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Research Paper

Investigation of Surface Morphology and Nano-Mechanical Behavior of PTB7-Th:PCBM Thin Films

Muhammad Riaz [a,*], Iqra Munir [a], Nida [a], Noor Ul Huda [a], Rabia Shahzad [a]

[a] Department of Physics, University of Jhang, Chiniot road, Jhang 35400, Pakistan.

ARTICLE INFORMATION

Received	21 July 2026
Received in revised form	27 Aug 2026
Accepted	27 Aug 2026
Available Online	15 Sep 2026

*CORRESPONDING AUTHOR

Muhammad Riaz
Department of Physics, University of Jhang, Chiniot road, Jhang 35400, Pakistan

Email mriaz@uoj.edu.pk

ABSTRACT

Purpose: This study investigates the morphological stability and nanomechanical properties of PTB7-Th:PCBM thin films on two substrates under thermal stress, focusing on the effects of thermal annealing on morphology and mechanical response.
Design/methodology/approach: PTB7-Th:PCBM thin films were deposited on glass substrates and subjected to thermal annealing at 150 °C. The morphological changes and nanomechanical properties of the films were investigated, including surface roughness, indentation depth, drift rate, and unloading behavior. The effect of changing the substrate from glass to glass/ITO on the nanomechanical response was also examined.
Findings: Thermal annealing at 150 °C resulted in a maximum reduction in surface roughness of 2.151 nm and promoted robust nanocrystalline ordering of PCBM. The nanomechanical response exhibited a distinct pop-out phenomenon during unloading, together with uniform film characteristics and excellent reproducibility. The pop-out phenomenon disappeared when the substrate was changed from glass to glass/ITO, indicating that its occurrence is influenced by both temperature and the substrate–film interface. An average indentation depth of 756.4 nm was obtained at an average drift rate of 0.346 nm/s.
Originality: This study highlights the substrate-dependent pop-out behavior of PTB7-Th:PCBM films, providing new insight into their nanomechanical response and stability.
Research limitations/implications: Further studies are needed to optimize annealing and substrate conditions for improved film stability.
Practical implications: Thermal treatment and substrate interfaces are crucial for stable and efficient organic photovoltaics.
Social implications: Improved film stability may support the development of durable, flexible, and low-cost solar-energy technologies.

***Keywords*:** Annealing, organic photovoltaics, nano-indentations, surface morphology

1. Introduction

Solution-processed organic solar cells (OSCs) have emerged as a cost-effective and more environmentally sustainable alternative to conventional silicon-based photovoltaic devices (Liu et al., 2017). This technology is also suitable for mass production as it does not contain any toxic elements (e.g., Pb or Cd) and can be fabricated using a process similar to the printing of newspapers. OSCs are expected to outperform their inorganic counterparts because of their many advantages such as low cost production, high flexibility, ease of synthesis and transparency (Shah et al., 2018; Shah et al., 2017; Shah et al., 2016). Because of their semitransparency and color tunability the most promising applications of OSCs are building integrated photovoltaics (BVPs) and power-generating windows. However, limited short term stability remains a major challenge for this technology.

Solution-processable PCBM and its derivatives have been widely employed as electron acceptors in organic solar cells (OSCs) and significantly influence their overall photovoltaic performance (Mikroyannidis et al., 2011; Morvillo et al., 2012). PTB7-Th has emerged as an effective electron-donor material and has attracted considerable attention in organic photovoltaics because of its favorable properties, including a broad optical absorption range extending from approximately 500 to 785 nm (Vogelbaum and Sauvé, 2017), band gap around 1.58 eV (Xiao et al., 2017) and good prospects for semitransparent and flexible solar cell fabrication (J. Ma et al., 2018; Xia et al., 2018; L. Zhou et al., 2018). However, one of the recent strategy to overcome low power conversion efficiency (PCE) and enhance transparency is using blend of PTB7-Th:PCBM and has attracted significant interest because of its promising results (Chen et al., 2018; Li-Juan et al., 2016; Song et al., 2019).

PCE of the OSCs can be enhanced using numerous optimization strategies such as optimizing different layers of the divice structure (Cui et al., 2017; Wan et al., 2017) and heat treatment (Kwon et al., 2015). Temperature-assisted treatment significantly modifies the topographical characteristics of the deposited thinfilms which play a key role in charge carriers generation and their transportation. While extensive research is reported on different optimization strategies, no considerable work is present on investigating the nano-mechanical properties (NMP) of organic materials (PTB7-Th:PCBM) thin films. Nano-indentation is one of the most accurate, reliable and popular technique used for investigating NMP of thin films (Shu et al., 2020; F. Zhou et al., 2020).

