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XRD STUDY OF PHASE EVOLUTION IN ELECTROSPUN PVDF NANOFIBERS AT DIFFERENT CNT/β-Ga_2O_3 RATIOS

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Electrospun poly(vinylidene fluoride) (PVDF)-based nanofibrous mats containing carbon nanotubes (CNTs) and β-Ga_2O_3 nanoparticles were fabricated to investigate composition-dependent phase evolution in the PVDF matrix. Pristine PVDF and composite mats with CNT/β-Ga_2O_3 ratios of 1:1, 1.5:0.5, and 0.5:1.5 wt% were prepared under identical electrospinning conditions using a DMF/acetone solvent system. The crystalline structure and phase composition were studied by X-ray diffraction with Co Kα radiation. The pristine PVDF mat exhibited a multiphase structure composed of α, β, and γ phases. After the incorporation of CNTs and β-Ga_2O_3, clear composition-dependent changes in diffraction peak position and intensity were observed. The 1:1 composition reduced the α-phase contribution and enhanced the γ-phase reflections, while the β phase remained dominant. Increased CNT content favored β-phase development, whereas increased β-Ga_2O_3 content promoted γ-phase formation. The results indicate that CNTs mainly support β-phase stabilization, while β-Ga_2O_3 has a stronger effect on γ-phase evolution and structural rearrangement. These findings show that the crystalline phase balance of electrospun PVDF nanofibers can be effectively tailored by controlled CNT/β-Ga_2O_3 incorporation.
Keywords: *Polyvinylidene fluoride; Beta-gallium oxide; Carbon nanotube; Composite materials; XRD*

INTRODUCTION

In recent years, functional polymer nanocomposites have become a major focus of materials science, since these systems make it possible to combine the flexibility, processability, and mechanical durability of the polymer matrix with the electrical, thermal, and structural advantages of inorganic and nanocarbon fillers [1-3]. In this regard, poly (vinylidene fluoride) (PVDF) is a semicrystalline fluoropolymer distinguished by its high chemical and thermal stability, flexibility, and favorable dielectric, piezoelectric, and pyroelectric properties, which provide broad application potential in sensors, actuators, capacitors, energy harvesting systems, and functional composites [4]. Current studies indicate that PVDF is an important fluoropolymer characterized by outstanding piezoelectric properties, thermal stability, and mechanical strength, providing both structural integrity and functional electrical response. The technological significance of PVDF arises from the combination of its piezoelectric and pyroelectric properties with mechanical, chemical, and thermal stability. PVDF is a semicrystalline thermoplastic fluoropolymer distinguished by high dielectric performance, electroactivity, and engineering durability, and is therefore widely used in energy-related applications, sensing, and functional composites. It is a well-known organic ferroelectric polymer and, due to its thermal and chemical stability, has been successfully applied in sensors, actuators, capacitors, and binder systems. Piezoelectric polymers of the PVDF/PVDF- trifluoroethylene (TrFE) type offer advantages such as simple processing, lightweight and thin structures, chemical resistance, low environmental sensitivity, and biocompatibility. PVDF is a multiphase material and can exist in the α, β, γ, and δ crystalline modifications; among these phases, the β-phase is considered particularly valuable from a functional point of view because of its high polarity and electro-activity [5].

It is well known that the α-phase is a non-polar modification that is readily obtained from the melt, whereas the formation of the β-phase or the α→β transformation is one of the main research directions in the development of PVDF-based functional materials, and this transformation can be enhanced through annealing, stretching, and the incorporation of suitable fillers. At the same time, studies show that within a composite system PVDF acts not only as a passive matrix, but also as an active functional polymer medium that improves surface smoothness, interfacial quality, and the overall piezoelectric response. In recent years, among the semiconductor- and nanocarbon-based

