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► **To cite this version:**

Alina Maksimova, Alexander Uvarov, Demid Kirilenko, Artem Baranov, Ekaterina Vyacheslavova, et al.. Trimethylboron as a precursor for boron phosphide plasma deposition at low temperature. *Journal of Vacuum Science & Technology A*, 2024, 43, pp.013402. 10.1116/6.0004065 . hal-04894267

**HAL Id: hal-04894267**

**<https://hal.science/hal-04894267v1>**

Submitted on 17 Jan 2025

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# Trimethylboron as a Precursor for Boron Phosphide Plasma Deposition at Low Temperature

Running title: Trimethylboron as a Precursor for Boron Phosphide Plasma Deposition at Low Temperature

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Boron phosphide (BP) thin films are potential selective contact layers for photovoltaic (PV) devices. However, lower deposition temperatures are preferred in the fabrication of many PV devices, such as solar cells, to reduce their cost. Here, boron phosphide layers were grown on silicon (100) substrates using standard plasma-enhanced chemical vapor deposition (PECVD) and time-modulated PECVD methods with trimethylboron ( $B(CH_3)_3$ , TMB) and phosphine ( $PH_3$ ) precursors. The effect of plasma power and Ar addition on structural properties and chemical composition is investigated, while material properties are analyzed by transmission electron microscopy (TEM). Chemical characterization by the electron diffraction x-ray spectroscopy (EDX) method showed high carbon content in the BP layer. Electron Energy Loss Spectroscopy (EELS) demonstrated almost stoichiometric B and P (1:1) content. Raman spectroscopy of

annealed samples showed an increase in carbon-related peaks, therefore indicating that annealing does not lead to the crystallization of boron phosphide. Thus, using TMB as a precursor of boron leads to carbon contamination in both standard and time-modulated modes. Optical emission spectroscopy (OES) showed that the low-temperature growth of BP without plasma (Ar or phosphine) assistance using TMB is impossible. We conclude that there is need to investigate other boron precursors for boron phosphide low temperature growth to avoid carbon contamination in BP film.

## I. INTRODUCTION

Boron phosphide (BP) is a new material for electronic devices that has attractive characteristics for a wide range of applications, ranging from solar cells to transparent electronics. It is quite chemically inert; it is resistant to oxidation at high temperatures; and it has high thermal conductivity and mechanical stability [1]. Thus, BP has the potential to create devices operating in extreme conditions. For example, it was shown that BP could be used as an efficient solid-state thermal neutron detector [2]. It was also theoretically shown that BP is one of the most promising binary compounds for creating transparent conducting p-type coatings since it is an indirect bandgap semiconductor presenting a large difference between energies of the direct (4 eV) and indirect transitions (2 eV), which implies low optical losses [3]. Therefore, boron phosphide can be used as a p-type transparent conductive material (TCM) for photovoltaic devices. Common transparent conductive oxides (TCOs) such as  $\text{In}_2\text{O}_3$ ,  $\text{SnO}_2$ ,  $\text{ZnO}$ , and  $\text{TiO}_2$  cannot be used as p-type coatings due to their large effective masses and low hole mobility, while the hole mobility of BP can be as high as  $500 \text{ cm}^2/(\text{V}\cdot\text{s})$  at a temperature of 300 K [1]. Another recently proposed approach for selective contact with silicon uses a structure

composed of an ultra-thin silicon oxide layer ( $\text{SiO}_2$ ) and a heavily doped polysilicon layer. The  $\text{SiO}_2$  layer provides good passivation of the surface of c-Si while still allowing charge carriers to pass through it (either by tunneling or through shunt nanocontacts). The highly doped polysilicon layer ensures carrier selectivity; it can be obtained by the deposition of amorphous silicon followed by high-temperature crystallization annealing. The conversion efficiency of such solar cells (SC) is over 26% for small cells and over 25% even for large area cells [4,5]. Nevertheless, such contacts may suffer from low temperature stability and parasitic absorption in the short-wavelength region [6]. Therefore, an intensive search for new materials that could avoid the disadvantages of Si-based layers and transition metal oxides is underway.

Consequently, the use of BP/Si heterojunctions instead of poly-Si/SiOx/Si or (p)a-Si:H/(i)a-Si:H/Si could lead to an increase in the short-circuit current of silicon solar cells. The BP/Si interface exhibits a valence band offset either slightly positive (type I straddling gap heterojunction) or slightly negative (type II staggered gap heterojunction), while having a large conduction band offset, making it an excellent candidate as a selective hole contact without requiring an additional Indium Tin Oxide (ITO) layer [7]. The high cost of indium implies a higher cost of fabrication of solar cells with an ITO layer. Using BP instead of ITO should allow one to reduce the cost of solar cell production.

