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Fabrication of Polymer MagneticNano-composites Containing Carbon Nanoparticles Doped with Cobalt Nano-clustersandStudytheirConductivity,Self-Healingand Adhesion Properties

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ABSTRACT
Thetechnologyoffabricationofpolymernanocompositesonbasisofcarbonnanoparticles doped with cobalt clusters, synthesized by original ChemicalVaporeDeposition(CVD)technologydevelopedbyauthors,waselaborated.Carbon shells provide both the protection of ferromagnetic impurities fromaggressiveenvironmentandnewuniquepropertiestothehybridenanostruc-tures. The self-assembling of magnetic clusters coated by carbon shells presentsjustsuchexamplewhichcouldbeusedinthecontemporarymaterials,forex-ample, in strong magnets, analytic instruments (nuclear magnetic resonancetomographs)andnanosensors.Theirgoodconductivity,self-healingandadhe-sion properties were demonstrated by applying the combined action of tem-perature, pressure, steady and alternating magnetic fields to stimulate diffu-sionofmagneticnanoparticlesindirectiontodefectsites.Duetotheseprop-erties fabricated magnetic polymer nanocomposites could have perspectiveforpotential.
Keywords: MagneticCarbonNanopowder,PolymerComposite,StimulatedDiffusion,Self-Healing,Self-Organization,Resistance

I. INTRODUCTION

In thelastdecade, theinvestigationof suchnew nanostructureformsof carbon as nanoparticles, nanotubes, nanothreads became very topical. This is relatedwith the fact that the pointed nanostructural particles due to their sizes and pe-culiaritiesoftheiratomicstructurerevealsuchuniquephysico-mechanicalprop-erties that the range of their perspective applications spreads over many humanactivities,startingfrommicroelectronicstomedicine.

Inrecentyearsespeciallyincreasedinterestinsuchtechnologiesofmaterialsproduction on carbon basis as ones oriented on production of doped carbon na-noparticlemodifications(nanotubes,nanoclusters,nanothreads).Thisgivesop-portunity to scientists and engineers in noted materials the possibility of aimedcontroloftheirnaturalproperties[1].

Asthematteroffact,thenanoobjectcontrolonnanometriclevelusingnano-particleswithaimtoarrangetheminrows,signaturesandgridsisthecluetotheproductionofnewfunctionalmaterials.Correspondingly,forobtainingofbuild-ing units with different nanometric sizes in the last years many methods ofself-assemblingandsynthesisweredeveloped.Inthisdirectionthepossibilitytocontrol perfectly the self-assembling and synthesis processes of nanoparticles is aseriouschallengefromthepointofviewbothfundamentalandappliedinvestiga-tions.

On the basis of fundamental principles, the process of self-assembling requirestheexistenceofinteractionbetweenatomsandclusters,suchasthermodynamicandkineticmovingforces,sothatitcouldberealizedtheorganizationofatomsandclustersforcreationofnanosizedomenstructures.Fromthispointofview,magnetic nanoparticles deserve a particular interest due to their unique physi-co-chemical properties and applicability in the new functional materials tech-nologies.

Carbon shells provide both the protection of ferromagnetic impurities fromaggressive environment and new unique properties to the hydride nanostruc-tures.Theself-assemblingofmagneticclusterscoatedbycarbonshellspresentsjust such example which could be used in the

contemporary materials, for ex-ample, in strong magnets, analytic instruments (nuclear magnetic resonancetomographs)andnanosensors.
Themoreso,currentlymagneticcarbonnanoparticlesduetotheirlowtoxici-tyareundertestingfortherapeuticanddiagnosticapplications.
Lastyearsthemagneticfieldwasappliedalsoforcreationofnanoscalemate-rials what resulted in significant achievements in the problem of formation ofmacro-andmicrostructuresynthesizedmaterialspossessinguniqueproperties.
In contrast with other existing self-assembling technologies, the induced bymagnetic field ordering defines the formation of magnetic nanoparticles in or-dered structures with unique properties. Therefore, the areas of application ofcarbonmagneticnanoparticlesareverylarge.Sufficienttonamesuchofthemasmagneticfluids,plasticglassesstableagainstscretches,informationstoragemag-netic media, sensors, biomedicine, etc. It should be noted here that in spite oftheirlargeperspectiveformultifunctionalapplications,thecarbonnanoparticlesdopedbyferromagneticclusters havenotstudiedsufficientlywell[2].

Inthisworkfollowingaimsareaddressed:
1) Onthebasesofobtainednanopowdersthepreparationofpolymercompo-sitefilmsandstudyofnanoparticlediffusionandself-healingprocessesinthemundercombinedinfluenceofheating,pressure,steadyandalternatingmagneticfields.
2) The study self-organization processes in magnetic polymer composite un-dertheinfluenceofaoutersteadymagneticfieldandheating.