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fillers introduced into PVDF, Ga_2O_3 and carbon nanotubes (CNTs) have attracted particular interest. Owing to its wide band gap of approximately 4.2–5.3 eV, high breakdown field, and favorable electron transport characteristics, Ga_2O_3 has emerged as a promising semiconductor material for high-temperature electronics, ultraviolet photodetectors, gas sensors, and radiation-resistant devices [6-9]. Zhang demonstrated the advantages of Ga_2O_3 in a power diode with record performance and confirmed its strong potential for high-voltage electronics with a breakdown voltage of 8.32 kV. Although several polymorphs of Ga_2O_3 are known, β-Ga_2O_3 is considered the most thermodynamically stable phase, and under high-temperature conditions the other modifications also tend to transform into this phase [10]. On the other hand, CNTs are regarded as one of the most promising nanofillers for PVDF due to their low mass density, high aspect ratio, large specific surface area, high electrical and thermal conductivity, and superior mechanical strength, and they not only create a conductive network within the composite but can also promote β-phase formation, heterogeneous nucleation, and the α→β transformation under appropriate dispersion and functionalization conditions [11, 12]. In addition, CNT incorporation can enhance the mechanical properties, increase the development of the trans/β-oriented structure, and sharply improve electrical conductivity because of the low percolation threshold, although excessively high loading may lead to agglomeration, porosity, and brittleness, resulting in deterioration of the properties. Therefore, the combined use of β-Ga_2O_3 and CNTs within the same PVDF matrix may be considered a favorable approach for obtaining multifunctional composite systems that exhibit a synergistic effect in terms of electron transport, interfacial interaction, mechanical stability, and phase regulation [13,14].

For such composite systems, PVDF nanofibrous mats produced by electrospinning are of particular importance because their large surface area and porous structure enable more efficient distribution of fillers such as CNTs and β-Ga_2O_3 along the fibers, thereby allowing the simultaneous tuning of electrical and structural properties. Nevertheless, accurate analysis of the phase composition and crystallographic structure is essential for a proper understanding of the interaction of fillers with the polymer matrix, the crystallization behavior, and the phase balance in composite nanofibrous materials [15]. In addition, structural defects such as vacancies, dislocations, lattice distortions, and interfacial defects can significantly influence the crystallization behavior, phase stability, and functional properties of polymer-based nanocomposites, and the investigation of these defects by structural analysis methods is important for understanding structure–property relationships. In this respect, X-ray diffraction (XRD) is one of the most reliable structural analysis methods for a wide class of materials ranging from nanopowders and metal alloys to ceramics, composite coatings, and nanofibrous systems, particularly for determining phase composition, identifying crystal structure, and evaluating the degree of crystallinity. Through XRD analysis, structural features such as the formation of new phases, phase transformations, changes in lattice parameters, texture, crystallite size, and microstrain can be monitored, which makes it possible to substantiate the relationship between preparation conditions, filler concentration, modification processes, and functional properties [16].

However, although numerous studies on PVDF-based composites have been reported in the literature, the combined and concentration-dependent effect of β-Ga_2O_3 and CNTs, particularly with respect to structural phase transformation and β-phase formation, remains insufficiently explored in a systematic and comparative manner. In most of the existing studies, these components have either been investigated separately or only their general electrical, dielectric, or mechanical effects have been evaluated. In contrast, the incorporation of β-Ga_2O_3 and CNTs into PVDF at different ratios can modify dispersion, interfacial interaction, crystallization behavior, and phase balance in different ways. Therefore, investigating the changes induced in the structure and functional properties of the PVDF matrix as a function of additive concentration is of considerable importance from both fundamental and applied perspectives.

