

Enhancing Stability of Few-Layer Black Phosphorus via Nitrocellulose Encapsulation for Optoelectronic Applications

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Summary:

Few-layer black phosphorus (FLBP) degrades rapidly in ambient conditions, hindering its practical applications. We demonstrate that a nitrocellulose encapsulation film enables real-time optical monitoring of FLBP while protecting it from degradation. Optical transmission measurements performed over 3 hours confirm the stability of encapsulated samples compared to unprotected references. This approach enables the integration of FLBP in photonic devices under ambient conditions.

Keywords: black phosphorus, nitrocellulose encapsulation, optical stability, degradation, fiber-optic interferometer

Introduction

Few-layer black phosphorus (FLBP) is a promising 2D material with strong potential for optoelectronic applications due to its anisotropic optical response and broadband absorption [1,]. However, its practical implementation is hindered by rapid degradation under ambient conditions, where oxygen and moisture induce chemical breakdown and surface roughening [2].

To address this limitation, we propose an encapsulation strategy based on nitrocellulose protective films. These transparent films not only shield FLBP from air and humidity but also enable real-time optical measurements to be performed directly on the protected material. The primary aim of this study is to evaluate the efficiency of this encapsulation in maintaining optical stability over time and to explore its feasibility for use in practical photonic platforms.

FLBP-nitrocellulose film preparation

FLBP was prepared by liquid-phase exfoliation of black phosphorus crystals (Smart Elements). 38 mg of ground BP was dispersed in 8 mL of argon-purged 95% ethanol (Sigma-Aldrich) and next the mixture was ultrasonicated using a Bandelin Sonopuls HD2200 (20 kHz, 40 W, 0.5 s ON/OFF pulses) for 4 hours at 0–3 °C under continuous argon flow. This method followed established literature protocols [3]. To fabricate the FLBP-nitrocellulose film, 5 μ L of FLBP suspension was drop-cast onto interdigital electrodes five times in an argon-filled glove box, with 30-minute drying intervals at room temperature. A

protective nitrocellulose layer was then applied from an ethanol solution. The composite film was dried under argon for 24 hours, then under reduced pressure for 2 hours.

Optical Measurements

The interference spectra were recorded by the Optical Spectrum Analyzer. A measurement setup consists of a 1320 nm light source, a fiber coupler, a sample, and a spectrum analyzer. The light emitted from the source is coupled into the fiber and directed towards the sample. The resulting signal observed in the analyzer displays an interference spectrum. The optical analysis was conducted on two samples, with spectra being recorded immediately after sample preparation (0 hours), as well as after 1 hour, 2 hours, and 3 hours. By measuring at different time intervals, it becomes possible to observe changes in the optical properties resulting from the degradation process of the samples. In this case significant difference between successive measurement from unprotected samples can be observed. On the other hand, as one can note the optical spectra have not changed after measuring time, which can mean that the process of degradation did not occur and did not influence the optical spectra of the measured signal.

Measurement Results

The optical stability of FLBP was evaluated by monitoring interference spectra over time using a fiber-optic setup and an optical spectrum analyzer. Two sets of samples: one encapsulated with nitrocellulose nitrate and one left unprotected were analyzed immediately after

preparation (0 h), and subsequently at 1 h, 2 h, and 3 h intervals. The results, shown in Fig. 1, reveal a clear difference in the behavior of protected versus unprotected FLBP. For unprotected samples, significant spectral changes occurred within the first hour, including a reduction in fringe visibility and a shift in baseline intensity, both of which indicate rapid degradation of the material due to environmental exposure.