II. EXPERIMENTAL RESULTS AND THEIR DISCUSSIONS

To achieve these aims experiments were carried out to study self-healing andself-organization processes in magnetic polymer composites filled with magneticcarbonnanopowderssynthesizedbyCVDmethod[3].Theseprocesseswerefa-cilitatedbyheatingfilmsabovetheglasstransitiontemperature.Particlesincon-ventionalcompositesareessentiallyimmobileincontrasttopolymernanocom-posites(PNC)particularlyabovetheglasstransitiontemperatureT_g.Thenano-particles mobility can affect polymer dynamics resulting in changing of viscosity,modulus,kineticsoftheparticle-clusterformation,etc.[4].
Tensile measurements have shown that below $T_g$conventional composites andPNCsbehavesimilarlywithrespecttomechanicalproperties.Butabove$T_g$thetoughnessofPNCcanincreasebytheorderofmagnitudewithincreaseoftem-perature. It has been suggested that the mechanism to the toughness enhance-mentisthemobilityofthenanoparticles.Thedevelopmentofself-healingmate-rials and coatings where nanoparticles migrate toward various defect sites, re-quiresabetterunderstandingofthenanoparticlediffusionprocesses.
The heating of PNC above $T_g$increases mobility of polymer chains what shouldfacilitateboundarydiffusionbetweenpolymerinterfacesandthiseffectcouldbevisualized using magnetic nanoparticles introduced in polymer. This process couldbeimprovedapplyingadditionalstimuli,inparticularalow-frequency(ac)mag-netic field, stationary magnetic field, pressure, heating, separately or in combi-nations. Such impact stimulate self-organization processes between prepared filmsin result of which one could produce “glued” to each other films without usingothertypegluesandpolymermeltingtemperature.Asresearchobjectforoneofthe aims of this work was the development of simple technology of productionofcarbonnanoparticlesdopedbyferromagneticclustersandstudyoftheirmor-phologyandcomposition.
Generally the most known method of production of ultra-small cluster-sizeparticles is based on the condensation principle from gas or vapor states, withthe possibility of obtaining the product on a substrate or as a powder. In thesametime,forthevaporproductiondifferentmethodswereused:laserablation,thermal,electron-beamandionirradiation.
In particular, for the production of nanopowders and nano-coatings on car-bonbasisitisusedthechemicalvapordeposition(CVD)method—mainlyalongwithapplicationofhydrogenreductionprocessofvolatilechlorides.Traditional

methods of production the pyrolytic carbon by the way of thermal decomposi-tionofcarboncontainingcompoundsontheheatedsurfacesarealsowellknownbut all of them have some significant defects: at formation of coatings—lowgrowth rate, high temperature of process (≥1300˚C), insufficient quality of ob-tained coating structures, impossibility to carry out the doping process in onecycle. The analysis of tendencies of development of the contemporary naturalscienceandtechnologytheexperimentalandtheoreticalinvestigationsofcarbonnanoparticles,nanotubes,nanothreadsanddopedbythemnewpolymercompo-sitesshouldgainpriority.Thecarbonnanoparticlesdopedbymagnetic(Co,Fe)nanoclasters with mean sizes in the range 50 - 100 nm were synthesized bytechnology using the combination of pyrolysis of ethylene spirit (and other hy-drocarbons) vapor pyrolysis and CVD process in the mode of recirculation closedcyclewithmonitoredtechnologyparameters[3].

Thedevelopedtechnologicalprocesswasrealizedintheinstallationwhereinthe construction of its reactor and in necessary units for functioning the possi-bility was foreseen of monitoring such parameters as: vapor content and con-centration in reactor zones, catalytic capacity of substrates, partial oxygen pres-sure. This allows one to carry out technology investigations with aim to establishthe optimal technological parameters for production of finely dispersed carbonnanopowders doped with magnetic nanoclusters and, as result, to produce thesematerialsinnecessaryamountsforinvestigationsandperspectiveapplications.

For the preparation of polymer films it was chosen polyvinylbutyral (PVB)polymer with low $T_g \sim 45^\circ C - 55^\circ C$. PVB is a resin mostly used for applicationsthat require strong binding, optical clarity, adhesion to many surfaces, toughnessandflexibility.

AsfillertheproducedbyuscarbonnanopowderdopedwithmagneticCona-noclusters(C/Co)wasused.Foracomparativestudyweusedalsothecommer-cial Co nanopowder (Sun firm, USA 28 nm—mean diameter). The Co dopedcarbonnanopowderswereмainlyusedattheirconcentrationsinpolymericcom-posite in range 10 - 50 wt%, but for Co nanopowder filled polymer compositeconcentrationwas20wt%.

Firstlythe10%alcoholsolutionwasprepared,thenthissolutionwaspouredintoteflonpress-forms(**Figure1**)andaftertheirdryingduring48hoursthe0.3 - 0.5mmthicknessfilmswereobtained.Thefilledcompositeswerepreparedinthefollowingway:magneticnanopowdersweretakenincorrespondingpropor-tion (in terms of dry weight) and the usual mixing of PVB with alcohol tookplace followed by the ultrasound 10 - 15 min treatment for the destruction ofmagnetic nanopowder agglomerates. Next, after thorough mixing during 7 - 10min magnetic polymer composite films were obtained similar unfilled ones inteflonpress-forms.Thensamplefilmswerecutfromthesefilms(**Figure2**).

Thetrialfilmswereplacesoneachotherandsubjectedtofollowingactions:

1) Impactofpressureandtemperatureinavacuumcabinetatpressurep=0.5MPaandtemperatureT=80˚C-85˚Cduringtimet=2.5hours.

Figure1. Teflonpress-formsforpreparationdifferentsizefilms.

(a) (b) (c)

Figure2. Filled(a)andunfilled,(b)filmsseparatelyandjointly,(c)withfilledfilmaboveunfilledone.

2) Impact of pressure and temperature and stationary magnetic field in a va-cuum cabinet at p = 0.5 MPa and temperature T = 80˚C - 85˚C. During the ex-periment pressure included a contribution from the magnet weight. The dura-tionofexperimentwast=2.5hours.

3) In above a and b items, the obtained double films were subjected to the ex-citationbya low-frequencymagneticfield during t=2hours.

In **Figure 3**, the vacuum cabinet is presented in turn on position, with placedinsidesamplesduringthepressureandtemperatureaction.

During an experiment temperature reaches 85˚C, pressure was approximate-ly 0.5 MPa. In the teflon form first of all magnets were placed, then an unfilledfilm and, finally, polymer composite film on which pressure acts. Then thelow-frequencygeneratorturnsonwithcoolingofcoilbywaterandheatingfol-lowing which the combined actions is realized which could continue severalhourstillthedoublelayersampleisgluedbydiffusionofnanoparticles.

In **Figure 4**the experimental setup is mounted accordingly the experimentalschemeispresented.