The structural and nanomechanical characteristics of PTB7-Th thin films have a substantial impact on their photovoltaic performance and long-term stability. Recent studies have shown that thermal stress can induce crystallization, phase separation, and morphological changes in organic photovoltaic active layers, leading to degradation of device performance (Luong et al., 2023; Z. Ma et al., 2022; Qi et al., 2022). In particular, PTB7-Th-based systems exhibit morphology-dependent changes under elevated temperatures, highlighting the importance of controlling molecular organization and phase separation (Luong et al., 2023). Recent reviews have further emphasized that thermal and mechanical stability are closely associated with the nanoscale morphology and structural integrity of OPV films (Ding et al., 2024; Liang et al., 2026; Qin et al., 2025). Therefore, AFM-based morphological and NM characterization of PTB7-Th films provides a useful approach for correlating surface roughness, local mechanical properties, and thermal-induced structural changes. While considerable attention has been devoted to the morphology and thermal degradation of PTB7-Th-based photovoltaic films, the correlation between their nanoscale surface morphology and local NMP, particularly after thermal treatment, remains comparatively underexplored.

A review of the existing literature indicates that, we are the very first to report use of nano-indentation technique to investigate the NMP of PTB7-Th:PCBM thin films on glass base material and glass/ITO substrate pre annealing and post thermal treatment at 150 °C. The present study may contribute to improving OSC performance by providing deeper insights into stress, flexibility, and stability limitations, identifying factors responsible for performance degradation, addressing existing uncertainties regarding their application, and establishing potential avenues for further investigation of the NM behavior of carbon-based materials and devices using nanoindentation.

2. Materials and Methods

All chemicals, including chlorobenzene (CB), PTB7-Th, PCBM, and ITO-coated glass substrates, were obtained from Luminescence Technology Corp., Taiwan. Prior to film deposition process, the glass and ITO-coated glass substrates were rinsed thoroughly using deionized (DI) water and acetone. A PTB7-Th solution with a weight ratio of 1:1.5 was prepared in chlorobenzene at a concentration of 40 mg/mL. The prepared blend solution was prepared and stored under an argon atmosphere inside a glovebox for one week. The selected composition was based on optimized ratios reported in the literature (He et al., 2015; Li and Brabec, 2015; Liao et al., 2013; Liao et al., 2014; Nam et al., 2015).

The prepared PTB7-Th:PCBM solution was deposited onto the cleaned glass and ITO-coated glass substrates using spin coating at 3000 rpm for 30 s. This procedure was employed to obtain uniform thin films for subsequent morphological and nanomechanical characterization. The substrates had dimensions of 25 × 20 mm². The deposited PTB7-Th layer had an approximate thickness of 2-2.5 µm, whereas the ITO-coated glass substrate had a thickness of approximately 0.7 mm. Following deposition, heat treatment of the thin film was conducted of the coated films at 150 °C for 30 min. The samples prepared on glass substrates were designated S1, S2, S3, and S4, while those deposited on glass/ITO substrates were designated S11, S22, S33, and S44. Optical microscopy was performed using a BHM model microscope manufactured by Olympus, Japan. Morphological features of the deposited thin films was characterized using AFM with a Nanosurf AFM system. The nanomechanical behavior of the films was evaluated using an iMicro nanoindenter.

The principle of the nanoindentation experiment is presented in Fig. 1(a), while the corresponding load-depth response is shown in Fig. 1(b). During testing, each specimen was securely positioned beneath the indenter tip. The trapezoidal indenter was then brought gradually into contact with the sample surface. Once contact was established, the indentation process proceeded at a controlled loading rate until the predetermined uppermost load was reached. The resulting load displacement data were evaluated using the Oliver Pharr analysis method. The principal parameters obtained from the indentation curves included the maximum applied load (Pmax), maximum penetration depth (hmax), contact depth (hc), and unloading stiffness. These parameters are schematically represented in Fig. 1(a).