EXPERIMENTAL PART

This study employed β-Ga_2O_3 nanopowder purchased from Sigma-Aldrich, characterized by a monoclinic crystal structure (space group C2/m), a purity exceeding 99.99%, an average particle size of approximately 20 nm, and a true density of 5.88 g/cm^3. Carbon nanotubes (CNTs) with a minimum purity of 99% were used as the conductive filler. The CNTs had a specific surface area of approximately 700 m^2/g, an average tube diameter of approximately 0.84 nm, and a material density of 1.7–1.9 g/cm^3 at ambient temperature. Here, the material density refers to the density of the CNT material itself, whereas the reported bulk density of approximately 0.1 g/cm^3 refers to the apparent density of the CNT powder, including the interparticle void space, prior to its incorporation into the composite. Poly(vinylidene fluoride) (PVDF) powder, supplied by BLD Pharm (China), served as the polymer matrix and had a molecular weight of 6.0×10^5 g/mol. For preparation of the electrospinning solution, a mixed solvent system consisting of N,N-dimethylformamide (DMF, 99.8%, Sigma-Aldrich, Germany) and acetone (99.9%, PanReac AppliChem, Spain) was used.

Composite nanofibrous samples were prepared using poly(vinylidene fluoride) (PVDF) as the polymer matrix, with gallium oxide (β-Ga_2O_3) and carbon nanotubes (CNTs as functional additives (Fig.1.)). The XRD pattern of the β-Ga_2O_3 nanopowder and CNT was recorded at room temperature using Cu Kα radiation (λ = 1.5406 Å), whereas the pristine and composite PVDF nanofibrous samples were measured using Co Kα (λ = 1.7890 Å) radiation. The corresponding diffraction patterns were analyzed in their respective 2θ ranges for phase identification and comparison. Electrospinning was carried out onto aluminum foil substrates with a thickness of 14.2 ± 0.5 μm. A mixed solvent

system consisting of N,N-dimethylformamide and acetone in a 60:40 volume ratio was employed, and the PVDF concentration in the spinning solution was fixed at 10 wt%. The total content of CNT and β-Ga_2O_3 additives did not exceed 2 wt% relative to the PVDF mass. Different composite compositions were fabricated to examine the effect of additive ratio, namely 1 wt% CNT–1 wt% β-Ga_2O_3, 0.5 wt% CNT–1.5 wt% β-Ga_2O_3, and 1.5 wt% CNT–0.5 wt% β-Ga_2O_3, along with a pristine PVDF sample prepared under identical conditions for comparison. Prior to polymer addition, CNT and β-Ga_2O_3 powders were dispersed in the solvent mixture via ultrasonic treatment at 23.59 kHz for 15 min, after which PVDF was added and the suspension was magnetically stirred at 300 rpm for 1 h to obtain a homogeneous electrospinning solution.