Figure3. Thevacuumcabinetwithsamplesunderthetesting.

Figure4. Theexperimentalsetup.

In **Figure 5**, neodymium magnets used in experiments are seen, as well as in**Figure6**thepressformforfabricationofsamplesisshown.

To study the self-organization of polymer nanocomposites it is necessary toincreaseourunderstandingofnanoparticlesdiffusionprocessingtakingplaceinthem[1].

Asitwasnoted,nanoparticlesinusualcomposites,indifferencewithpolymernanocomposites (PNC) are essentially immoble is particular as compared withdiffusionprocessesrateinPNCabovethetemperatureofglasstransitionT_g.

Besidesit,theknowledgeofnanoparticlesdiffusionprocessesisimportantforthedevelopmentofself-healingmaterialsandcoatingswherenanoparticlesmi-gratetodifferentdefectsites.

Forthestudyofcobaltdopedcarbonnanopowderdiffusionprocessesaseriesofabovedescribedpolymersampleswe reprepared.Asampleisa0.3mmthick-ness polymer nanocomposite film (polymer filled with C/Co nanoclasters, thenanoparticle composition was in range of 10 - 50 wt%) and 0.5 mm thick purepolymersubstratewithtotalpiledimensions28×10×0.8(**Figure7**).

ThediffusionprocessesofC/Conanoparticlesacrossinterfacebetweenlayers at different stimuli at $T > T_g$: stationary magnetic field, alternating magnetic field(20kHz,1kWpower),andtheircombinations,theadhesionand,consequently,relatedwiththeadhesionself-healingprocesseswerestudied.

Figure5. Neodymiummagnetsusedinexperiments.

Figure6. Pressformforsamplesfabrication.

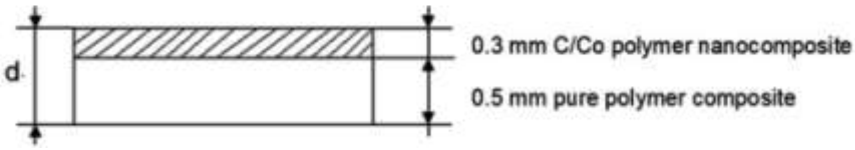

Figure7. Experimentalpolymerpile.

To study these processes the layer by layer removal was carried out startingfrom pure polymer layer down to composite one using a fine grinding. Thisgrindingprocesswasfollowedbythesimultaneouscontrolofsampleresistance.

The method appeared to be very visual and effective and allowed one the as-sessment of nanoparticle mobilities at different excitations and their combina-tions. It was revealed that the best diffusion of nanoparticles and adhesion ofcomposite film was obtained at the combined effect of temperature, steady andalternatingmagneticfields.

In **Figures 8-13**, the distribution of resistance along a sample is presented atfine grinding describing the degree of penetration of nanoparticles in the poly-mersubstrate.

The good conductivity of polymer composite was stipulated by the presence inthecompositeofthecobaltcarbonnanoparticlesatstudiedC/Cowt.50%poly-mernanocomposite/purepolymerpile.

Duringtreatmentofsampleshownin**Figure11**,thepeelingofuppercompo-sitelayerwasobservedwhatwasnotobservedatothercases.

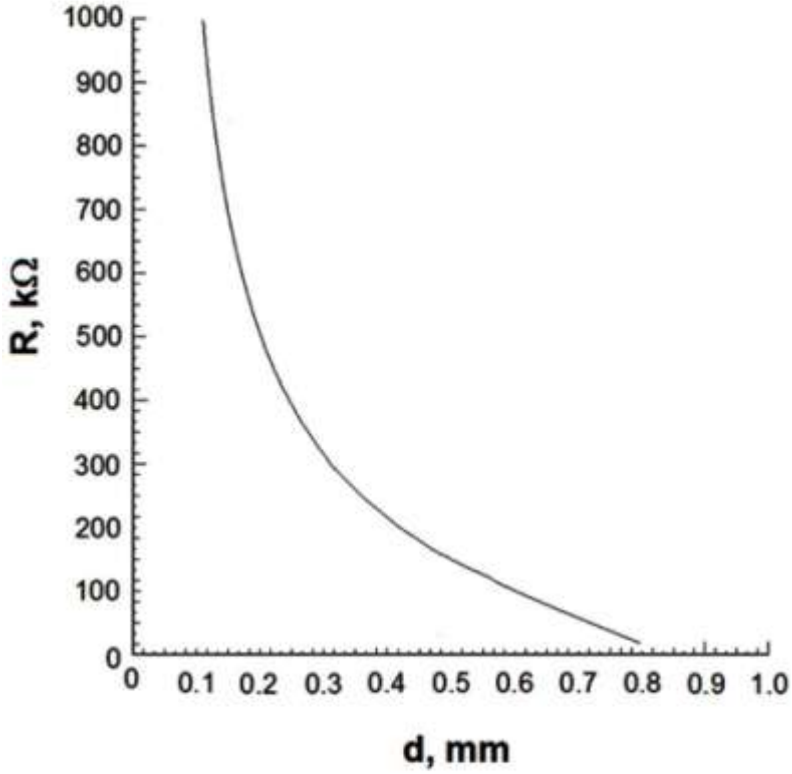

Figure8. Theresistancedependenceonthesamplethickness.Thetreatmenttookplaceatapplicationofsteadyandal ternatingmagneticfieldsatT=85˚C.

