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Vibrational Spectra and Structural Features of Polymer Composites with Carbon Nanomaterials as Fillers

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Polymer composites containing carbon nanomaterials have attracted considerable attention of researchers due to their unique physicochemical properties and a wide range of applications. In particular, composite materials based on epoxy resin and polyethylene doped with carbon nanoparticles demonstrate improved mechanical, electrical, and optical characteristics, making them promising for use in the aviation, electronics, automotive, and biomedical industries. One of the key aspects of the investigation of such composites is the study of their structural features and interactions between the polymer matrix and the filler. An important tool for this is a set of spectroscopic-analysis methods, in particular, vibrational spectroscopy, which allows obtaining information about the molecular structure and interfacial interactions in materials. The study of Raman scattering (RS) of light and infrared (IR) absorption spectra allows us to assess changes in intramolecular dynamics and the nature of bonds in the polymer matrix, taking into account the influence of carbon nanofillers. This article examines the features of the vibrational spectra of epoxy and polyethylene composites with carbon nanomaterials, analyses their effect on the polymer-matrix structure, and discusses possible mechanisms of interaction between the composite components.

Полімерні композити, що містять вуглецеві наноматеріяли, привернули значну увагу дослідників завдяки своїм унікальним фізико-хемічним властивостям і широкому спектру застосувань. Зокрема, композитні матеріяли на основі епоксидної смоли та поліетилену, леґованих вуглецевими наночастинками, демонструють поліпшені механічні, електричні й оптичні характеристики, що робить їх перспективни-

ми для використання в авіаційній, електронній, автомобільній і біомедичній промисловостях. Одним з ключових аспектів дослідження таких композитів є вивчення їхніх структурних особливостей і взаємодії між полімерною матрицею та наповнювачем. Важливим інструментом для цього є спектроскопічні методи аналізи, зокрема коливна спектроскопія, яка дає змогу одержувати інформацію про молекулярну структуру та міжфазні взаємодії в матеріялах. Вивчення спектрів комбінаційного розсіяння світла (РС) та інфрачервоного (ІЧ) вбирання уможливлює оцінити зміни внутрішньомолекулярної динаміки та характер зв'язків у полімерній матриці з урахуванням впливу вуглецевих нанонаповнювачів. У цій статті розглядаються особливості коливних спектрів епоксидних і поліетиленових композитів з вуглецевими наноматеріялами, аналізується вплив їх на структуру полімерної матриці й обговорюються можливі механізми взаємодії між компонентами композиту.

Key words: polymer nanocomposites, carbon nanotubes, graphene nanoplatelets, FTIR spectroscopy, Raman spectroscopy.

Ключові слова: полімерні нанокомпозити, вуглецеві нанотрубки, графенові нанопластинки, ІЧ-спектроскопія на основі Фур'є-перетвору, Раманова спектроскопія.

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1. INTRODUCTION

Polymer composites containing carbon nanomaterials have attracted considerable attention from researchers due to their unique physicochemical properties and a wide range of applications. In particular, composite materials based on epoxy resin and polyethylene doped with carbon nanoparticles demonstrate improved mechanical, electrical and optical characteristics, which makes them promising for use in the aviation, electronics, automotive and biomedical industries [1]. Such materials are characterized by high thermal stability, improved wear resistance and increased conductivity, which is especially important for applications in sensor devices, flexible electronics and composite materials [2–4].

One of the key aspects of the study of such composites is the study of their structural features and interfacial behaviour within the polymer matrix in the presence of nanofillers. An important tool for this is spectroscopic analysis methods, in particular vibrational spectroscopy, which allows obtaining information about the molecular structure and interfacial interactions in materials. The study of Raman scattering (RS) of light and infrared (IR) absorption spectra allows assessing changes in intramolecular dynamics and the nature of bonds in the polymer matrix under the influence of carbon nanofillers. For example, the introduction of multiwalled

carbon nanotubes (MWCNTs) or graphene nanoplatelets (GNP) can cause a shift in the characteristic bands in the Raman and IR spectra, which indicates the appearance of supplementary intermolecular interactions or a change in the polymer chain conformations.

Incorporating carbon nanomaterials into epoxy composites can encourage the formation of interconnected bonds within the polymer matrix, which improves its mechanical strength and thermal stability. In addition, such composites often exhibit a change in spectral characteristics in the range of 1500–1700 cm^{-1}, which is associated with the modification of the C=O and C–N bonds. In polyethylene composites, the addition of nanofillers can affect the crystallinity of the polymer, which is manifested in a change in the intensity of the bands in the range of 2800–3000 cm^{-1}, associated with stretching vibrations of CH_2 groups.