In previous work [8], the efficiency of the structure with the p-BP/n-Si/n-GaP structure was calculated using AFORS-HET software [9], suggesting the possibility of reaching values up to 28% for the SC photovoltaic conversion efficiency.

The growth of BP on Si can be carried out by different methods: halide vapor-phase epitaxy (HVPE) at the temperature of 1030 °C [10], chemical vapor deposition (CVD) at 800-900 °C [2], organometallic chemical vapor deposition (MOCVD) at 850 °C [11], and radio frequency (RF) reactive magnetron sputtering at 450 °C [12]. Precursors used for these methods are mostly diborane B<sub>2</sub>H<sub>6</sub> and phosphine PH<sub>3</sub>.

Low-temperature processes are preferred during the manufacturing of most solar cells. However, low-temperature synthesis of BP layers is difficult due to the difference in the reactivity of B and P [13]. A plasma deposition approach could potentially solve this problem. In this paper, the growth of boron phosphide thin films using the plasma-enhanced chemical vapor deposition (PECVD) method is proposed. It is a reliable industrial method that allows electronic-quality films to be grown at low temperatures (250 °C) in industrial scale. In addition, the time-modulated approach (or PE-ALD like, pseudo PE-ALD), in which the B and P precursors are decomposed with RF plasma, is considered. Here we use phosphine (PH<sub>3</sub>) as a precursor of phosphorus and propose trimethylboron (B(CH<sub>3</sub>)<sub>3</sub>, TMB) as a precursor of boron, which is preferable compared to B<sub>2</sub>H<sub>6</sub> for safety reasons. The composition and structure properties of the layers obtained using TMB will be studied.

Another issue that should be considered for photovoltaic applications is the passivation of the interface. The deposition of an ultra-thin amorphous hydrogenated silicon (a-Si:H) layer before the deposition of selective contact is known to be a way to achieve high-quality passivation of the Si surface. This approach was successfully applied for selective contacts based on Si and transition metal oxides [14]. Promising results in terms of passivation quality were obtained for the BP/Si interface with a thin

intermediate a-Si:H layer [15]. However, the structural properties of the BP layer grown on a-Si:H have not been studied yet.

## II. EXPERIMENTAL

The growth of BP films was carried out in a standard Oxford PlasmaLab 100 PECVD (13.56 MHz) plasma chemical deposition unit using capacitive-coupled RF plasma with precursor flow control and temperature control on a heating table. Gas mixtures of hydrogen with TMB and phosphine were used as precursors, with gas ratios (TMB)/H<sub>2</sub> and PH<sub>3</sub>/H<sub>2</sub> of 2% and 5%, respectively. BP layers were deposited on n-type double-side polished (100) c-Si wafers produced by the Czochralski method with a thickness of 380 μm in two modes: standard PECVD and time-modulated PECVD. Immediately before loading into the working chamber, substrates were treated in a 10% HF/H<sub>2</sub>O solution for 30 s to remove the native oxide.

In the time-modulated PECVD mode, the first step is the decomposition of the TMB/H<sub>2</sub> mixture using the addition of Ar, which is necessary to ensure the guaranteed ignition of the plasma with a power of 100 W. It was previously noticed that without Ar addition, the growth rate is extremely slow and deposition is not possible, as well as without plasma on that step. The second step of the process is the decomposition of PH<sub>3</sub> at an RF plasma power of 200 W. With the process consisting of 200 cycles, a total layer thickness of about 60 nm is obtained. Deposition parameters and details of the process sequence are described in our previous work [15].

The a-Si:H layer was deposited on the Si surface before BP in the standard PECVD mode at a temperature of 250 °C with a plasma power of 5 W during 40 sec from pure SiH<sub>4</sub> at a constant pressure of 350 mTorr. In the standard PECVD mode, the

BP layer is deposited on the a-Si:H layer at a temperature of 250 °C with a plasma power of 30 W and a pressure of 350 mTorr.

For all samples, layer thicknesses were determined using a Horiba PZ2000 laser ellipsometer with a 632.8 nm He-Ne source. BP film deposition properties such as temperature, number of cycles/deposition time, plasma power, and thicknesses of layers are presented in Table 1.