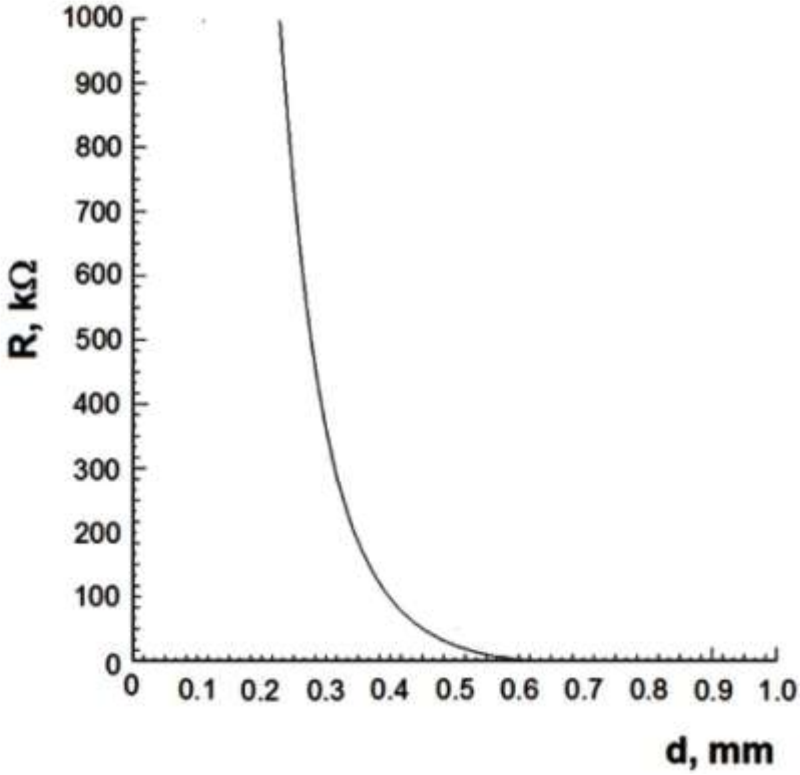

Figure9. The resistance dependence on the sample thickness. The treatment was madebysteadymagneticfieldat85˚C.

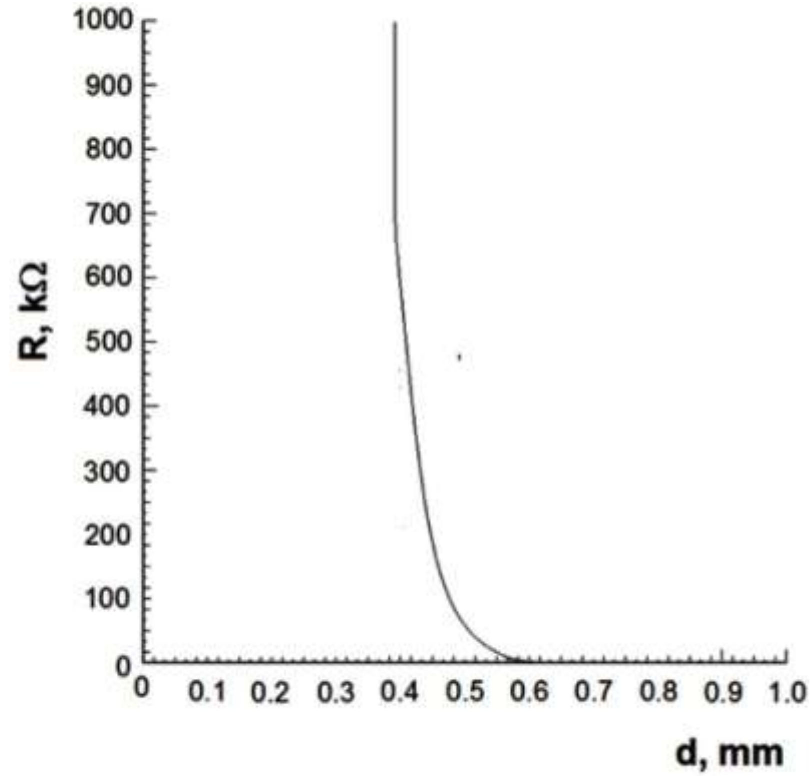

Figure10. Theresistancedependenceonsamplethickness.Thetreatmentiswithoutmagneticfieldat85˚C.

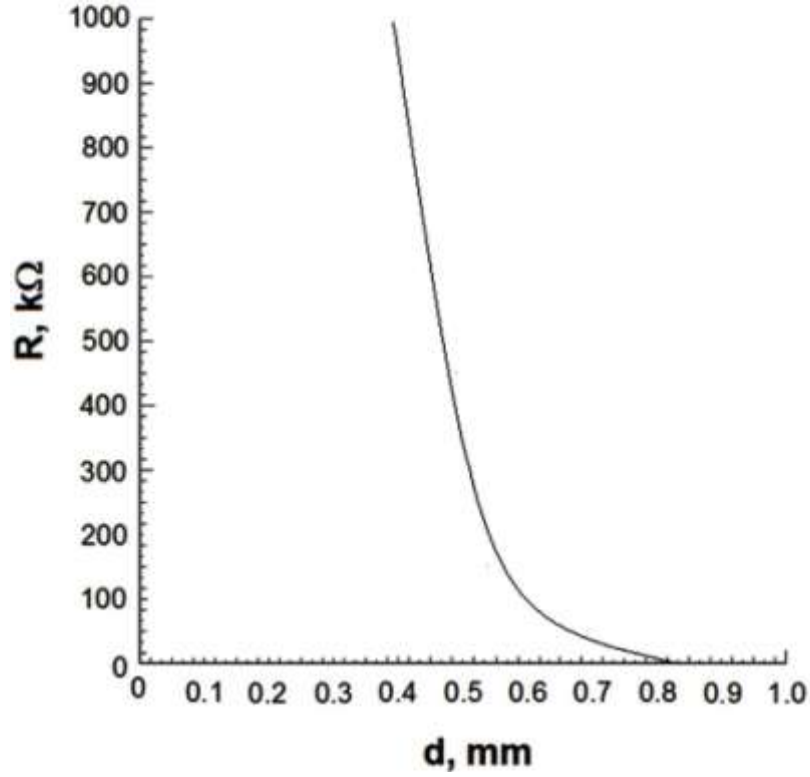

Figure11. Theresistancedependenceonsamplethickness.TreatmentisbyalternatingmagneticfieldsatT=85˚C.