This article examines the features of the vibrational spectra of epoxy and polyethylene composites with carbon nanomaterials, analyses their influence on the structure of the polymer matrix, and considers various possible mechanisms of interaction between the composite components. In particular, the effects of different types of fillers (single- and multiwalled carbon nanotubes, graphene sheets, and nanodispersed forms of carbon) on the spectral characteristics of the materials are considered. The prospects for using these methods to optimize the properties of polymer composites and develop new functional materials for advanced technologies are also discussed.

2. MATERIALS AND METHODS

The preparation of polymer nanocomposites with carbon-based fillers was performed through a multistep procedure, adapted according to the type of polymer matrix, namely, either high-density polyethylene (PE) or epoxy resin (L285 with hardener H286).

Preparation of Nanofillers. Multiwalled carbon nanotubes (MWCNT) and graphene nanoplatelets (GNP) were initially dried and then ultrasonically dispersed in an appropriate solvent (*e.g.*, ethanol or acetone) to ensure deagglomeration. For improved dispersion, surfactants (such as Triton X-100 or SDS) could be used.

Polymer-Matrix Preparation. For PE-based composites, PE granules were melt-blended with dispersed nanofillers using a twin-screw extruder at $\cong$ 130–160°C, followed by hot pressing into sheets. PE globules and dispersed nanofillers were mixed in the IKA ULTRA TURRAX Tube Drive homogenizer test tube with a stirrer, with 3000 rpm as the rotation speed. Then, the mixture was hot-pressed in an enclosed hot die heated to 160°C, after which it was cooled down until reaching room temperature. For epoxy-based composites,

Nanofillers were first dispersed in the epoxy resin via ultrasonic agitation (30–60 min) and then incorporated with the stoichiometric amount of hardener. The mixture was vacuum-degassed and cast into moulds. Polymerization was carried out at room temperature for 24 h, followed by post-curing at a temperature that gradually increased from 40 to 80°C for 5 h.

Composite Shaping and Suring. Shaped samples were formed by compression moulding (PE) or casting (epoxy). Final samples were polished for further characterization.

Characterization. Prepared samples were subjected to Raman and FTIR spectroscopy to analyse structural and vibrational changes, as well as SEM for morphology. FTIR spectra were obtained using Shimadzu IRTracer-100. The Raman spectra were recorded at room temperature in a quasi-backscattering configuration using the Horiba Jobin-Yvon T64000 triple spectrometer integrated with Olympus BX-41 microscope equipped with a motorized *XYZ* stage and Peltier-cooled CCD detector. Micro-Raman spectroscopy is a very useful technique allowing non-destructive studies of the structure and electronic properties. Ar^+ laser (488 nm, 100 mW) was used to excite the samples.

3. RESULTS AND DISCUSSION

Below, we will consider the features of the vibrational spectra of polymer matrices, carbon nanofillers and the created polymer nanocomposites.

3.1 Polymer Matrixes

Epoxy resins are among the most important thermosetting polymers [5–7]. These polymers have excellent properties such as resistance to heat, moisture, and chemicals, strength, electrical, and mechanical stability, and good adhesion to many substrates [8].

The vibrational spectra of epoxy resin and polyethylene are well studied [9, 10]. The following characteristic bands of the IR absorptions spectra of epoxy resin: (1) a broad band in the range of 3200–3600 cm^{-1} (IR) corresponds to the stretching vibrations of hydroxyl groups; the bands in the range of 2800–3100 cm^{-1} (IR and RS) correspond to the stretching of C–H vibrations; (3) the bands at 918, 1200–1250 cm^{-1} (IR) and 1675 cm^{-1} (IR) are related to the vibrations of the epoxy ring (C–O–C groups) and aldehyde groups, respectively; (4) the bands at $\cong$ 1600 cm^{-1} correspond to the stretching vibrations of aromatic C=C bonds (aromatic rings). Intense Raman bands at $\cong$ 800–1200 cm^{-1} [10] are characteristic of aromatic groups

and epoxy ring: vibrations of epoxy group are in the range from 1230 cm^{-1} to 1280 cm^{-1} according to [11], and the breathing mode of epoxy ring is at 1252 cm^{-1} [11]. The intensity of this peak depends linearly on the concentration of epoxy groups in the resin [12].

The IR and Raman spectra of polyethylene [13, 14] exhibit vibrational modes corresponding to the listed below functional groups: (1) strong bands at 2800–2900 cm^{-1} (IR) correspond to the stretching C–H vibrations; (2) deformation vibrations of the CH_2 group are recorded in the range of 1460–1475 cm^{-1} (IR and RS); (3) bands at $\cong$ 720 cm^{-1} (IR) are wagging vibrations of the CH_2 group. The spectra of polyethylene can display different intensities of the bands depending on the degree of crystallinity [15].