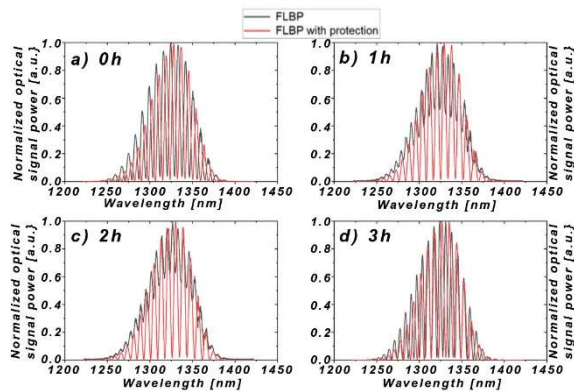


Fig. 1. Optical spectra of the samples of unprotected and protected with nitrocellulose FLBP, after a) 0h b) 1h, c) 2h and d) 3h.

In contrast, the encapsulated samples maintained stable interference patterns over the entire measurement period. No measurable spectral shifts or amplitude variations were detected, suggesting that the nitrocellulose nitrate layer effectively prevents oxidation and moisture-induced damage. This confirms the suitability of the encapsulation for maintaining long-term optical performance of FLBP under ambient conditions.

Simulation results

Parameters for simulation with nitrocellulose setup were as follows, note that this was setup consisting of 3 layers with different refractive index (n) and thickness (l): $n_0=1.47$ (fiber core); $n_1=1$ (air), $l_1=100 \mu\text{m}$ (distance from the sample); $n_2=1.46$ (FLBP), $l_2=5 \cdot 10 \mu\text{m}$ (FLBP thickness); $n_3=2.5$ (nitrocellulose), $l_3=1280 \mu\text{m}$ (nitrocellulose thickness). To compare the results with or without nitrocellulose parameter n_2 was simply set to 1. Fig Y shows compared results for setup with FLBP and FLBP covered with nitrocellulose.

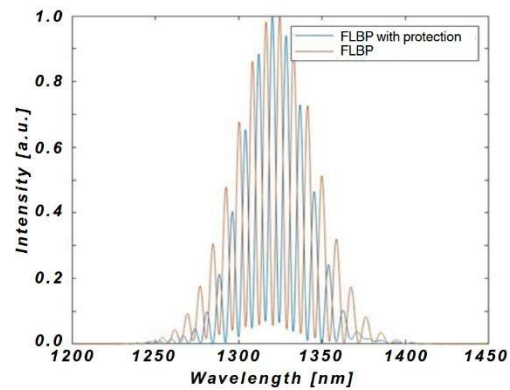


Fig. 2 Simulation results for setup with FLBP and FLBP covered with nitrocellulose (protected).

As can be observed on Fig. Y similar phase shift as on measurement results can be seen. This proves that additional layer of nitrocellulose is responsible for phase shift. This proves that addition of nitrocellulose film did not affect optical properties of FLBP. Nitrocellulose, as demonstrated here, provides a straightforward and effective means to overcome the primary barrier to FLBP's technological adoption, paving the way for its use in advanced optoelectronic and photonic systems.

References

- [1] M. Buscema, D.J. Groenendijk, S.I. Blanter, G.A. Steele, S.J van der Zant, A. Castellanos-Gomez, „Fast and Broadband Photoresponse of Few - Layer Black Phosphorus Field-Effect Transistors“, *Nano Letters* 14, 3347-3352 (2014)
- [2] W. Li, Z. Wang, F. Zhao, M. Li, X. Gao, Y. Zha, J. Wang, J. Zhou, Y. Hu, Q. Xiao, X. Cui, M. J. Es-lamibidgoli, M. H. Eikerling, R. Li, F. Brandys, R. Divigalpitiya, T.-K. Sham, X. Sun*, „Quantification of Nanoscale Phase Evolution by Scanning Transmission X-ray Microscopy“, *Chem. Mater.* 32, 1272-1280 (2020)
- [3] M. Szczerska, M. Kosowska, J. Gierowski, M. Cieřlik, M. Sawczak, P. Jakóbczyk, „Investigation of the Few-Layer Black Phosphorus Degradation by the Photonic Measurements“, *Adv. Materials Inter.* 10, 2202289 (2023)

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