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Characterizations of Nanodiamond Composite Film for Photonics Applications

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Abstract

Due to its flexibility, small size and low cost, the nanomaterials films are widely desired in optical applications. In this study, a solid nanodiamond (ND) film was fabricated and characterized for photonics applications. The nanocomposite films were prepared by drop casting method where ND was embedded into poly Vinylidene fluoride-trifluoroethylene (PVDF-TrFE) as a host polymer. Both physical and optical properties of the prepared film were investigated. To analyze the structure of fabricated ND-PVDF-TrFE film, various measurement methods were adopted, including a field emission scanning electron microscopy (FESEM), Fourier transform spectroscopy (FTIR), and x-ray diffraction. The nonlinear absorption property of ND-PVDF-TrFE film was studied by balanced twin detector technique (BTDT) using mode-locked fiber laser with pulse width of 1.4 ps at wavelength of 1566 nm. The results showed that the saturable-absorption features of ND-PVDF-TrFE film possess a good nonlinear performance. Furthermore, a good nonlinearity with a high modulation depth makes the ND-PVDF-TrFE film a good candidate to use in many photonics systems such as saturable absorber (SA) in passive Q-switched or mode-locked fiber lasers.

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1. Introduction

Nanomaterials are interesting materials due to their possessing of evident optical, mechanical, thermal, conductive, and electrical features as compared to their bulk counterparts [1, 2]. Therefore, nanomaterials are successfully used in wide and numerous fields of potential applications, from cosmetic products to energy generation devices and aerospace equipment to tissue engineering, because of their outstanding characteristics such as excellent chemical and physical stabilities, very large surface area and lower density [1, 2]. Nanomaterial is responsible for the reduction of size-to-power ratio of cell-phone and computers and numerous other potential applications at work, at home, and everywhere [3-5].

Recently, technological progression seeks for novel and improved type of nanomaterials in order to evolve devices with increasing complexity and enhanced functionality. In such devices, each nanomaterial possesses its own objective and implements given function. Therefore, to produce the desired performance and characteristics, the formation process of films can efficiently manipulate the features and structure of material. The films feature and structure can be guided through choosing suitable synthesis factors and deposition process [6, 7]. These films can be fabricated by several methods depending on their required structure, such as physical vapor deposition, chemical vapour deposition, and the sputtering deposition methods [3, 8]. The progress of such wide

range of fabrication methods has acquired more attentions due to increasingly different demands for the nanomaterials film feature (e.g., purity, composition, structure, electrical, and mechanical properties) as well as requirements on production properties (e.g., time, scale, substrate shapes and efficiency) [8-10]. For example, inorganic metal-based nanoparticles, type of nanomaterials, in form of quantum dots, nanowires, and nanorods [8, 11].

Due to their attractive electrical and optical characteristics, carbon nanomaterials were used in various fields of photonics and optoelectronics applications [10, 11,]. Fullerene is a category of carbon allotropes, which are conceptually sheets of graphene rolled in a form of tubes or spheres [12, 13]. The carbon nanotubes have drawn great attention due to their mechanical strength and their electrical properties, which are convenient for various photonics applications [14, 15, 71]. Carbon is a well-known form of solid-state allotropes with various properties and structure, such as sp^3-bonded diamond, and sp^2 bonded graphite [16, 17]. In the past decades, novel carbon-based nanomaterials entirely composed of sp^2 hybridized carbon atoms have been sophisticated in dimensionalities extended from fullerenes with zero-dimensional, carbon nanotubes (CNTs) with one-dimensional and CNTs to graphene with two-dimensional [1, 14, 18, 72]. Diamond is a sp^3 hybridization protracted network of three-dimensional of carbon atoms. A close-packed structure is resulted from the strong bonds formation between each atom of carbon and its four nearest neighbour, making diamond material possess the highest atom number density on earth at terrestrial pressures [19-21]. As results of this high atomic density and the strong covalent bonds structure, diamond shows extreme features such as the highest elastic modulus and hardness of any other known material [19, 21]. In addition, it has low thermal expansion co-efficiency, high thermal conductivity, and it is a semiconductor material possessing a wide band gap of 5.5 eV [22, 23].

