sitting face to face. The distance between antennas remains the same during measurements. By means of signal generator (Frequency range: 250kHz – 40GHz) a amplitude signal constant in the frequency range 1-18GHz is generated and this signal captured by receiver antenna.

The sample is set up between antennas then an identical signal of the same amplitude as at measurement without sample and measure again the signal noting the obtained values is generated. The diminishing is calculated and plots of diminishing graph depending on frequency.

As the listing is in tables 1-3, the experimental results of transmission diminishing measurements in the range 1-18 GHz made on composite polymeric materials, PNSI,SI, PNSN and SN are performed.

Table 1

Frequency (GHz)	1	1,6	2	2,6	3	3,6	4	4,6	5	5,6	6
Attenuation PNSN(dB)	1.448	7.886	1.94	4.97	5.56	2.3	4.65	8.03	12.14	14,947	16,933
Attenuation SN (dB)	1.448	7.886	1.94	4.97	5.56	2.3	4.65	8.03	12.14	14,947	16,933
Attenuation PNSI(dB)	3,35	11,011	3,987	5,93	7,09	16,62	14,68	17,24	19,58	19,972	23,393
Attenuation SI(dB)	2,348	0,672	0,885	7,39	0,64	0,52	0,66	1,04	2,51	0,217	0,469

Table 2

Frequency (GHz)	6,6	7	7,6	8	8,6	9	9,6	10	10,6	11	11,6	12
Attenuation PNSN(dB)	18,63	22,048	22,355	23,075	28,5	27,295	30,436	33,83	33,77	33,5	34,43	38,33
Attenuation SN (dB)	0,25	0,524	0,911	0,653	0,549	0,18	0,445	1,43	2,38	1,26	2,9	2,33
Attenuation PNSI(dB)	28,58	31,522	23,356	27,339	27,339	28,895	31,973	34,86	36,21	35,49	35,99	33,15
Attenuation SI(dB)	0,341	0,417	0,709	0,058	0,058	0,268	0,527	1,99	2,38	1,86	4,15	2,32

Table 3

Frequency (GHz)	12,6	13	13,6	14	14,6	15	15,6	16	16,6	17	17,6	18
Attenuation PNSN(dB)	42,36	39,27	36,27	40,53	39,16	41,46	38,54	41,34	38,11	34,67	26,76	23,63
Attenuation SN (dB)	1,31	1,41	2,72	1,35	2,49	2,82	3,15	9,27	3,88	5,08	6,31	7,46
Attenuation PNSI(dB)	30,75	31,15	34,43	36,64	32,72	36,36	36,59	35,12	28,94	29,15	19,28	12,76
Attenuation SI(dB)	1,57	1,41	2,67	2,17	2,83	3,36	3,67	4,46	3,99	5,47	4,43	5,48

As a material can be considered efficiently in point of radio diminishing, this must perform a minimum diminishing of 20 dB in band of work frequency.

The behaviour composite system with polymeric matrix PNSI , as tables 1-3, performs minim values of radio diminishing (maximum of transparency) of 3.35 dB at a frequency of 1 GHz for system and the maxim value of radio diminishing is presented by the same composite material at a frequency of 14 GHz corresponding to value of 36.64 dB.

The behaviour composite system SI, as tables 1-3, performs minim values of radio diminishing (maximum of transparency) of at 7.39dB a frequency of 2.6 GHz for system and the maxim value of radio diminishing is presented by the same composite material at a frequency of 8 GHz corresponding to value of 0.058dB .

The behaviour composite system with polymeric matrix PNSN , as tables 1-3, performs minim values of radio diminishing (maximum of transparency) of 1.448 dB at a frequency of 1 GHz for system and the maxim value of radio diminishing is presented by the same composite material at a frequency of 12.6 GHz corresponding to value of 42.36 dB.

The behaviour composite system SN, as tables 1-3, performs minim values of radio diminishing (maximum of transparency) of at 0.18 dB a frequency of 9 GHz for system and the maxim value of radio diminishing is presented by the same composite material at a frequency of 6 GHz corresponding to value of 16.933 dB .