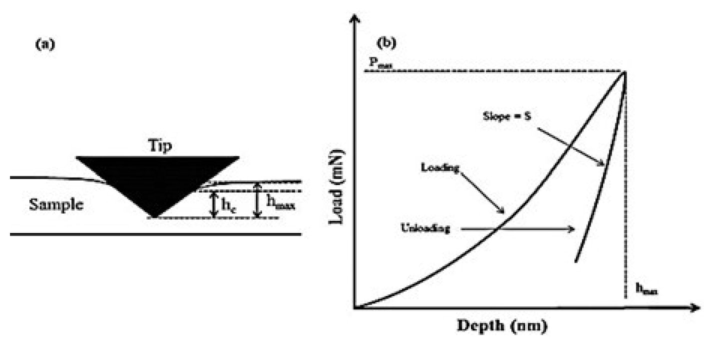

Figure 1. The schematic presentation of nano-indentation measurement (a) the testing mechanism (b) load-depth relation

3. Results and Discussion

Figure 2 compares the optical micrographs of PTB7-Th:PCBM thin films deposited on both distinct supporting layer, where Fig. 2(a, c) are the images obtained for pre-annealed samples of thin films and Fig. 2(b, d) are the images obtained after heat treatment at 150 °C for 30 min. The results indicate that the PTB7-Th:PCBM film deposited on glass/ITO substrate turned out much smoother as compared to films deposited on only glass substrates. Annealing resulted in crystallization of PCBM where most of the crystals are needle shaped however; few disc like structure were also observed which can be the the early stages of crystallization. A comparable trend was also identified in previous reports (Karagiannidis et al., 2011).

The morphological characteristics of the active layer is a critical factor in governing the globally device performance and stability of sensors and

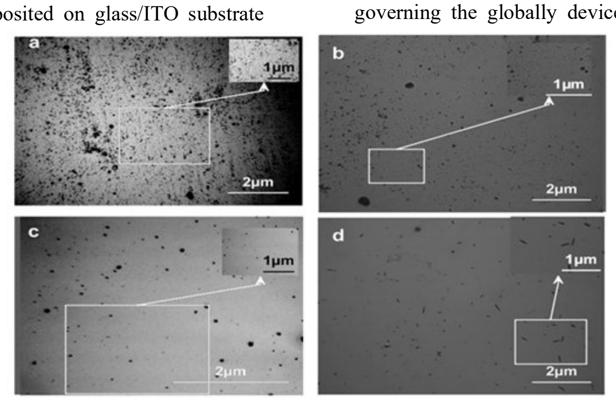

Figure 2. Optical microscope images for PTB7-Th:PCBM thin films on ITO substrate (c, d) and PTB7-Th:PCBM films on glass substrate (a, b) pre annealing (a, c) post annealing (b, d).

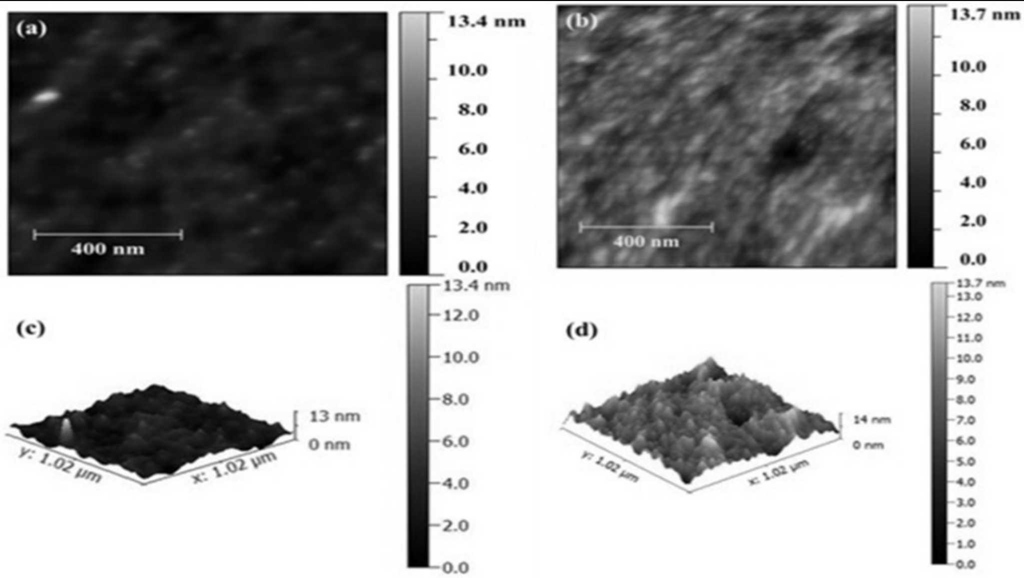

Figure 3. AFM results before annealing for PTB7-Th:PCBM on (a,c) for Glass/ITO substrate (b,d) and Glass substrate.