Nanodiamond (ND) particles are the densest allotropic form of carbon in which the atoms are networked through covalent sp^3 bonds in a tetrahedral close-packed crystalline lattice [26, 27]. ND was first fabricated by detonation method in 1960, but they stayed mainly anonymous to the rest of the world until 1980 [28, 29]. In the 1990s, a number of essential breakthroughs led to attracting more attention on this nanomaterial [29, 30]. The ND particles have been fabricated by different processes such as detonation technique, laser ablation, plasma-assisted chemical vapour deposition, high-energy ball milling of high-pressure high-temperature, autoclave synthesis from supercritical fluids, ion irradiation of graphite, chlorination of carbides, and ultrasound cavitation [30, 31]. The ND particles in the form of colloidal suspensions with diameters of 4–5 nm have become obtainable for different applications [26, 32]. Thus, potential applications of ND suspensions have been broken-into additives for diamond sensors, grain refining agent, lubricants, antioxidants, bio imaging, diamond optical limiters, and drug delivery [26, 33-35]. The function of ND suspensions in each one of these applications is unique [31, 36]. Low limiting threshold and fast optical nonlinearity allow one to use ND suspension as optical limiters in the near infrared and visible spectrum range [26]. These optical limiters make a great importance on ND for the quenching of the intensity fluctuations in data processing systems and optical telecommunication systems that operate within the range of 1400-1600 nm [27]. The optical limiting of ND suspension is mainly originated from the nonlinear scattering, nonlinear absorption, and nonlinear refraction [37, 38].

Now, diverse materials have been suggested that could be used for photonics and optoelectronic devices [26, 39-41]. However, the field is still seeking new ways to produce easier and cost effective fabrication processes as well as high reliable and nontoxic nanomaterials. In this paper, a ND film was prepared by the drop casting method for photonics application and short pulse generation. The ND film was produced as a solid nanocomposite film, which is easy to use in photonics applications by embedding ND particles within PVDF-TrFE polymer. Optical properties of such a film are measured to prove its ability to act as a nonlinear device for short pulse generation.

The results revealed that the fabricated film has relatively high modulation depth, high saturation intensity, good morphology, and sharp absorption spectral region. Thereby, nonlinear optical features of ND film were considered as a key parameter for the design and implementation of nanomaterial–based photonics and optoelectronic devices.

To the best of the authors' knowledge, this was the first time that a solid ND film for photonic applications has been prepared and experimentally characterized.

2. Experimental Method

In this section, we describe the materials and process of preparation of ND-PVDF-TrFE film. In addition, the propertied of prepared film characterized to show its surface topography and composition of the nanocomposite as well as its nonlinear optical properties.

2.1. Preparation of ND Film

The ND-PVDF-TrFE composite film was prepared by the casting method to produce a solid nanocomposite film that is easily used in photonics applications. Drop casting technique is a simple solid composite film- formation in which the required materials are blended in a convenient solvent and then dried by spreading it on the substrate. In this experiment, the PVDF-TrFE polymer is a definitely notable organic ferroelectric materials not only for their large remnant polarization, but also for their fast switching time, low processing temperature, and flexibility [42, 43]. Therefore, PVDF-TrFE polymer is efficiently used for many fields of electronic applications due to its unique and excellent properties for producing thin films [44, 45]. PVDF-TrFE (70/30) powder, which was supplied by Kureha, Japan, is adopted as a host material to fabricate the film. Diamond nanopowder from Sigma Aldrich with > 97% purity and <10 nm size is used to form ND film.

The film preparation was carried out by mixing 0.0338 g (10% of total mass) of ND powder with 0.30505 g (90% of total mass) of PVDF-TrFE and then 8 ml of methyl alcohol ketone (MEK) was added to the mixture. Using a warm plate magnetic stirrer , the solution was stirred with constant speed at 85 °C for about 4 h. Sequentially, the resultant solution of ND-PVDF-TrFE composite was casted on flat substrate by spreading it on tight-covered petri-dish and was left to dry at room temperature for about one day. The thickness of the prepared distributed film was measured to be 50 µm. Indeed, the feature and thickness of solid film are depended on the concentration of material and dispersion volume. Other parameters that influence the film composition are the evaporation rate, drying method and the wetting of substrate [66].