Table 1. Properties of BP film deposition by PE-ALD and PECVD methods

	Time-modulated PECVD		Standard PECVD	
	BP		BP	a-Si:H
T(°C)	250	T(°C)	250	250
Cycles	200	t(sec)	180	40
Pressure(mTorr)	350	Pressure(mTorr)	350	350
RF power(W)	200	RF power(W)	30	5
Growth per cycle(nm/cycle)	0.3	Growth rate(nm/sec)	0.11	0.18
Thickness(nm)	60	Thickness(nm)	20	7

The structural properties and surface morphology of the BP layer deposited on the silicon substrate were studied by means of transmission electron microscopy (TEM) using the Jeol JEM-2100F set-up with a 200 kV acceleration voltage (point resolution of 0.19 nm). Cross-section specimens for TEM were prepared by a conventional route involving mechanical grinding with subsequent ion milling by Ar<sup>+</sup> at 2-4 kV. Energy dispersive X-ray spectroscopy (EDX) and electron energy loss spectroscopy (EELS) implemented in the TEM were used for the BP layer composition evaluation. Scanning transmission electron microscopy (STEM) mode was used for measuring element distribution maps.

Compared to EDX, EELS provides increased sensitivity to the lower atomic number elements, making it an important tool for determining boron and carbon concentration. EELS spectra were acquired in the image mode and an entrance aperture



of 1 mm was installed. The element ratio was determined from the number of counts in a chosen energy range of the core-excitation edge using the conventional EELS Quantification plug-in for Gatan Digital Micrograph software. Three elements were selected for quantification: boron, carbon, and phosphorous; all the elements have corresponding core-loss edges in the 100-400 eV range, so that 0.3 eV/channel dispersion was enough. Power-law background extrapolation was implemented, convergence angle was set as 9 mrad (almost focused beam @ Alpha = 2, Spot = 2, "TEM" beam mode for Jeol 2100F TEM), and collection angle with the chosen objective aperture was estimated as 10 mrad. The results of quantification are presented in atomic percents of relative content, that is, in the assumption that the sum for all elements is 100%.

Rapid temperature annealing (RTA) was performed using Jipelec JetFirst 100 in ambient N<sub>2</sub> environment at temperatures of 500–800 °C. Raman spectroscopy of annealed samples was performed using an ENSPECTR R532 Raman spectrometer with a 532 nm laser wavelength and a holographic grating of 1800 lines/mm. The measurements were carried out with an optimal signal integration time of 3000 ms and averaging equal to 100 to reduce the influence of the noise of the CCD sensor.

The optical emission spectra of TMB/H<sub>2</sub> plasma and PECVD processes with and without Ar addition with a power of 5-200 W during deposition were measured by the Avantes AvaSpec-ULS2048XL EVO spectrometer in the range of 300–700 nm.

### III. RESULTS AND DISCUSSION

#### A. *Structural properties*



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TEM images of time-modulated PECVD BP samples (Fig. 1a) demonstrate the amorphous structure of the film compared to the clearly distinguishable crystalline state of the Si substrate. The BP layer has a smooth surface and a homogeneous structure with no visible defects. Heterointerfaces between Si substrate and BP layer grown either by PECVD or PE-ALD are sharp and smooth (insets of Figs. 1a, b). No initial epitaxy or intermediate layer is observed. A thin layer (10 nm) is observed on the top of the BP films, which seems to be related to oxidation.