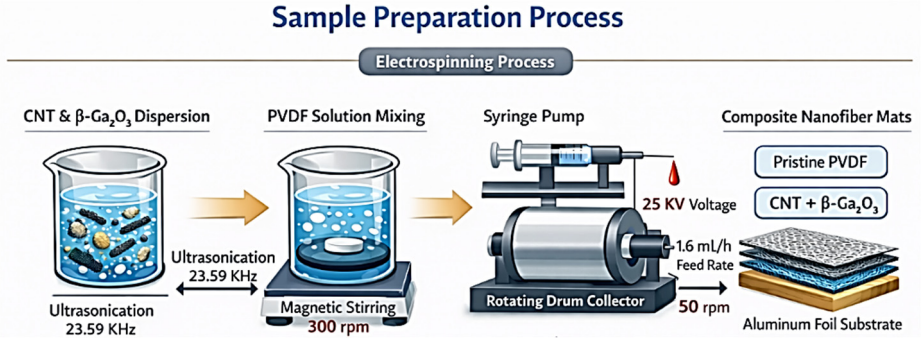

Figure 1. Schematic illustration of the sample synthesis process

Electrospinning was performed using a Nanon-01A system (MECC Co., Ltd., Japan) equipped with a rotating drum collector (F90XSH200). The process parameters included an applied voltage of 25 kV, a solution feed rate of 1.6 mL/h, a spinneret diameter of 0.413 cm, and a spinneret-to-collector distance of 15 cm at a perpendicular orientation. The collector drum rotation speed was maintained at 50 rpm, while the spinneret traversed along the X-axis at 1 cm/s, with a total spinning volume of 10 mL for each sample. The resulting mats formed an interconnected PVDF nanofiber network, either pristine or incorporating CNT and β-Ga_2O_3 additives. Thickness measurements were carried out using a LITEMATIC VL-50 instrument (Mitutoyo, Japan) under a minimal measuring force of 0.01 N. The pristine PVDF mat exhibited a thickness of 15.3 ± 3.1 μm and an areal density of 13.8 ± 0.2 g/m^2, whereas the composite mats showed increased values of 24.1 ± 2.3 μm and 17.4 ± 0.3 g/m^2, respectively, after careful removal from the aluminum foil substrates.

X-ray diffraction (XRD) measurements were carried out to determine the phase composition and crystalline structure of the electrospun PVDF-based nanofibrous mats and their CNT/β-Ga_2O_3-loaded composites. Diffraction patterns were recorded at room temperature using a PANalytical EMPYREAN diffractometer with Co Kα radiation [17]. XRD patterns were recorded over the 2θ range of 20°–100° with a step size of 0.026°. The obtained XRD data were used to compare the pristine PVDF mat with the composite mats (1 wt% CNT–1 wt% β-Ga_2O_3, 0.5 wt% CNT–1.5 wt% β-Ga_2O_3, and 1.5 wt% CNT–0.5 wt% β-Ga_2O_3), enabling evaluation of additive-induced changes in crystallinity and diffraction features relative to the polymer matrix.

RESULTS AND DISCUSSIONS

The experimentally observed diffraction peaks were indexed to the monoclinic β-Ga_2O_3 phase (space group C2/m) and compared with the reference diffraction data (PDF No. 43-1012). Differences in relative peak intensities are attributed to differences in crystallite morphology and preferred orientation. The experimentally observed reflections of monoclinic β-Ga_2O_3 were located at 2θ = 15.70°, 18.95°, 24.24°, 30.11°, 31.73°, 33.49°, 35.22°, and 38.42°, corresponding to the (001), (−201), (201), (400), (−202), (−111), (111), and (−402) crystallographic planes, respectively [18].

The α↔β phase transition in PVDF is one of the main topics of interest in current research. To examine the occurrence of α-to-β or reverse phase transformation, Ga_2O_3 and CNT compositions were introduced into PVDF in different ratios. Fig. 3. shows the concentration-dependent changes in the diffraction peaks of the PVDF samples. In the initial state, the α and β phases are observed in the spectrum of pristine PVDF. Since the XRD analysis was carried out using Co-Kα radiation, the scattering centers of the peaks appear shifted toward higher angles. Thus, the α phase, which is observed at about 18.2° with Cu-Kα radiation, appears at approximately 20.3° in our study. Accordingly, the diffraction peak begins at ~19.75° and ends at 20.98°. This peak corresponds to the (100) reflection of the α phase. The XRD spectra show that the region from 21.75° to 22.5° corresponds to diffraction from the γ phase, associated with the (002) and (110) Miller indices [19]. In the literature, these peaks assigned to this phase have also been observed in the spectrum with slight shifts. The intensity of these peaks corresponding to the γ phase is significantly lower than that of the other peaks, and they appear as a small fraction within the background in a polymorphic manner. The most intense peaks, however, correspond to the diffraction peaks of the β phase. The peak attributed to the β phase is located in the range from 23.19° to 25.5°. It is known that this peak corresponds to indexing as the (110) and (200) reflections.