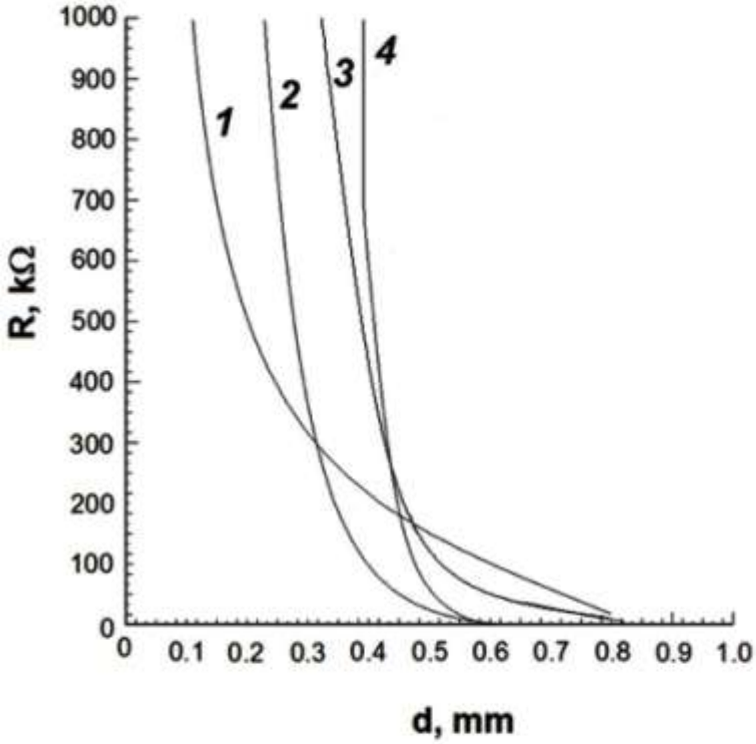

Figure 12. The resistance dependence on sample thickness (summary): Curve 1 corres-ponds to one given in **Figure 8**; Curve 2 corresponds to one given in **Figure 9**; Curve 3correspondstoonegiveninFigure10;Curve4correspondstoonegiveninFigure11.

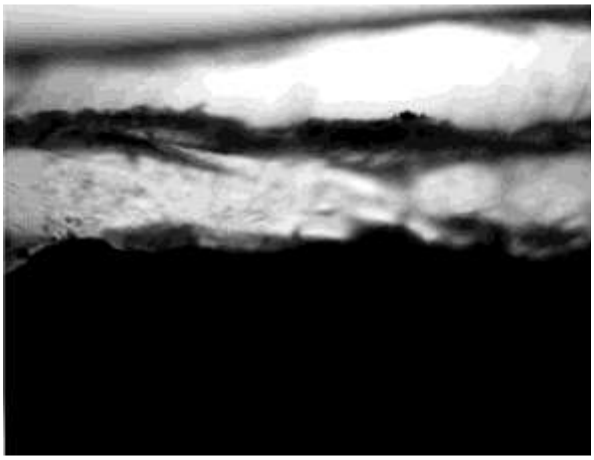

Figure 13. Treatment of C/Co wt. 50% sample at steady magnetic field and at T = 85˚Cduring2.5hours.

Note also that in a similar experiment when as a filler was used commercialConanoparticles(22nmismeandiameter)passivatedbyoleicacidthecompo-sitesampleprovedtobenon-conductiveduetonon-conductivepolymercoatingofConanoparticlesandabsenceofconductivityofpurepolymer.
For the control of nanoparticle diffusion in both polymer composites (withC/Co and Co nanoparticless) we used optical microscope OMAX with up to2500enlargement.

For these optical measurements a small plate was cut from a sample with thewidth 1 mm and taken pictures of its transverse cutting during any type of in-fluenceortheircombinations.
Fordifferentcasespicturesaregivenin**Figures13-15**.
The results obtained by this method are in agreement with resistance mea-surementsones.
In result of carried out experiments the conclusion was made that the stimu-lateddiffusionofnanoparticlesismostfastwhentemperatureandbothmagnet-icfield(steadyandalternating)actedincombination.
Tostudyfurtherself-assemblyprocessesinthispolymercompositesatdiffer-ent concentrations of carbon magnetic nanopowder we used a simple methodfromwork[5].Inthiscasecircularshapepolymercompositesamples(diameter20 - 32 mm, thickness 1mm) were exposed to a magnetic field which was pro-videdbytwoattachedpermanentneodymiummagnetsandheating(**Figure16**),andtemperature85˚Cduringtwohours.
Resulting self-assembly of C/Co nanopowders caused changing of their con-centrationandmodulationoflocalresistanceRalongtheradiusofsamplewhichwasmeasuredbytwo-contactmethodasinwork[5](**Figures17-22**).

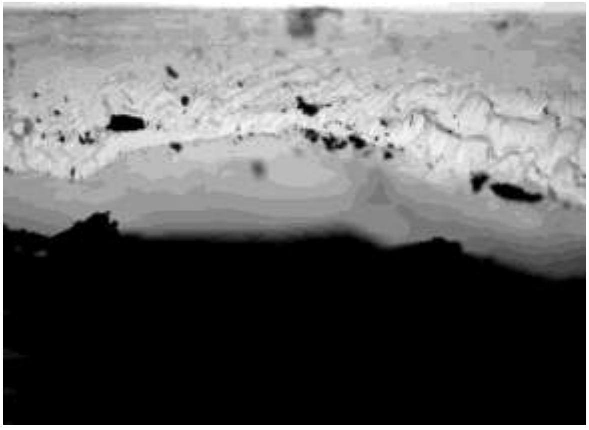

Figure14. TreatmentofC/Cowt.50%samplewithoutmagnetatT=85˚Cduring2.5hours.