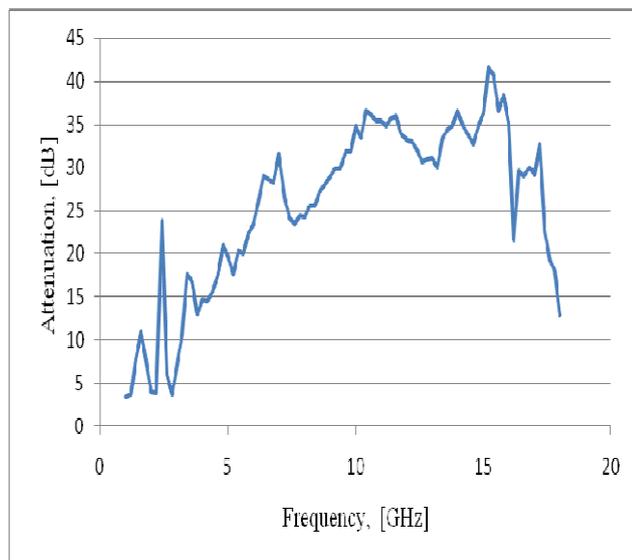


Fig. 10. Variation of the transmission attenuation (dB) frequency, the sample material, PNSI

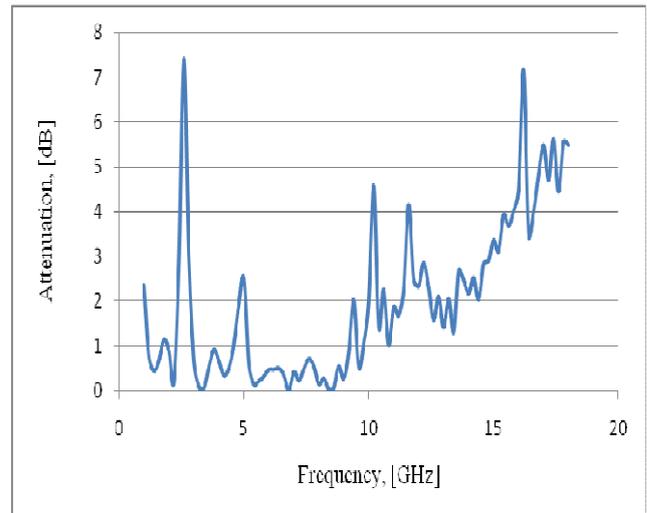


Fig. 11. Variation of the transmission attenuation (dB) frequency, the sample material, SI

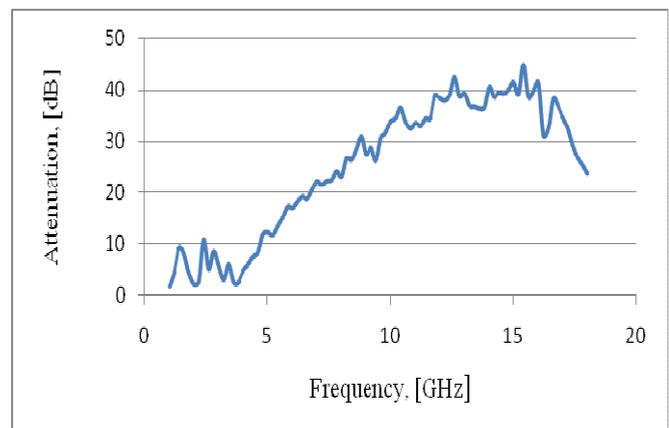


Fig. 12. Variation of the transmission attenuation (dB) frequency, the sample material, PNSN

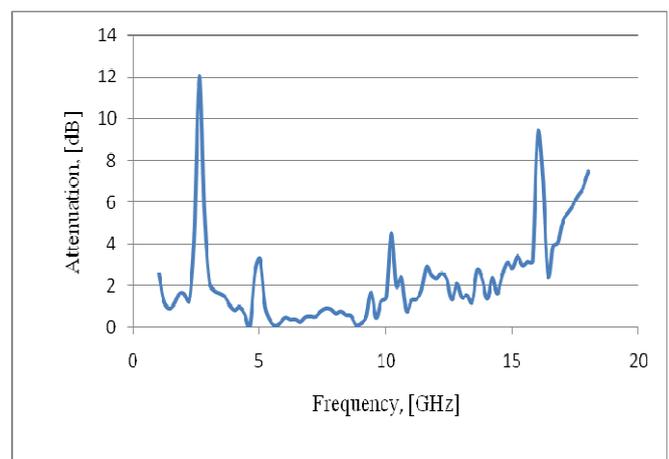


Fig. 13. Variation of the transmission attenuation (dB) frequency, the sample material, SN

Table 4. Evaluation of transmission diminishing of composite systems obtained on frequencies range covered between 1-18 GHz

Sample	Diminishing (dB) measured on frequencies range		
	1-6 GHz	6,6-12 GHz	12,6-18 GHz
PNSI	11,90	30,98	30,32
SI	0,33	1,16	3,45
PNSN	7,54	28,84	36,84
SN	1,27	1,10	3,93