When analysing the vibrational spectra of nanocomposites, it is important to account for the features of the bonds and the chemical structure of the matrices in the initial state.

The initial epoxy resin contains functional groups for bonding with nanofillers (for example, through hydroxyl or epoxy groups). High thermal and mechanical stability is attributed to the presence of aromatic rings in the polymer. Epoxy rings during polymerization can interact with fillers or modifiers, which change the spectral profile (decrease in the intensity of the C–O–C bands). Therefore, analysis of changes in the intensity of the epoxy ring bands allows us to assess the level of polymerization and interaction with the filler. At the same time, polyethylene is a chemically inert material with a simple structure, which provides low interaction with fillers in the initial state. The hydrophobic nature of polyethylene limits adhesion to most fillers without prior functionalization. Crystallinity may be elevated due to nanofiller incorporation, as indicated in the spectra by an increase in the intensity of the corresponding bands (for example, at 720 cm^{-1}). Analysis of the crystallinity degree and changes in the spectra after the introduction of nanofillers helps to predict the mechanical and thermal properties of the composite.

Figure 1 illustrates FTIR spectra of MoS_2 nanoparticles, GNP and MWCNTs dispersed in an epoxy matrix in the region associated with epoxy resin characteristic vibrational modes. The figure clearly shows the effect of introducing carbon nanofillers (MWCNT—line 2; GNP—line 3) on the epoxy ring, and this effect is enhanced with the additional introduction of molybdenum disulphide nanoparticles—the intensity of the bands around 1100 and 1250 cm^{-1}, which are characteristic of aromatic groups and epoxy ring, increases significantly, and a decrease in the full width at half maximum (FWHM) relative to peak intensity is noted (line 4). In addition, the different influence of nanofillers of different na-

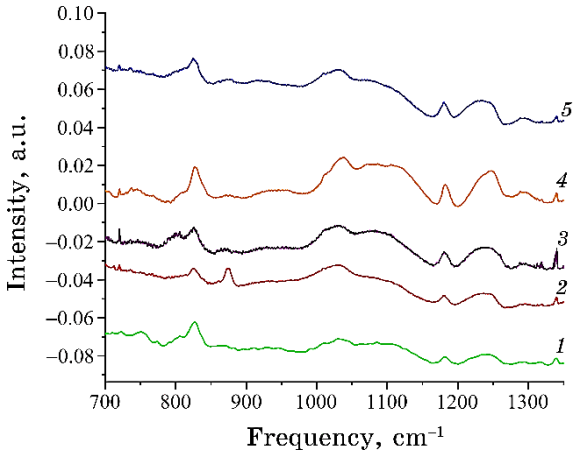

Fig. 1. FTIR spectra of MWCNTs (*2*), GNP (*3*), MoS_2 (*5*), and MWCNTs + MoS_2 (*4*) dispersed in epoxy polymer matrix L285 (*1*).

ture (nanotubes or graphene nanoflakes) on the redistribution of the intensities of complex bands around 850 cm^{-1} is clearly manifested.

3.2. Carbon Nanofillers

The Raman spectra of carbon-graphite materials are characterized by *G*- and *D*-bands, their shifts and intensity ratios [16, 17]. The *G*-band around ≅ 1580 cm^{-1} corresponds to the stretching vibrations of C=C bonds in graphite-like structures, its intensity and position can shift depending on the degree of defectivity and interaction with the matrix. The *D*-band (≅ 1350 cm^{-1}) is associated with vibrations caused by defects in the crystalline structure of graphene walls. An enhanced *D*-band intensity reflects a higher degree of structural defects, for example, due to functionalization or interaction with the matrix. The 2*D*-band (*G*′) around ≅ 2700 cm^{-1} is sensitive to the number of layers in the nanotube structure.

IR spectra are weakly expressed due to the low dipole activity of C=C bonds in MWNTs. Peaks of functional groups (*e.g.*, C=O, O–H) may appear in the spectra if the nanotubes are pre-functionalized [18].

The Raman spectrum of graphene nanosheets (GNPs) is also characterized by typical *D*- and *G*-bands (≅ 1350 cm^{-1} and ≅ 1580 cm^{-1}, respectively) [19]. The presence of a peak at ≅ 1620 cm^{-1} (*D*′) may indicate enhanced interlayer interaction or structural defects. The infrared vibrational spectra, as in the case of MWCNTs, also demonstrate low activity for C=C bonds, but upon functionaliza-

tion, the bands of C=O ($\cong 1720$ cm^{-1}) or O–H ($\cong 3200$–3600 cm^{-1}) vibrations appear.