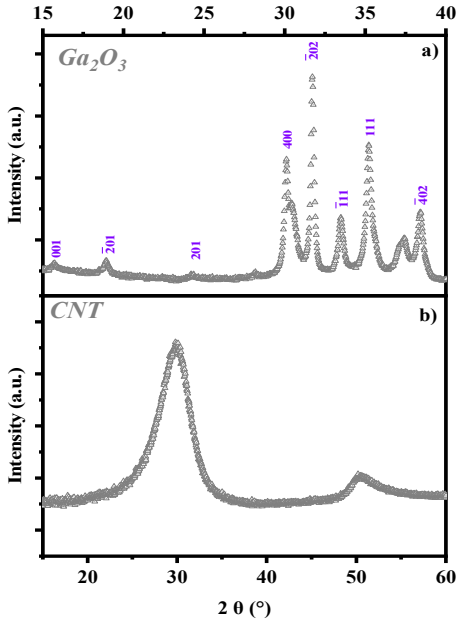

Figure 2. X-ray diffraction spectra of the β-Ga_2O_3 and CNT samples, shown in panels a and b, respectively

In general, it is evident that PVDF exhibits a three-phase structure. If we consider the effect of the compositions and examine the influence of the ratio difference, it can also be seen from the spectrum shown in red that when CNT/Ga_2O_3 is 1/1, a pronounced decrease in the intensity of the α phase is observed together with an increase in the peak corresponding to the γ phase. The addition of the content into the system also changed the scattering centers. When the components were introduced into PVDF in equal proportions, the scattering peak of the α phase was shifted to the range of approximately 19.7° to 20.05°. This may indicate that the introduced composition brought a certain degree of ordering into the system, and the shift of the peak center toward lower angles may be interpreted as an increase in the unit-cell volume. As is known, the γ phase has an orthorhombic structure, and therefore growth in different directions can lead to orientation-dependent changes that result in entirely different lattice parameters.

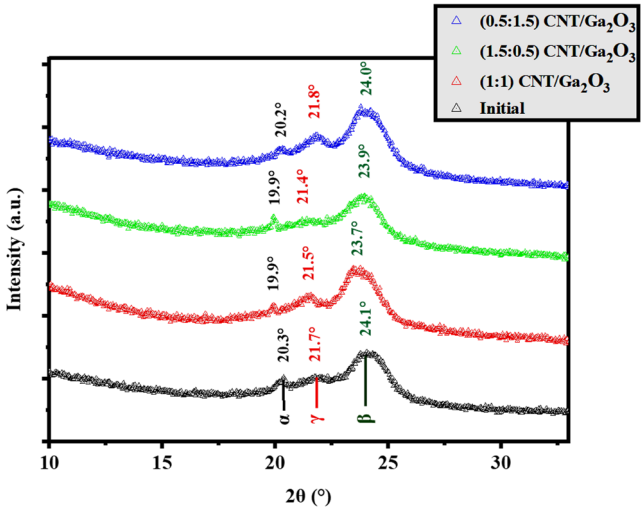

Figure 3. Change in the XRD spectra of PVDF as a function of the embedding amount. Black points correspond to the initial state, red points correspond to the (1:1) ratio, green points correspond to the (1.5:0.5) ratio, and blue points correspond to the (0.5:1.5) CNT/Ga_2O_3 embedding ratios. The centers of the peaks belonging to the α - phase are marked in black, the peaks belonging to the γ – phase are marked in red, and the peaks corresponding to the β - phase are marked in green

In general, it is difficult to distinguish reflections with close d-spacings in PVDF systems. During crystal growth, the periods along the chain in the c-direction are large, and therefore the sample cannot be ideally oriented. It is this imperfection that causes different orientations to appear, allowing the (002) peak of the γ phase to dominate over the (200) reflection. Of course, in this case it should also be noted that the compositions added in equal proportions may have had a stronger effect on a lattice parameter. Thus, growth along the (200) direction may dominate over that along (002). Overall, it becomes clear that the CNT/ Ga_2O_3 additive introduced in equal proportions does not lead to a phase

transition between the α and β phases. Rather, it mainly affects the γ phase and promotes a change in its orientation. Studies show that the addition of oxide ceramics to PVDF leads to a decrease in the α phase and the development of the γ and β phases. However, the CNT introduced into the system improves interfacial interactions and makes the microstructure more heterogeneous [15]. A number of studies have shown that CNT addition promotes the increase of the β phase and raises the β/α ratio, and this difference may even reach 95% [20].