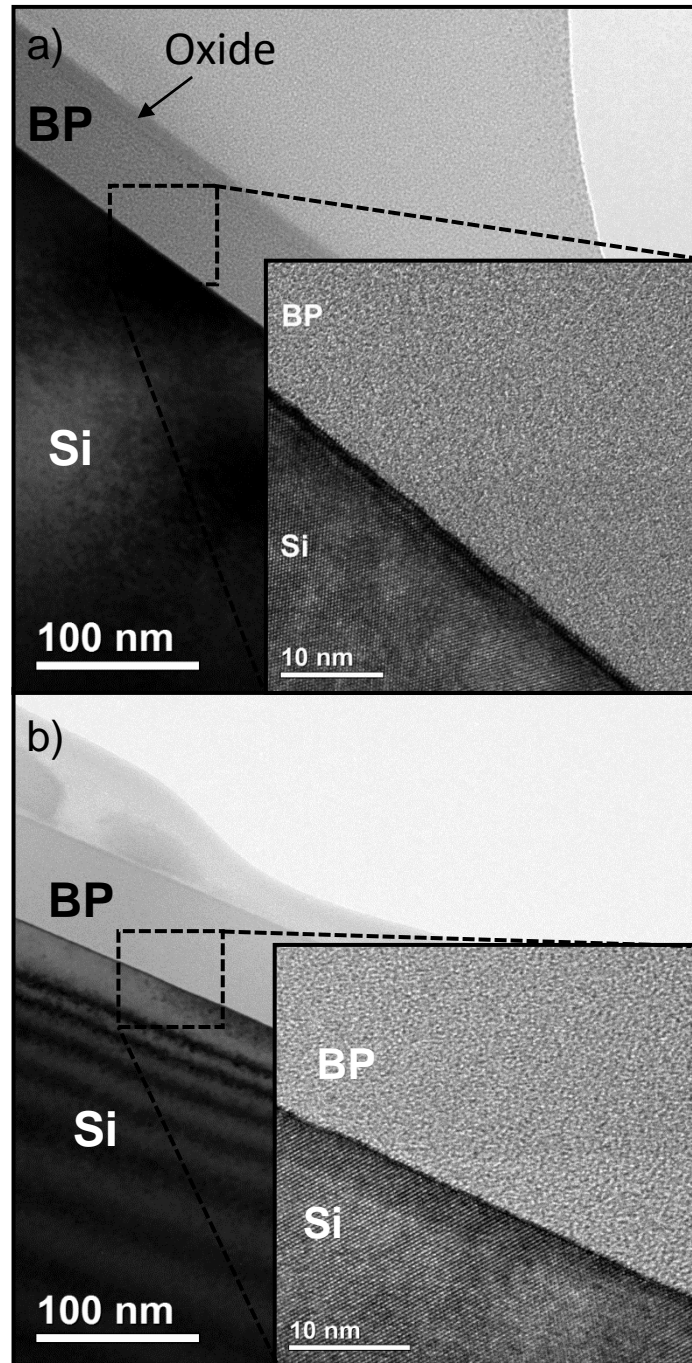


FIG. 1. TEM and HRTEM images of BP/Si interfaces grown by a) time-modulated PECVD at 250 °C; b) standard PECVD at 250 °C.

The BP layer grown by standard PECVD on the a-Si:H interlayer (Fig. 2) exhibits similar structural properties as the one grown without an interlayer. The BP film is

amorphous and uniform with a smooth interface between a-Si:H and BP, while it is hard to distinguish the interface between two amorphous layers in the high-resolution TEM image (inset of Fig. 2). On the contrary, the heterointerface between the Si substrate and a-Si:H has a rough surface due to the partial epitaxial growth of silicon at the initial stage of a-Si:H deposition. Such initial epitaxial growth of Si at 250 °C is commonly observed for a-Si:H deposition on (100) Si wafers [16]. Formation of nano-size crystallites could also be expected for BP layers, while it was not observed for the PECVD process performed even at 380 °C. To analyze the reason for crystallization inhibition, the composition of the BP layers was studied using EDX.

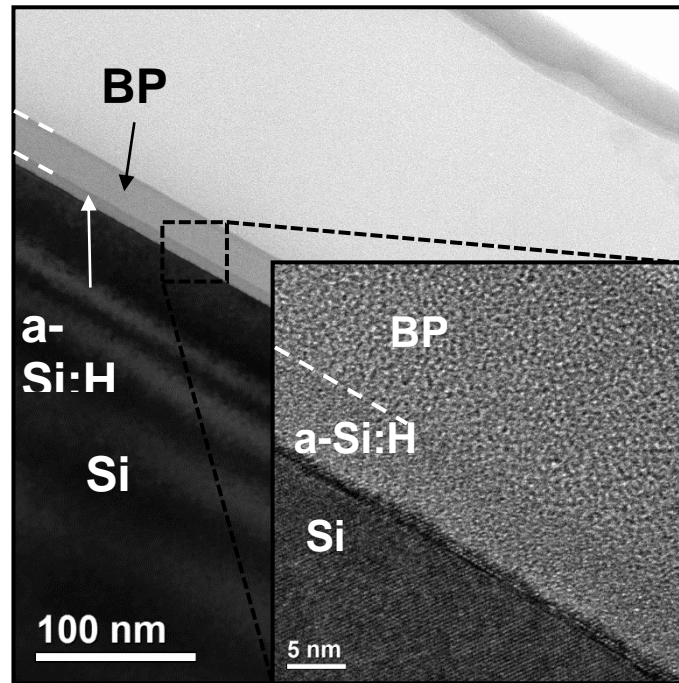


FIG. 2. TEM and HRTEM images of the BP/Si interface with a thin a-Si:H interlayer. Both BP and a-Si:H were grown by standard PECVD at 250 °C.