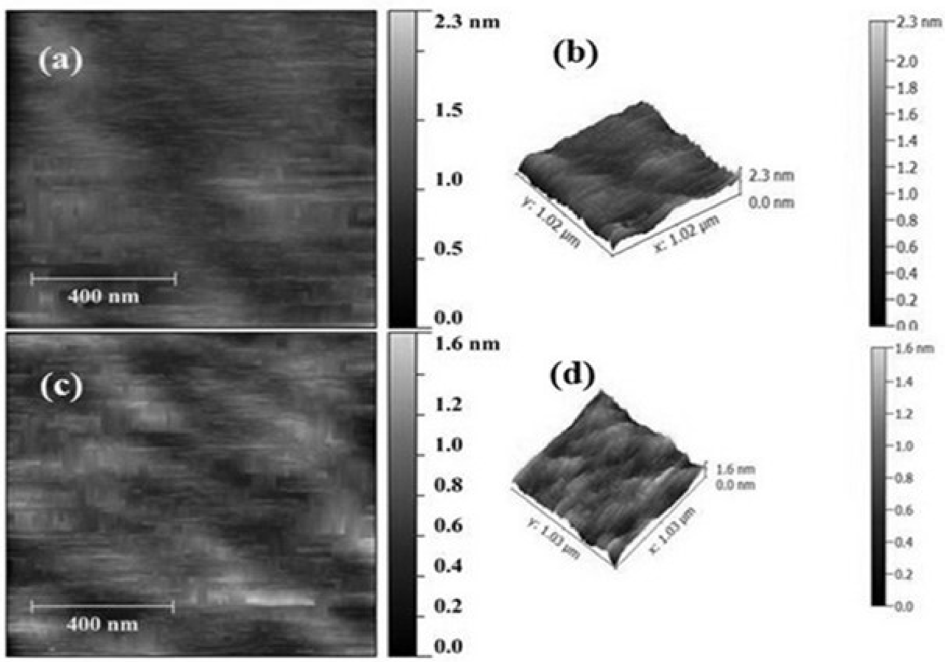

Figure 4. AFM topographical images of the PTB7-Th:PCBM thin films following thermal annealing (a, b) on Glass and (c, d) Glass/ITO supporting substrate.

solar cells. In this study, the surface characteristics of PTB7-Th:PCBM films were investigated by considering both thermal treatment and substrate type. As presented in Fig. 3, the films deposited on substrates S1 and S11 exhibited average surface roughness values of 7.552 nm and 2.969 nm, respectively. The lower roughness observed for S11 indicates that the ITO substrate provides a considerably smoother surface for PTB7-Th:PCBM film deposition compared with bare glass. Subsequently, both samples were thermally annealed at 150 °C for 30 min, and the corresponding AFM images are presented in Fig. 4(a,b) for S1 and Fig. 4(c,d) for S11. Following annealing, the nominal surface roughness decreased substantially to 1.0925 nm for S1 and 0.8180 nm for S11, corresponding to reductions of 6.4595 nm and 2.151 nm, respectively.

This morphological improvement is consistent with the relatively uniform nano-mechanical response observed after annealing. The reduction in surface roughness may also minimize local variations in tip sample contact and deformation, resulting in more reproducible indentation measurements. However, the present data do not establish a direct quantitative relationship between roughness and indentation modulus; therefore, the observed correlation is interpreted qualitatively.

Nanoindentation tests were conducted for the thin film of PTB7-Th:PCBM on a glass and glass/ITO suporting layers, with the corresponding results presented in Figs. 5 and 6, respectively. The modulus as a function of applied load for the unannealed and annealed films is shown in Fig. 5(a,c), where the samples were thermally treated at 150 °C for 30 min. These findings indicate that thermal annealing enhances the load-bearing capability of the PTB7-Th:PCBM films. It also proves the uniformity of NMP throughout the thin film resulting in good device reproducibility suitable for large scale printing.