It is worth mentioning that this was the first time the drop casting technique was used to prepare ND-PVDF-TrFE composite film. Further, PVDF-TrFE co-polymer was newly utilized to formation photonic films. Recently, Gennady M. Mikheev and coauthor, in 2017, have reported a ND in form in aqueous suspensions. However, the preparation method was complex and needs a specific purification technique [67].

2.2. Characterization Methods

After that, the surface topography of the solid film was examined by using FESEM test. The absorption properties of ND, pure co-polymer and the nanocomposite film were confirmed by FTIR test. Phase identification crystalline lattice parameter of ND, PVDF-TrFE co-polymer and nanocomposite film was verified by X-ray diffraction (XRD) technique. The concentration of the different element included within the composite film was examined by using Energy Dispersive X-ray Spectroscopy (EDX) analysis. The nonlinear optical characteristics of solid composite film were measured by utilizing balance twin detector measurement technique.

3. Result and Discussion

3.1. FESEM Analysis

A field emission scanning electronic microscopy (FESEM) test was achieved to examine the quality of the ND-PVDF-TrFE film surface. Figure 1 shows the FESEM images of ND-PVDF-TrFE at several magnification factors, verifying the quality of the surface distribution of ND-PVDF-TrFE film. The film exhibits free from bubbles and holes that may add scattering losses. Further, the figure shows clear indication about the surface morphology and surface texture of the composite film.

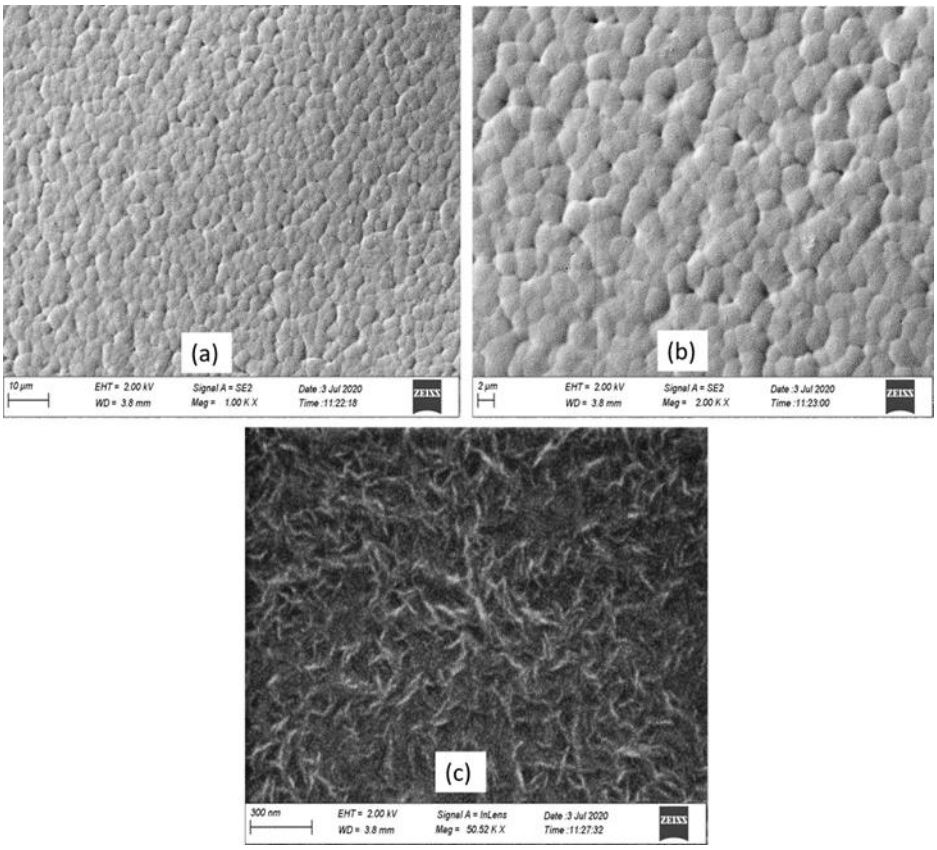


Figure 1: FESEM images of ND-PVDF-TrFE film.