### **B. Composition properties**

Typical energy-dispersive X-ray (EDX) spectra measured at the top and base of the BP film, which are similar for all samples, are presented in Figure 3. The EDX spectra contain peaks of low and high intensity at 0.183 and 2.01 keV, which correspond to  $K\alpha$  lines of the characteristic emission for boron and phosphorus, respectively. Due to the small thicknesses of layers and the low sensitivity of EDX to low-mass B atoms, a precise determination of the stoichiometry is difficult. The EDX spectrum from the top layer exhibits peaks of carbon and oxygen as well as peaks of Ca, Cu, and S (Fig. 3b). Obviously, those are mostly related to epoxy and surface contamination. However, in the spectra from the base layer, a carbon peak could still be detected (Fig. 3 a), while other peaks disappeared, including that of O. Analysis of the element distribution maps presented in Figure 3 allows one to clarify the composition.

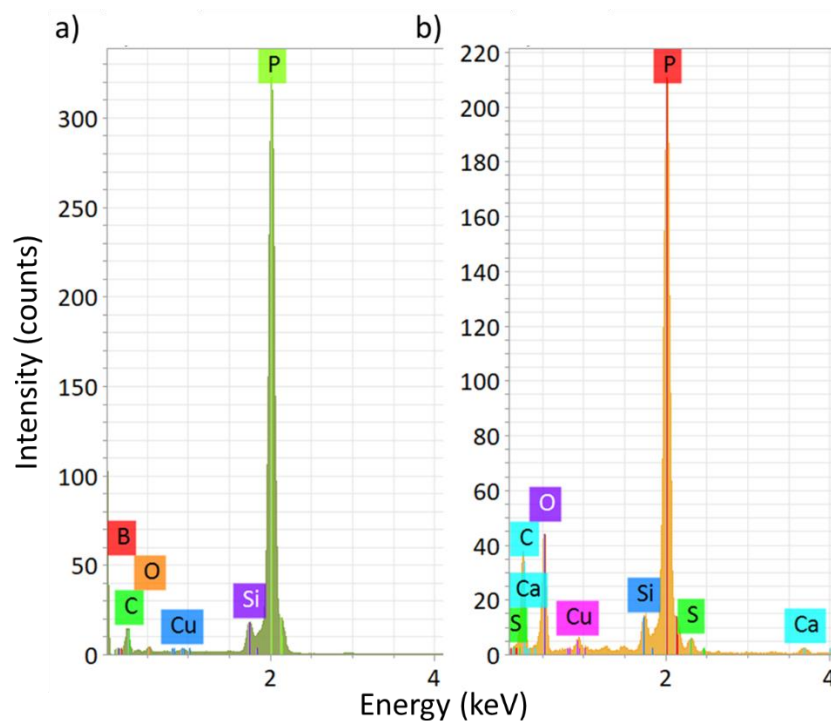


FIG. 3. EDX spectra of a BP sample grown by time-modulated PECVD at 250 °C, a) from the base layer; b) from the top layer.

It can be clearly seen on EDX distribution maps (fig 4) that the BP layer has high carbon contamination evenly distributed across the film depth, however the concentration of C in the film is less than in the sample holder used for sample attachment to the TEM setup. The EDX map also clearly reveals that the thin top layer detected by TEM images is an oxide. The calcium and iron detected at the surface of the film is caused by contamination. We should note that phosphorus is homogeneously distributed within the layer thickness. Boron content was detected in epoxy as well as in the BP layer, which is caused by low sensitivity of this method to boron.