It is important to note that when the GNP surface is functionalized (*e.g.*, with –COOH, –OH, or $-NH_2$ groups), the character of the vibrational spectrum changes: in the Raman spectrum, the intensity of the *D*-band increases due to the disruption of the graphene-like structure, and new bands corresponding to chemically active groups appear in the IR spectrum. Functionalized graphene nanoflakes demonstrate increased interaction with the matrix (*e.g.*, through hydrogen bonds or chemical adsorption).

3.3. The Influence of Filler Type on the Polymer Matrix

Figure 2 demonstrates the influence of functionalized carbon/carbon graphite fillers (multiwalled carbon nanotubes, graphene nanoflakes) on the vibrational spectra of the polyethylene matrix in the vicinity of the manifestation of wagging vibrations of the CH_2 group (A) and stretching C–H vibrations. The figure demonstrates that the incorporation of carbon nanofillers significantly influences the symmetric and antisymmetric wagging modes of CH_2 groups, as evidenced by a redistribution of their vibrational intensities and an alteration in the band profile and half-width of one of them (Fig. 2, *a*). Table contains data on the spectral positions of the bands.

Embedding MWCNTs into a polymer matrix (epoxy or polyethylene) significantly affects the vibrational spectra (IR and Raman), reflecting the chemical and physical interactions at the matrix–nanofiller interface.

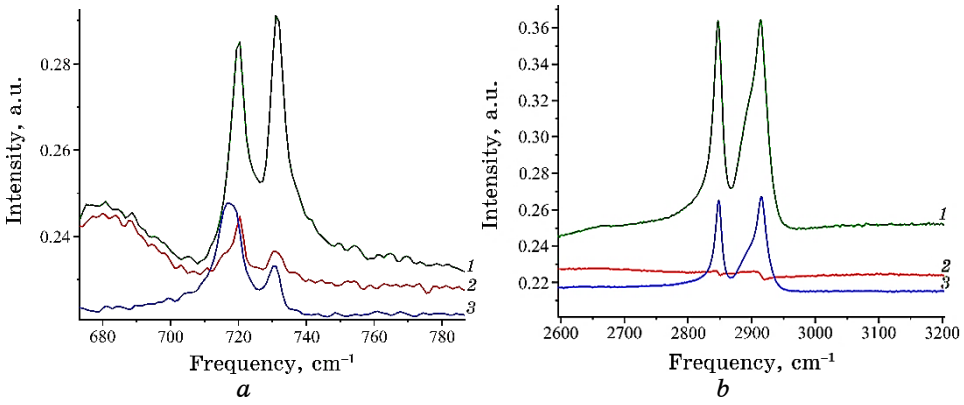

Fig. 2. The effect of MWCNT (*1*), GNP (*2*) on FTIR spectra of PE matrix (*3*) in different spectral regions (*a*, *b*).

TABLE. Effect of modified carbon nanoparticles on the PE matrix.

PE	NiFe		30% Fe_3O_4		Fe		30% MoS_2		Attribution
	CNT 3.4%	GNP 1%	CNT 1%	GNP 3.4%	CNT 5%	GNP 5%	CNT 5%	GNP 5%	
717.4	720.2	719.9	719.4		719.5	719	719.3	720	CH_2 scissors
730.5	731.0	731	730.9		730.7	730.3	730.7	730.5	
1462.7	at the noise level	1462.5	1462.2	at the noise level			1462.4	at the noise level	CH_2 band
1472.1	at the noise level	1472.8	1472.7	at the noise level			1472.8	at the noise level	
2848	2847.2		2848		2847.7	2846.7	2848.3	2846.3	CH stretching
2915	2913.4		2915		2915.5	2913.3	2915.2	2912.9	

Changes in vibrational frequencies in the infrared (IR) spectra (associated with functional groups of the matrix, for example, C–H, O–H, C–O in epoxy resin or C–H in polyethylene) may suggest the establishment of hydrogen bonding between the polymer matrix and the surface of functionalized carbon nanotubes [20], or indicate the presence of polar interactions or covalent linkages involving functional groups on the MWCNT surface [21]. The shift of the characteristic Raman *G*- ($\cong 1580$ cm^{-1}) or *D*-band ($\cong 1350$ cm^{-1}) of MWCNTs may indicate the transfer of stresses from the matrix to the nanotubes. The observed frequency shift can also result from interactions involving the π-electron system of the carbon nanotubes

and the polymer matrix.