3.2. FTIR Analysis

Figure 2 displays the absorption FTIR spectra of the composite film, ND, and pure PVDF-TrFE. The FTIR of the pure PVDF-TrFE, in which the peaks were observed at 1186 cm^{-1} and 887 cm^{-1}, were related to antisymmetric CF2 stretching vibrations. The three intensive sharp peaks about 850 cm^{-1}, 1288 cm^{-1}, and 1400 cm^{-1} emphasized the β-phase of the PVDF-TrFE that were responsible for its highest dipole moment, such that both (850 ,1288) cm^{-1} peaks were referred to the CF2 symmetric stretching mode, while the 1400 cm^{-1} peak mentions the wagging vibration of CH2. The FTIR spectra of nanodiamond, in most cases, contained different functional groups, which were formatted during purification of diamond using strong acids through manufacturing process [46]. The peaks at 3389 cm^{-1} and at 1650 cm^{-1} were assigned to the C–OH deformation and stretching vibrations. The peak was specified to the mode of 1384 cm^{-1} related to the OH bending vibration in COOH. The bands at 2640 cm^{-1}, 2287 cm^{-1}, 2070 cm^{-1} and 1159 cm^{-1}were attributed to COOH, C –N, C –O and C –O-C functional groups that were connected to the nanodiamond surface [47]. The peaks observed at (1050–1150 cm^{-1}) confirmed the (C–O) stretching, at (1710-2858 cm^{-1}) confirmed (C=O) stretching vibration, and the observed peak at 3450 cm^{-1} confirmed the (O–H) stretching [48]. The FTIR spectra of the ND-PVDF-TrFE nanocomposite film proved that the absorption feature of the co-polymer was strongly affected by the ND adding. It was obvious, most of the observed absorption peaks of the composite film increased as compared to that of both ND and PVDF-TrFE spectra. Because, the β and γ-phases owns the same chain structures of polymer, they show a similar number of bands, and most of them observed at identical wavenumbers. The strong peak at 840 $cm^{-1,}$ which is the characteristic peak of the β-phase was very close to the peak at 833 cm^{-1} for γ-phase [49]. The other peaks underlined the dynamic contact of the suggested co-polymer and the NDs surface. The band observed around (1000-1300) cm^{-1} together with notable peaks are referring to C-O-C stretching vibration. Other peaks observed about 1250 cm^{-1} and 1500 cm^{-1} assign to the CO bending vibration. The peaks watched about 1332 cm^{-1} confirm the C-C stretching vibration of the diamond lattice, and the peak around 1340 cm^{-1} were because of the C-F stretching which proved the presence of both ND and the suggested polymer within the film [48, 50].

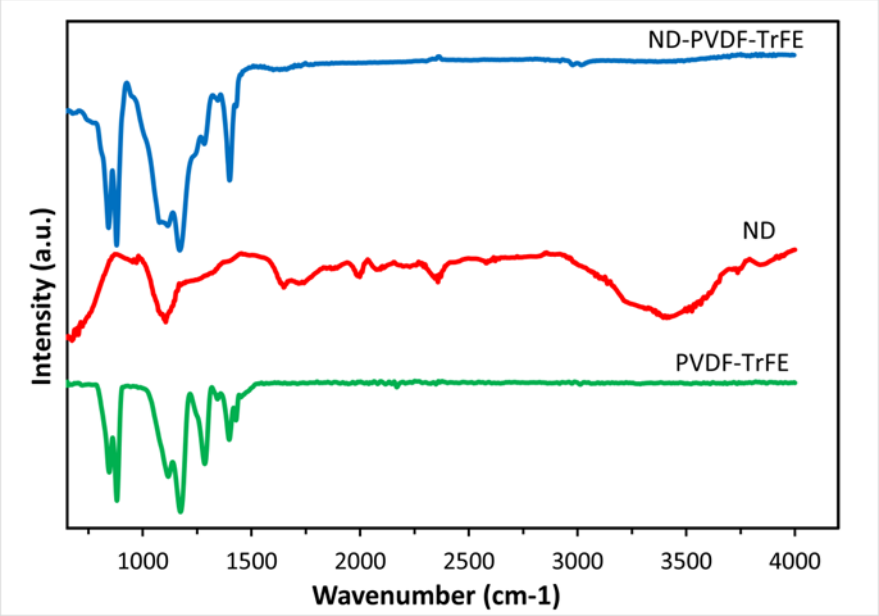


Figure 2: FTIR spectra of ND-PVDF-TrFE nanocomposite film, ND particles and PVDF-TrFE polymer.