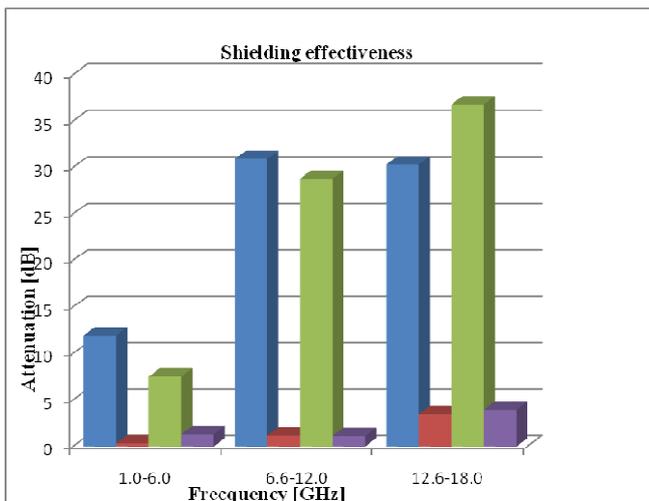


Fig. 14. Variation of shielding effectiveness for materials PNSI (bleu), SI (red), PNSN (green), SN (purple)

According to the graphs and and table 4, we can say that, in terms of attenuation by transmission, powders by nanocarbon wich are conductive powders are superior the powders iron silicon wich have magnetic character.

The value of radio transmission diminishing on frequency range 1-6GHz according to 4 and for the system PNSI of 11.90 dB and for SI only 0.33 dB on the frequencies range 6.6 – 12 GHz are met high values for both systems and namely 30.98 dB for PNSI and 1.16 for SI. On the frequency range 12.6 – 18 GHz it is seen that the value of transmission diminishing performs values increasing for SI system of 3.45 dB and for PNSI values are given decreasing up to 30.32 dB.

The value of radio transmission diminishing on frequency range 1-6GHz according to 4 and for the system PNSN of 7.45 dB and for SN only 1.27 dB on the frequencies range 6.6 – 12 GHz are met high values for both systems and namely 28.84 dB for PNSN and 1.10 for SN. On the frequency range 12.6 – 18 GHz it is seen that the value of transmission diminishing performs values increasing for PNSN system of 36.84 dB and for SN values are given increasing up to 3.93 dB. According to the diminishing graphs (10-13), it is observed that for systems which have as reinforcer wire netting, the diminishing decreases with increase of frequency and systems that have not reinforcer the values of diminishing increase with the frequency increasing.

The composite systems developed in this research perform, generally, low values of diminishing for samples without plated netting.

V. CONCLUSIONS

The purpose of this paper is to carry out complex structural composites, polymer with matrix and metallic mesh represented the core network (plated netting) with filling agents micron powders.

The materials are flexible, wearing light, they can be of different thicknesses.

The composite systems developed in this research perform, generally, low values of diminishing for samples without plated netting.

According to the diminishing graphs (10-13), it is observed that for systems which have as reinforcer wire netting, the diminishing decreases with increase of frequency and systems that have not reinforcer the values of diminishing increase with the frequency increasing.

At present, in many countries worldwide research programs in the electromagnetic pollution field of environment having as main objectives the evaluation and application some reducing methods of electromagnetic fields values operated by power main, stations, transducers, electric – power – supply network of dwellings as well as household appliances.

Thus, by obtaining and using these materials is envisaged, reducing the electromagnetic field produced by all sources that surround the human subject.

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Professional experience

1982 – Eng at "Calimani" – Sulphur Mine.

1982 – 1984 – Applications Engineer at "UPET" Targoviste – for "Heavy Water Plant" – Drobeta – Turnu Severin.

1984 – 1994 - Applications Engineer at "UPET" Targoviste – for "Nuclear Power Station" Cernavoda.

1993 – 1998 – English Language Substitute.

1998 – so far - Materials Science and Engineering, Mecatronics and Robotics at "Valahia" University of Targoviste as part of RESEARCH CENTER ACADEMIC SCHOOL OF MATERIALS SCIENCE, Director Prof.dr.doc.eng. OPREA FLOREA.

Addition education

- Master – *Research on atmospheric pollution level in metallurgical areas of Romania. Industrial ecology, Sustainable Development*

- Ph.D. candidate, Research Theme – *Heavy Metals on Environmental and Health Risks*

Publications

- 24 ISI papers, 7 International conferences., 6 papers on site <environmental-expert.co>., 5 papers B+, 4 Research contracts – co-worker.

- 2 books, - "Explanatory Technical English Dictionary" (1100 pages).

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