Thus, the results and comparisons show that embedding CNT/Ga_2O_3 in equal proportions (1:1) leads to an increase in β-phase formation and promotes the development of the γ phase. Confirmation of this interpretation can be clearly seen in the case of 1.5/0.5 CNT/ Ga_2O_3 embedding. The corresponding spectrum clearly shows that although the relatively higher CNT content appears to have a positive effect on β-phase formation, the peaks assigned to the γ phase are weaker than those observed for the 1:1 composition. This indicates that Ga_2O_3 promotes the formation of the γ phase. The main confirmation of these results is also clearly observed in the final ratio. The spectrum shown by the blue points in Fig. 3. indicates that, in the case of 0.5/1.5 CNT/Ga_2O_3 embedding, the formation of the γ phase is significantly improved. However, no sharp change is observed in the peaks corresponding to the β phase.

CONCLUSIONS

Overall, the results show that the PVDF system, which initially consists of the α, β, and γ phases, exhibits different phase development after embedding depending on the introduced CNT/Ga_2O_3 ratio. It was found that CNTs mainly promote the stabilization of the β phase, whereas β- Ga_2O_3 exerts a stronger influence on the development of the γ phase. The changes observed in the α phase are mainly associated with the effect of CNT incorporation. Although no significant change in the α phase was recorded when CNT/Ga_2O_3 was introduced in equal proportions, it was determined that α-phase formation was also supported to a certain extent in the case of 1.5/0.5 CNT/Ga_2O_3 embedding. This indicates that the phase balance in the system is determined not only by the presence of the additives, but also by their relative ratio.

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XRD ДОСЛІДЖЕННЯ ФАЗОВОЇ ЕВОЛЮЦІЇ В НАНОВОЛОКНАХ PVDF, ОТРИМАНИХ МЕТОДОМ ЕЛЕКТРОФОРМУВАННЯ ПРИ РІЗНИХ СПІВВІДНОШЕННЯХ CNT/β-Ga_2O_3

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Для дослідження залежної від складу фазової еволюції в матриці PVDF було виготовлено електроформовані нановолокнисті мати на основі полівініліденфториду (PVDF), що містять вуглецеві нанотрубки (CNT) та наночастинки β-Ga_2O_3. Мати з чистого PVDF та композиту зі співвідношеннями CNT/β-Ga_2O_3 1:1, 1,5:0,5 та 0,5:1,5 мас.% було отримано за ідентичних умов електроформування з використанням системи розчинників DMF/ацетон. Кристалічну структуру та фазовий склад вивчали за допомогою рентгенівської дифракції з випромінюванням CoKα. Мати з чистого PVDF демонстрували багатофазну структуру, що складається з α, β та γ фаз. Після додавання CNT та β-Ga_2O_3 спостерігалися чіткі залежні від складу зміни положення та інтенсивності дифракційного піку. Співвідношення 1:1 зменшувало внесок α-фази та посилювало відбиття γ-фази, тоді як β-фаза залишалася домінантною. Збільшений вміст вуглецевих нанотрубок (CNT) сприяв розвитку β-фази, тоді як підвищений вміст β-Ga_2O_3 сприяв утворенню γ-фази. Результати показують, що CNT переважно підтримують стабілізацію β-фази, тоді як β-Ga_2O_3 має сильніший вплив на еволюцію γ-фази та структурну перебудову. Ці результати показують, що кристалічний фазовий баланс електроформованих нановолокон PVDF може бути ефективно регульований шляхом контрольованого включення CNT/β-Ga_2O_3.

Ключові слова: *полівініліденфторид; бета-оксид галію; вуглецеві нанотрубки; композитні матеріали; ХРД*