3.3. X-Ray Riffraction Analysis (XRD)

Phase identification crystalline lattice parameter of the ND, PVDF-TrFE co-polymer and the nanocomposite film could be verified by X-ray diffraction (XRD) technique. The XRD patterns of the ND, PVDF-TrFE polymer and the ND-PVDF-TrFE film depicted in Fig. 3. At 2θ, ND nanopowder have two broad peaks of 44.54^0 and 75.4^0, which are identical to the (1 1 1), (2 2 0) cubic diamond planes. The two peaks show that the ND crystal was cubic and the diffraction peaks were clearly broadened due to the tiny size of the ND crystal [47]. The XRD pattern of the PVDF-TRFE co-polymer shows distinctive peaks, which are related to the β phase and located at 2θ = 19.92° assigned to (110/200) planes of reflection. The elevation of such diffraction peak marks a high crystalline structure in the β phase, which means the co-polymer possess good piezoelectric characteristic. From such XRD pattern, the crystallite size was calculated using 2θ, full width at half maxima and intensity values [51]. It was found that the crystallite size is 23.13 nm. The other observed two polymer peaks at 2θ =35°, and 2θ =40° which is in agreement with the value given by Duo Mao et al [52, 73]. Furthermore, Fig. 3 illustrates the XRD pattern of the composite film; underline the implementation of the ND created by the proposed preparation method. The same peaks obtained in XRD pattern of the ND and PVDF-TRFE co-polymer is clearly observed and at the same position. The peaks positions totally coincide with the previously published articles [50, 53]. The sharp diffraction peaks obtain in XRD spectrum demonstrating that the polymer and nanodiamond within the prepared film possess high purity with good crystallinity due to no other XRD peaks are observed.

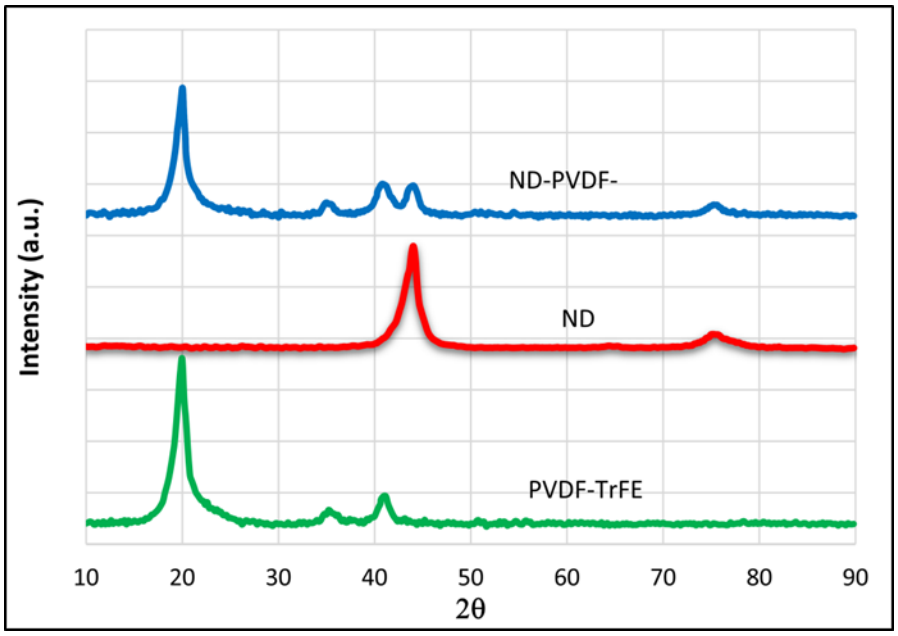
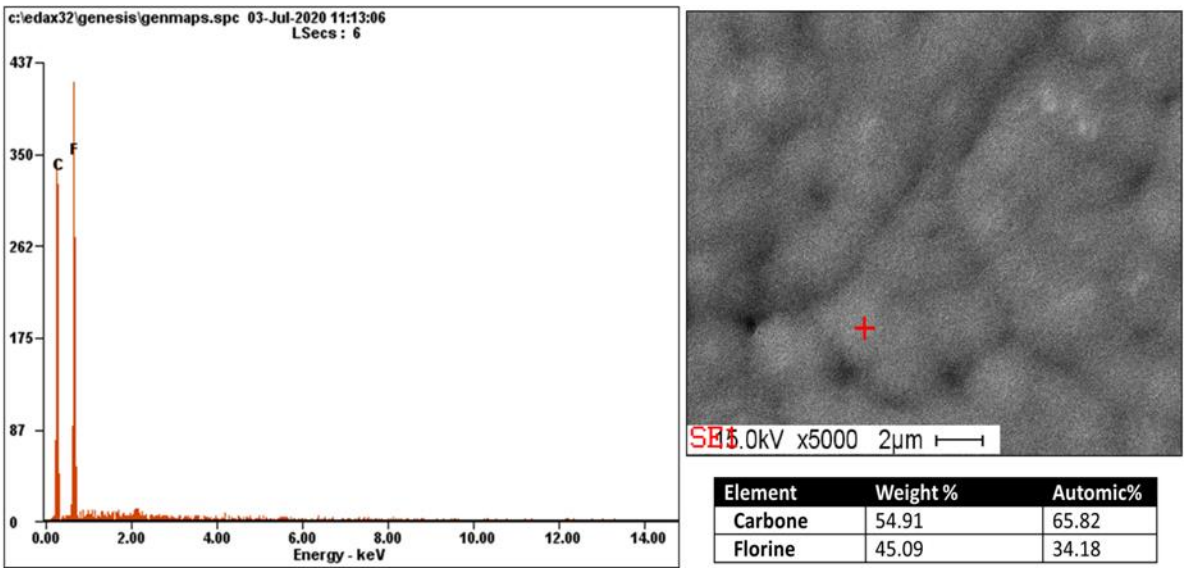