Fig. 5(b) presents the load versus depth response of the samples before annealing. The loading curve increases smoothly without any noticeable pop-in or other abrupt discontinuities, indicating a predominantly elastic re-

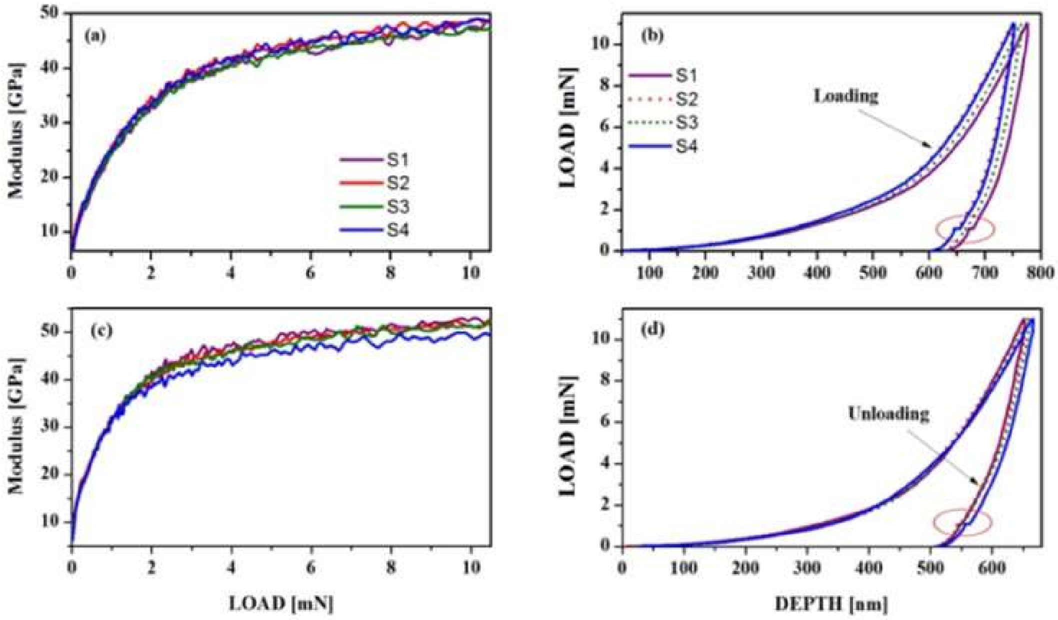

Figure 5 NMP Response of PTB7-Th:PCBM Films Deposited on Glass.

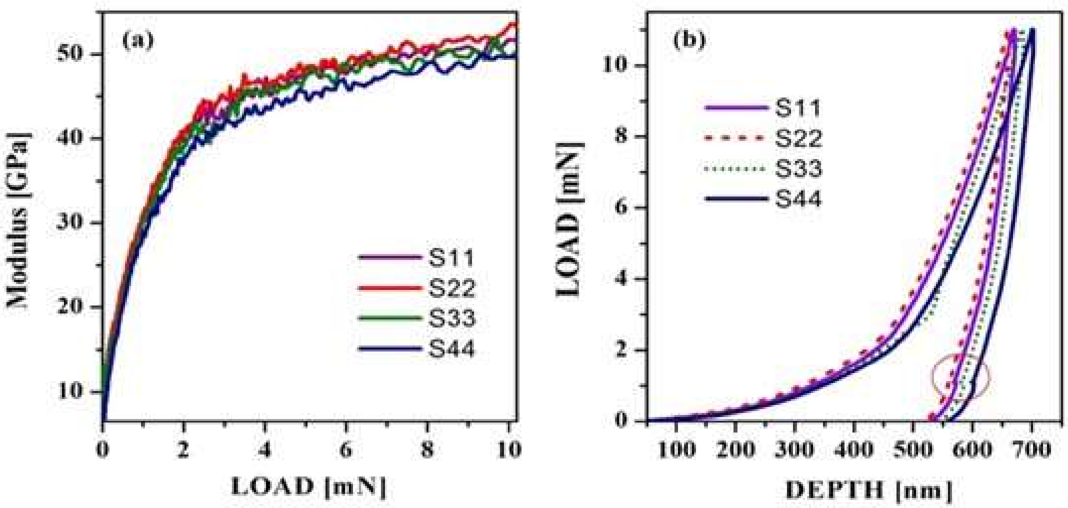

Figure 6 NMP Response of PTB7-Th:PCBM Films on glass/ITO substrate.