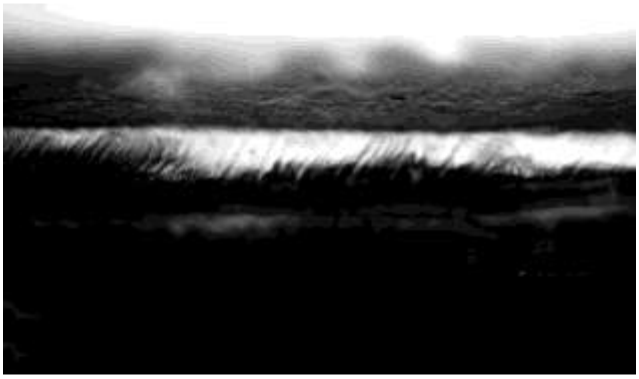

Figure15. TreatmentofCo/Cwt.50%sample bybothtypemagneticfieldsattempera-tureT=85˚Cduring2.5hours.

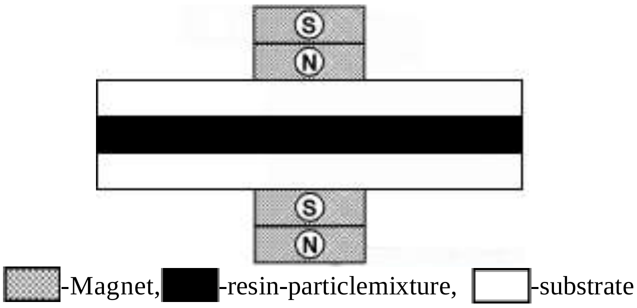

Figure16. Geometryofsamples.

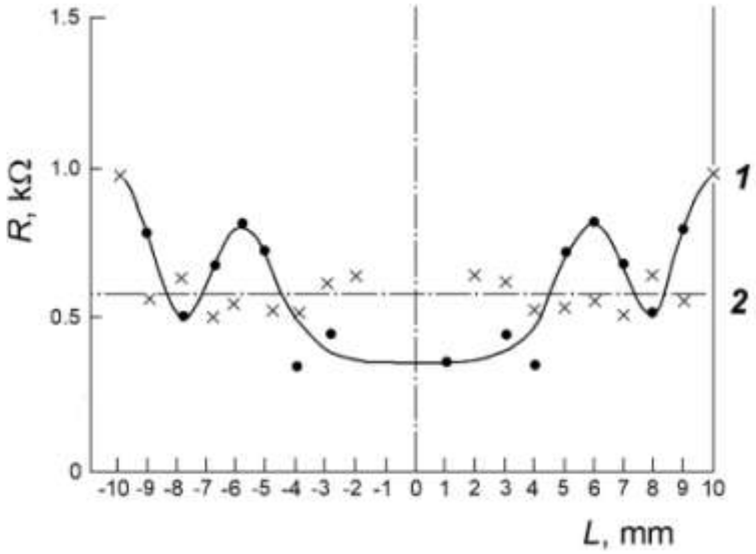

Figure17. PolymercompositeC/Co,wt.30%;1:resistantdistributioninthesampleasresultoftreatment;2:resistant inthesamplebeforethetreatment.

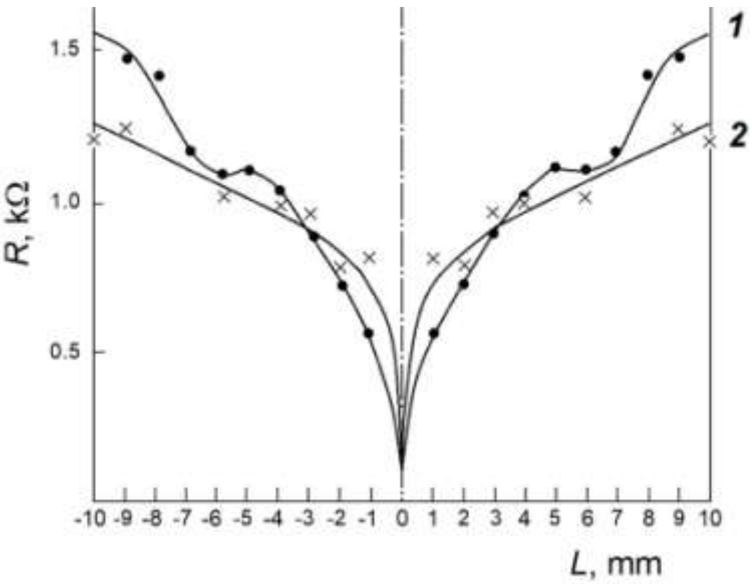

Figure18. PolymercompositeC/Co,wt.30%;1,2:treatedanduntreatedsamples,cor-respondingly.

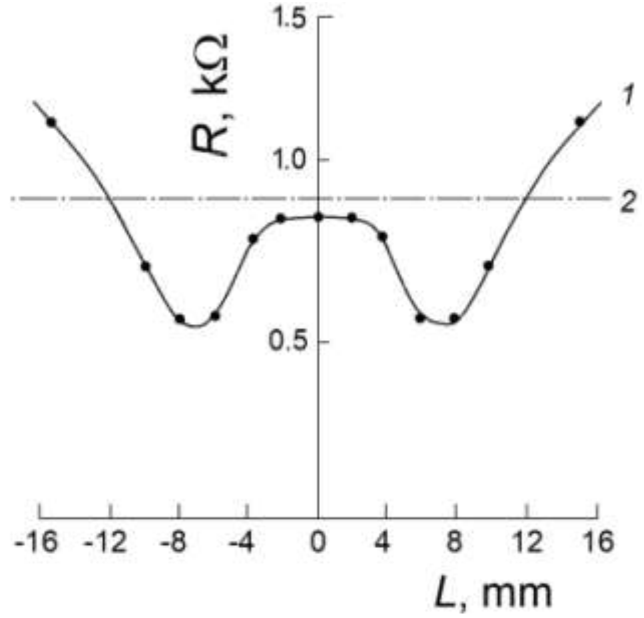

Figure19. PolymercompositeC/Co,wt.20%;1,2:treatedanduntreatedsamples.