Figure 3: XRD spectrum of ND-PVDF-TrFE film, ND, PVDF-TrFE.

3.4. Energy Dispersive X-ray Spectroscopy (EDX)

EDX analysis of the ND-PVDF-TrFE film with scanning electron microscopy (SEM) was made to show the concentration of the different element within its composition as shown in Fig. 4. Depending on the EDX measurements, the composition of the Carbone element is about 54.91 %, which mainly belongs to the nanodiamond base material, and composition of Florine is about 45.09 %, that mainly belongs to the polymer.



Element	Weight %	Automic%
Carbone	54.91	65.82
Florine	45.09	34.18

Figure 4: Energy Dispersive Spectroscopy (EDX) analysis of DNBSA.

3.5. Nonlinear Absorption Measurements (NLAM)

In this subsection, a nonlinear absorption of the prepared distributed film is experimentally explored. The maximum change in the absorption of SA is called modulation depth, which can be stimulated by launching a light with specific wavelength. The modulation depth represents one important parameter in the design passively mode-locked lasers. A relatively large modulation depth saturable absorber has a robust pulse shaping ability, in which it can produce a short pulse width and credible self-starting operation. In order to study this feature of the suggested composite film, an effective and easily executed balanced twin detector technique (BTDT) is utilized.

The BTDT comprises an Erbium mode-locked fiber laser, as an optical source possesses pulse width of 1.4 ps centered at 1566 nm with a repetition rate of 1.2 MHz. The intensity launched to ND-PVDF-TrFE film is changed by fine-tuning electrical attenuator, as shown in Fig. 5. The nonlinear absorption curve (NLAC) is measured in real time as presented in Fig. 6. Three important features of the ND-PVDF-TrFE film could be extrapolated from the NLAM, namely: modulation depth, non-saturable absorption, and saturation intensity. The maximum possible change in the optical loss is denoted by modulation depth. The non-saturable absorptions represent the unwanted part of absorption, which cannot be saturated. The saturated intensity represent the optical intensity (power per unit area) that it takes, in a steady state value, to minimize the absorption to half of its unsaturated value. It can be observed from Fig. 6, the modulation depth of the prepared composite film is 15%. This value for modulation depth is suitable and reasonable for using the film in pulse shaping, specifically, in operation of a short pulse generation and reliable self-starting of mode locked lasers [54]. The non-saturable absorption of our film is 10%, such value is not very low but still lower than value obtained from other films in previous work such as CBNM [33]. Indeed, in most cases it is resulted from the film thickness and the presence of impurity within the film. The saturation intensity of the proposed film is 2000 MW/cm^2. It is relatively high value but it is expected because of the strong nature of nanodiamond particles. They can cope with high intensity laser radiation without degradation, activating its operational regimes with high saturation intensities [55].