sponse over the applied loading range. The absence of a pop-in event suggests that no sudden slip or localized plastic deformation occurs during loading. In contrast, a distinct pop-out event is observed during the unloading stage. Such pop-out behavior has also been reported in various materials and is generally associated with changes in the deformation mechanism during unloading. We observed this pop-out before and after annealing for PTB7-Th:PCBM thin films. For some other materials (diamond cubic-Si) this behavior was observed only after annealing. It later disappeared as annealing temperature was further increased. For all samples, the nanoindentation tests were conducted up to a peak load of 11 mN at a constant strain rate of 0.2 s^{-1}, with an average drift rate of 0.68 nm/s recorded during the measurements.The Fig. 5 and Fig. 6 data show that PTB7-Th:PCBM is almost 4 times softer than PMMA, PC, PS and UHMWPE polymers studied in (Liao et al., 2014). That's meant PTB7-Th:PCBM is more plastic in nature in comparison to other materials and such a materials is more valuable for flexible electronics.

Fig. 6 illustrates the nanoindentation response of the Glass/ITO/PTB7-Th:PCBM thin films. The loading and unloading data were achieved at average drift rate of 0.346 nm/s. The average obtained depth was 756.4 nm. By comparing Fig. 5 and Fig. 6 it can be observed that the pop-out behavior previously present disappeared. This demonstrates that not only temperature, but also the substrate nature has an effect on the pop-out. The pop-in is circled in Fig.5(d) and Fig.6(b) for the sake of clarity. The pop-in phenomenon observed in the loading curves indicates a sudden increase in indentation depth at nearly constant load, suggesting a localized deformation instability. In PTB7-Th films, this behavior may be associated with localized plastic/viscoelastic deformation, molecular-chain rearrangement, or local reorganization of donor–acceptor domains (Ohmura and Wakeda, 2021). Similar pop-in events have been reported during nanoindentation and are generally related to the onset of irreversible deformation (Göken and Kempf, 2001; Ohmura and Wakeda, 2021). Therefore, the observed discontinuity is attributed to localized structural rearrangement within the heterogeneous organic film rather than being conclusively assigned to a phase transformation.

The interpretation of the observed pop-in phenomenon is subject to certain limitations, as the present nanoindentation measurements alone cannot conclusively identify the underlying deformation mechanism. The observed discontinuity may be associated with localized plastic/viscoelastic deformation, molecular rearrangement, or morphological heterogeneity within the PTB7-Th film. Therefore, the proposed mechanism should be considered tentative. Future investigations combining post-indentation AFM/SEM imaging, structural characterization such as XRD, and nanoindentation at different loading rates and maximum depths would help clarify the origin of this phenomenon and provide a more definitive understanding of the deformation mechanism.

Conclusions

In this work, the surface morphology and NMP of PTB7-Th:PCBM thin films deposited on glass and glass/ITO substrates were investigated using AFM and nanoindentation. The PTB7-Th:PCBM blend was prepared by dissolving the two components in the selected solvent at a 1:1.5 weight ratio in chlorobenzene (40 mg/mL) and spin-coated at 3000 rpm for 30 s, producing films of approximately 2–2.5 µm thickness. Annealing at 150 °C for 30 min reduced the surface roughness from 7.552 to 1.0925 nm for glass and from 2.969 to 0.8180 nm for glass/ITO, indicating improved surface uniformity. Nanoindentation showed comparable mechanical responses among the samples, with a localized deformation event observed in the indentation curves. However, its exact mechanism cannot be conclusively established from the present measurements. Therefore, future investigations involving different indentation depths and loading rates, together with post-indentation AFM/SEM and structural characterization, are required to clarify the deformation mechanism and further understand the interfacial and nanoscale properties of the ITO/PTB7-Th:PCBM system.

Author Contributions (Mandatory)

Muhammad Riaz: Conceptualization, Methodology, Validation, Supervision, Project Administration, Writing, Review & Editing. **Iqra Munir:** Investigation, Visualization, Writing, Original Draft. **Nida:** Investigation, Data Curation, Validation. **Noor Ul Huda:** Investigation, Resources, Visualization. **Rabia Shahzada:** Investigation, Data Curation, Writing, Review & Editing.

Acknowledgements

'The authors are grateful to the GIK Institute of Engineering Sciences and Technology, Topi, Swabi, Khyber Pakhtunkhwa 23640 for supporting to this work and providing the necessary research facilities. In particular, the authors are thankful to Tauheed Shehbaz and Muhammad Sohail for his guidance and help at conducting experiments by use of the nanoindentor and AFM respectively.'

Conflicts of Interest Statement

The authors declare no conflict of interest

Funding Information

'This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.'

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