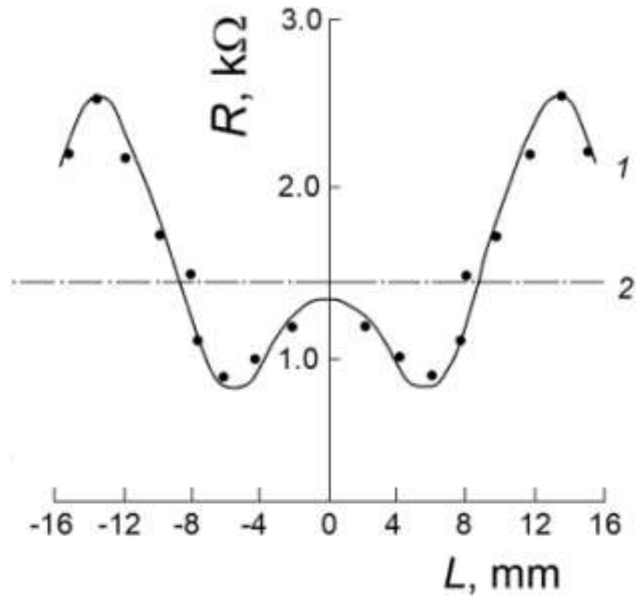

Figure20. PolymercompositeC/Co,wt.15%;1,2:treatedanduntreatedsamples.

Resistancewasmeasuredbetweenpointsseparatedby2mmalongradiusinall following cases besides **Figure18**where resistance was measured betweensample's center andpoints on radiuswiththe interval 2mm.
Based on the obtained experimental results, we were able to investigate theself-assemblingprocessesinthemagneticpolymernanocompositefilmssynthe-sized by the developed technology using the carbon magnetic nanopowdersfa-bricated by the method of [4] under the combined influence of magnetic fieldand heating using sensitive and simple non-contact RF resonant magnetometrymethod[5].

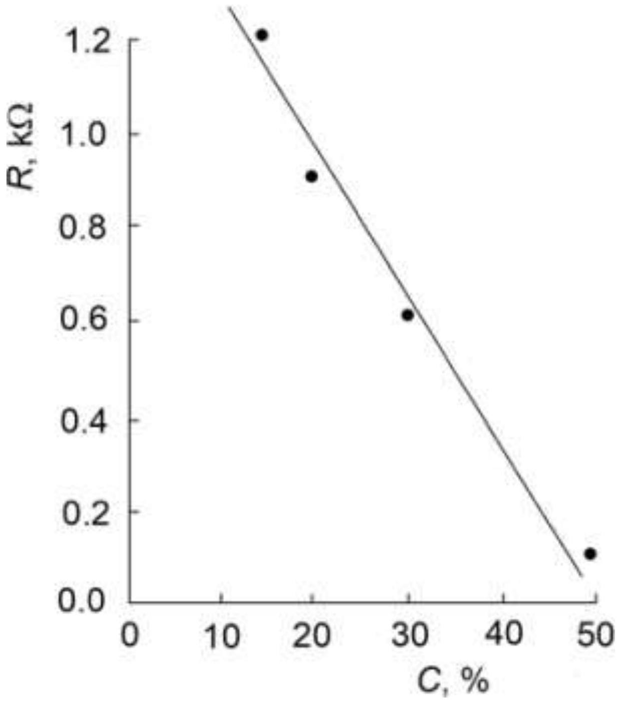

Figure 21. The dependence of resistance of initial untreated samples on the nanopowderconcentrationC.

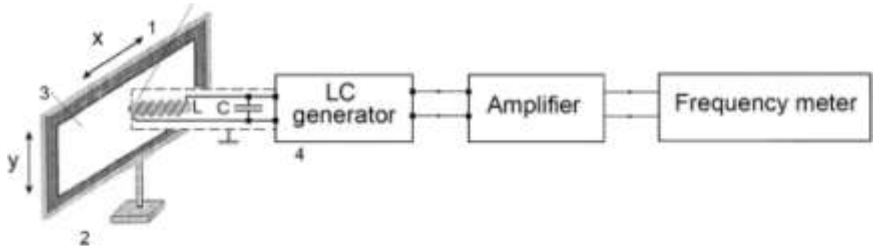

Figure 22. The setup for study of the self-assembling processes in the polymer magneticnanocopositefilms.1:ferriteprobe;2:frame;3:polymericmagneticnanocompositefilm;4:LCgenerator.

IntheinductivecoiloftheRFresonantcontourofLC-generatoracylindricaltippedferriterodisusedasaprobe.Theinvestigateddiskshapemagneticpoly-mernanocompositefilmisdisplacedrelativelytheimmovableferriteprobetip.The scanning of the film surface is realized along the previously marked diskdiameter.

During the scanning of magnetic polymer composite film by moving ferriteprobe the variation of RF resonant magnetometer frequency is observed due tothe change of the cobalt nanocluster concentration in the polymer compositefilmandrelatedwithitchangeofthecoilinductance.