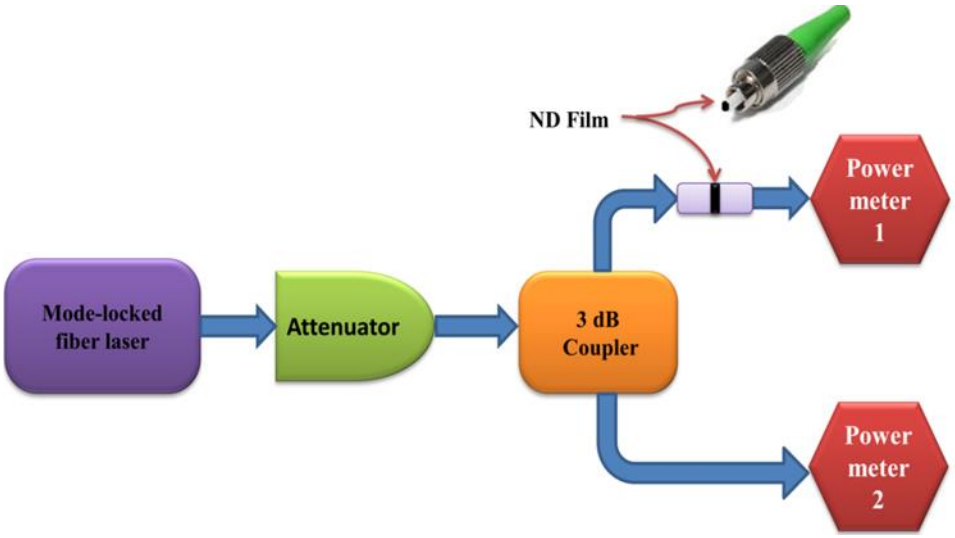


Figure 5: BTDT measurement setup configuration.

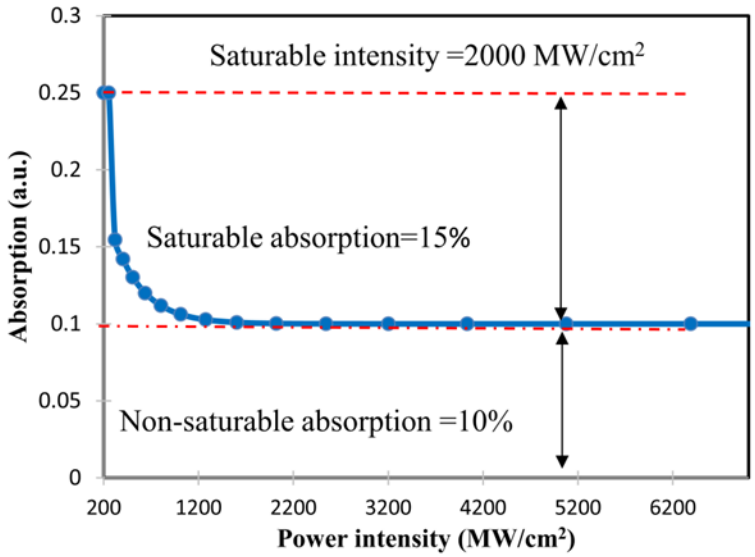


Figure 6: Nonlinear curve of the 10% ND-PVDF-TrFE film.

3.6. Discussion

Figure 1 confirms the surface distribution of suggested film by using the FSEM technique. The FTIR spectra used to confirm the absorption characteristics of ND, pure co-polymer and the nanocomposite film as shown clearly in Fig. 2. The composite film have a specific band of absorption span for limited range of a wave number. Figure 3 illustrates the XRD measurement of the ND, co-polymer and the ND-PVDF-TrFE film, the sharp peaks obtained in such figure, confirm the high purity and good crystallinity of the nanomaterial used in the presented research. The nonlinear absorption of the suggested film was measured by using effective and easily executed balanced twin detector technology BTDT.