The enhancement of some bands in the IR spectrum can be attributed to the formation of novel chemical bonds (*e.g.*, C–N, C–O–C). Variation in the intensity of the *G*- and *D*-bands in the Raman spectrum is often used to assess the degree of functionalization of the nanotube surfaces or to identify local deformation in the 'matrix–filler' zone. By analysing the ratio of intensities $I(D)$ to $I(G)$, one can assess defects on the surface of SWCNTs caused by processing or polymer matrix interaction.

Therefore, the use of both IR and Raman spectroscopy allows for the identification of bonding types, assessment of filler dispersion uniformity, and measurement of mechanical stress transfer efficiency between the matrix and carbon nanotubes.

The adding of carbon flakes (graphite or graphene) to a polymer matrix induces significant changes in vibrational spectra (IR and Raman), reflecting the formation of new bonds between components and structural changes within the matrix structure [22]. For example, shifts and changes in the intensity of IR bands associated with O–H, C=O and C–O groups may indicate the formation of hydrogen bonds [23, 24]. Covalent bonding between the matrix and the filler typically appears as new bands in the IR spectrum (*e.g.*, C–O–C, C–N) or as changes in the intensity ratio of the *D*- and *G*-bands in Raman spectra: the enhancement of the *D*-band (≈ 1350 cm^{-1}) reflects an increased number of defects on flakes' surface due to chemical functionalization or matrix interactions, and changes in the intensity of the *G*-band (≈ 1580 cm^{-1}) and its shift are indicators of stress transfer from the matrix to the flakes. The appeared additional Raman bands indicate interfacial interactions. In non-polar polymer matrices (*e.g.*, polyethylenes), weak intermolecular interactions (van der Waals and π–π stacking) are the main ones, which also affect the spectral bands (changes in the intensity of C–H vibrations). In polyethylene, the shift of the characteristic C–H bands in the range of 2800–3000 cm^{-1} can be caused by local stresses due to interaction with GNPs.

The obtained Raman spectra of MWCNTs introduced into the epoxy matrix are shown in Fig. 3. Further compositional complexity *via* MoS_2-nanoparticle incorporation causes a rise in the $I(D)/I(G)$ ratio and a shift of intensity maxima towards lower frequencies.

3. CONCLUSIONS

The incorporation of multiwalled carbon nanotubes (MWCNTs) and graphene nanoplatelets (GNPs) modifies the molecular organization of the polymer matrix, as evidenced by changes in FTIR and Raman spectra. The variations of line shapes and shifts of their positions

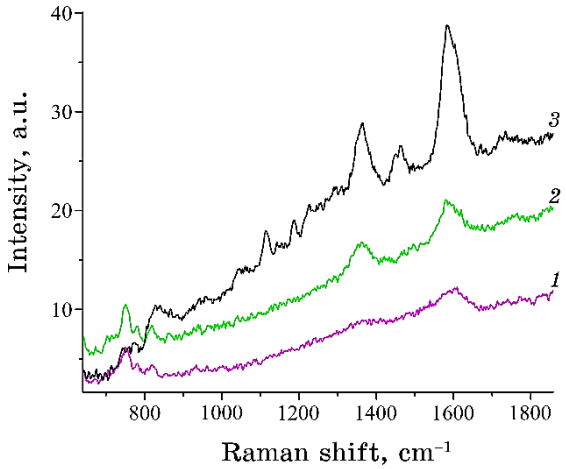

Fig. 3. Raman spectra of MWCNTs (*3*), MoS_2 (*1*) and MWCNTs + MoS_2 nanoparticles (*2*) introduced into an epoxy matrix.

were detected, indicating interactions between the fillers and polymer chains.

Vibrational spectroscopy analysis revealed which chemical bonds are most affected by the inclusion of MWCNTs and GNPs: these are C–H and C–OH vibrations in PE matrix and vibrations of epoxy rings. An enhancement of characteristic peaks in Raman spectra (particularly, the *D*- and *G*-bands) was detected, confirming dispersion and defect levels of graphene structures within the polymer.

The type of nanofiller affects the spectral properties of composites: MWCNTs cause more pronounced changes in the spectra due to the possibility of forming chemical bonds, especially in the polar epoxy matrix. GNPs provide π–π stacking, which is reflected in the band shift.

The chemical structure of the matrix affects the type of interactions, which is reflected in changes in vibrational spectra: polar epoxy resin interacts better with functionalized fillers, its properties are improved with the introduction of dual-type fillers; to improve its properties, non-polar polyethylene requires the use of functionalized fillers.

Employing FTIR and Raman spectroscopy methods together allows for a complete characterization of polymer–nanofiller interactions, aiding in the development of new functional materials.

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