Forexample,wepresenttheresultsofself-assemblyprocessesmeasurementsofcarboncobaltnanoclustersinpolymernanocompositefilmundertheinfluence ofthemagneticfieldofneodymiummagnetwithdiameterD=12mmandheat-ing(**Figure23**and**Figure24**),whicharesimilartotheresultsobtainedduring

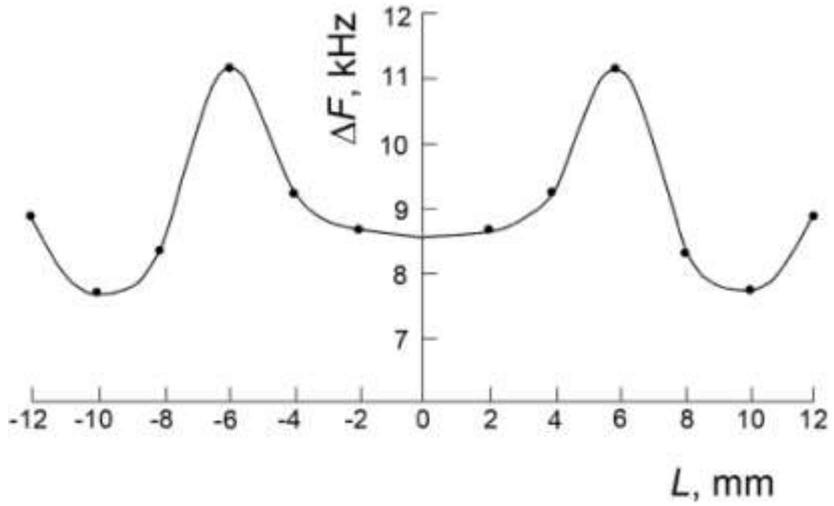

Figure 23. Dependence of the RF resonant magnetometer oscillation frequency at dis-placement of ferrite probe along the diameter of C/Co, wt. 50% carbon cobalt polymernanocompositediskfilm,D=24mm.

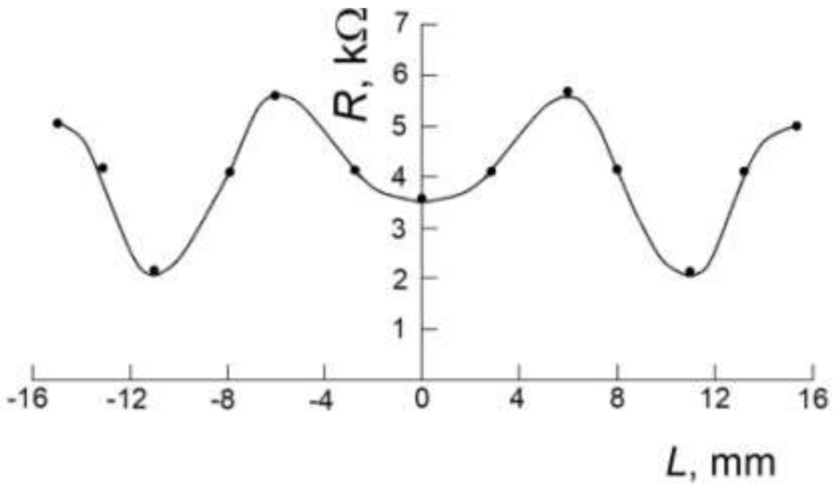

Figure 24. Dependence of the RF resonant magnetometer oscillation frequency at dis-placement of ferrite probe along the diameter of C/Co, wt. 20% carbon cobalt polymernanocompositediskfilm,D=30mm.

the study of self-assembly processes by the two-contact electrical resistance mea-surementsofthesamesamples.
Inwork[6]thepossibilitytorepairphysicallyseparatedphermoplasticpoly-methacrylatescontainingsuperparamagneticnanoparticlesusingoscillatingmag-netic field (OMF) was shown without any chemical intervention while main-taining film mechanical properties and the repair can be repeated several timesonthesamearea.
Thefirststepinthisdevelopmentwastopreparepolymerfilmswithuniformlydispersed magnetic nanoparticles. This uniformly dispersed superparamagneticnanoparticles dispersed in the polymer matrix provides an opportunity for de-signingself-healingmaterialswithamagneticsignature.Itwasshown [6] that

whenOMFisapplied,thefilmsrestoretheirmechanicalproperties.Thiswasat-tributed to the oscillating magnetic γ-Fe_2O_3nanopartices, thus resulting in anamorphous flow in the damaged region and repair. It should be also noted thatthe films retain their dimensional stability. As OMF excites the magnetic mo-ment in magnetic nanoparticles, energy resulting from the Neel and Brownianrelaxationisconvertedtothermalenergy.

Accordinglywork[7]applicationofstationarymagneticfieldonpolymercom-posite containing mixture of carbon black and magnetite nanoparticles resultedin diffusion of oxide particles toward the magnetic field lines and their alignment.The magnetic stimulated iron oxide particles put pressure on the nonmagneticcarbonblackparticlesenhancingparticleconnectionleadingtodecreasedresis-tivity.

Similarthis,thepressureandtemperaturealsocontributetotheincreaseddif-fusionofmagneticnanoparticlesindirectiontodefectareas.Resultsofcitedworkscould be used to understand effects observed in this work. As a result of com-binedstimulateddiffusionofC/Conanoparticlespreparedfilmsshowgoodselfhealingandadhesionpropertiesaswellasgoodelectricconductivityduetothepassivated conducting carbon coated magnetic Co nanoclasters. These effects weremostintensiveinthecaseofcombinedapplicationofalternatingandstationarymagneticfieldsattemperatures abovethetemperatureofglasstransition.

III. CONCLUSION

The self-healing and self-organization processes were studied in the magneticpolymernanocompositessynthesizedonbasisofcarbonnanoparticlesdopedbycobalt nanoclasters, synthesized by original CVD technology developed by au-thors.Theseprocessesweretakingplaceundercombinedstimulateddiffusionofmagnetic nanoparticles by outer alternative and steady magnetic fields, as well asheating and pressure. Polymeric composite samples have good electric and ad-hesivepropertiesandareperspectiveforpotentialpracticalapplications.

ConflictsofInterest

Theauthorsdeclarenoconflictsofinterestregardingthepublicationofthispa-per.

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