It is worth mentioning, the performance of nanomaterials as SA was determined by several key parameters such as modulation depth, saturated intensity, recovery time, saturation fluence, and damage threshold [56]. In general, the relatively high modulation depth value and low non-saturable losses value was required to produce a shorter pulse width either in the mode locking or in the Q-switching operation. However, the diameter, excitation wavelength, size, crystalline structure, and other conditions are effected on the modulation depth of SA film [57]. Figure 6 displays NLAM of the ND-PVDF-TrFE film in which the modulation depth is 15%, the saturation intensity is 2000MW/cm^2 and non-saturable absorption is 10%. The nonlinear optical feature of the composite film, the distribution of its surface, its crystalline structure, its simple, and easily preparation method make our ND-PVDF-TrFE film a suitable candidate for operating as saturable absorber (SA) in the passively ultra-short pulse generation. Table 1 shows the comparison between various carbon-based nanomaterials (CBNMs) in terms of modulation depth, saturation intensity, and non-linear absorption. It can be concluded from the table that the CBNMs has good nonlinear properties. The CBNMs, including graphene[58], CNT[59], single wall CNT (SWCNT) [58], dual wall CNT (DWCNT) [60], and graphene oxide (GO)[61], have played crucial roles on photonics applications including ultrashort-pulse generation. Generally, the graphene has great ability to produce high nonlinear absorption and the highest modulation depth. However, it still has certain limitations related to its weak absorption coefficient and its low damage threshold [62, 63]. The CNTs also have low damage threshold, and their operating wavelength depends on their diameters [62].

Although its modulation depth is low, the aqueous ND suspension has been utilized as an optical limiter [62]. However, highest saturation intensity with highest damage threshold can be obtained with ND nanomaterial. In contrast to our ND film, the preparation of detonation ND (DND) suspensions has been carried out by a series of chemical treatments to produce an aqueous DND suspension [55]. Such aqueous suspensions must be put in an optical transparent container to perform the different optical measurements. The container adds complexity to the measurement system and introduces additional effects and losses added to the measured parameters. Furthermore, aqueous DNDs have different surface groups affecting its saturable absorption performance [55]. In contrast to aqueous DNDs, the proposed ND-PVDF-TrFE film has higher modulation depth and solid form, which makes the film more compatible to utilize in photonics applications as a passive nonlinear device.

Table 1: Non-linear optical characteristics of various CBNM.

CBNM	Modulation depth (%)	Nonlinear absorption α_{NS} (%)	Saturation intensity (MW·cm^{-2})	Reference
Mono-layer graphene	65.9	3.24	0.53	[45]
1-4 layer graphene	42.69	31.48	0.77	[45]
4-8 layer graphene	18.2	67.85	1.09	[45]
Graphene/ PVA	28.3	-	0.75	[33]
CNT	28	1.07	25.9	[64]
GO/PVA	16.1	-	1.32	[65]
Aqueous ND suspension	3.8	-	89 000	[26]
This work	15	10	2000	

As a final result, both the ND film formed from ND and PVDF-TrFE polymer, kept their physical and optical features within composite solid film. The physical characteristics of ND, namely, good optical properties and nontoxic feature with tuneable surface structure and large surface area with a high optical damage threshold, more than 500 GW/cm^2, and large band gap of 5.5 eV have been reported [68]. Further, ND has a good physical performance, such as superior thermal conductivity, highest hardness, high refractive index, and high dispensability [69, 70]. In this research, optical parameters of ND film are examined to explore its saturable absorption feature such as modulation depth, saturation intensity, and non-saturable absorption. They confirm that ND film is proper to employ in the passive short pulse generation, optical limiting and nonlinear optics.

4. Conclusion

In this paper, the ND film for photonics applications has been prepared, and its physical and optical properties have been experimentally demonstrated. The ND-PVDF-TrFE film has been fabricated in a simple and easy drop casting method to form solid nanocomposite film by embedding a ND powder in PVDF-TrFE polymer film. The physical and optical properties of the proposed film have been demonstrated by different measurement techniques, such as, FSEM, FTIR, XRD, EDX, and NLAM. The obtained results show that, ND film has a strong optical nonlinearity, making it very attractive for generating short pulses in fiber laser. The fabricated ND-PVDF-TrFE film has a high modulation depth of about 15% and saturation intensity of 2000 MW/cm^2.

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