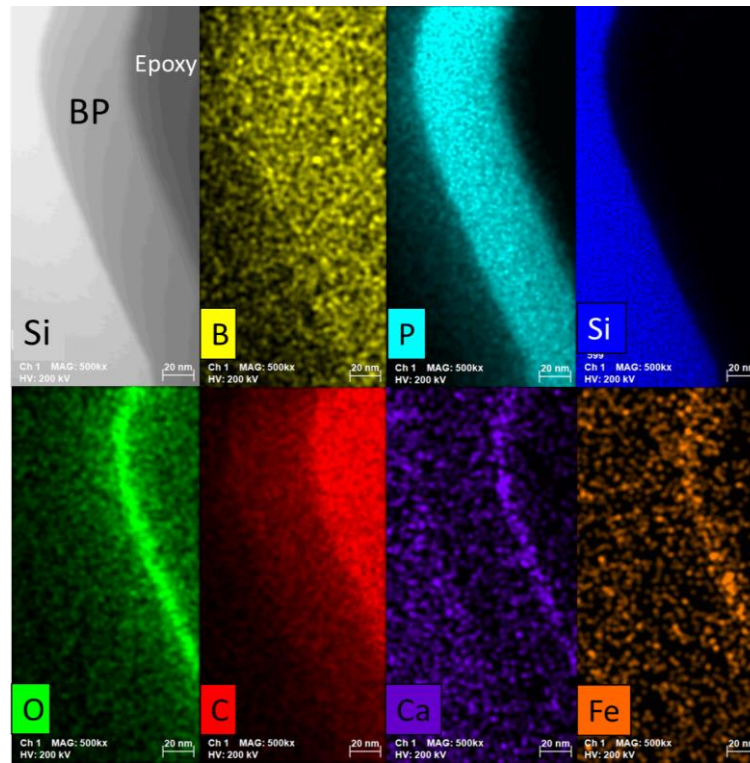


FIG. 4. Energy dispersive X-ray spectra distribution maps of elements of the BP/Si structure grown by time-modulated PECVD at 250 °C.

Detection of boron may be difficult with EDX due to its closeness to the H line; therefore, it is necessary to use a method with high sensitivity to low-atomic-number elements, such as electron energy loss spectroscopy. On the EELS spectra, a clearly



distinguishable boron peak was detected at 189 eV (Fig. 5), which proves the successful growth of a boron phosphide film. In addition, the phosphorus K absorption edge at 132 eV and the carbon K edge at 284 eV were detected. Relative quantification showed almost stoichiometric B and P (1:1) content within 10% accuracy of EELS measurement, while an extremely high carbon content of about 25% was observed.

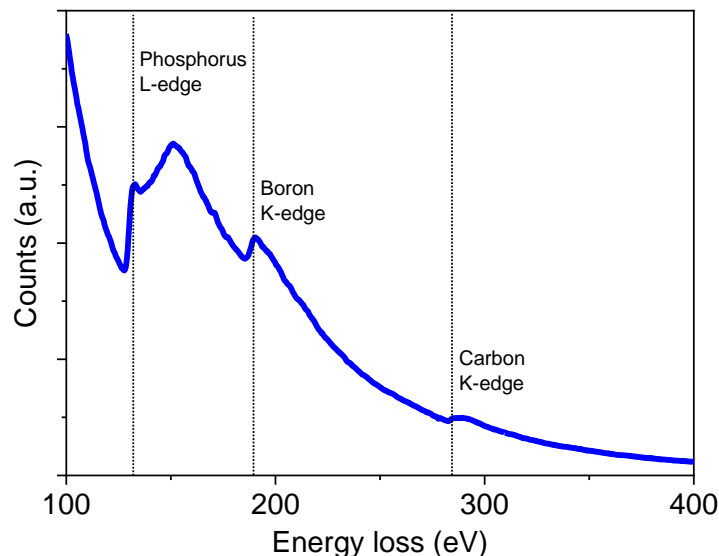


FIG. 5. Electron Energy Loss Spectra of BP/Si structure grown by time-modulated PECVD at 250 °C.

### C. Rapid Thermal Annealing

The high-detected carbon concentration detected in the films from EDX/EELS could inhibit the crystallization process of BP. To check this assumption, rapid temperature annealing (RTA) was performed at temperatures of 500, 600, 700, and 800 °C and the results were analyzed by Raman spectroscopy. The Raman spectrum of the sample before any annealing treatment exhibits no peaks (Fig. 6), which is probably due to its amorphous state [17]. Raman spectra of the sample after RTA show peaks at 1581, 1349, and 2646  $\text{cm}^{-1}$  related to carbon, denoted as the G band and D bands. These bands

correspond to the degenerate in-plane E<sub>2g</sub> optical mode at the center of the Brillouin zone and to the harmonic (second-order Raman scattering) of an in-plane transverse optical (TO) mode close to the zone boundary K point, respectively [18]. The intensity of peaks is increasing with the increase in annealing temperature up to 700 °C, indicating a high carbon concentration in the grown films. However, no peak related to the BP crystalline phase at 823 cm<sup>-1</sup> was detected after annealing. Thus, no crystallization of boron phosphide occurs even after annealing at 800 °C due to extremely high carbon contamination during the deposition process.

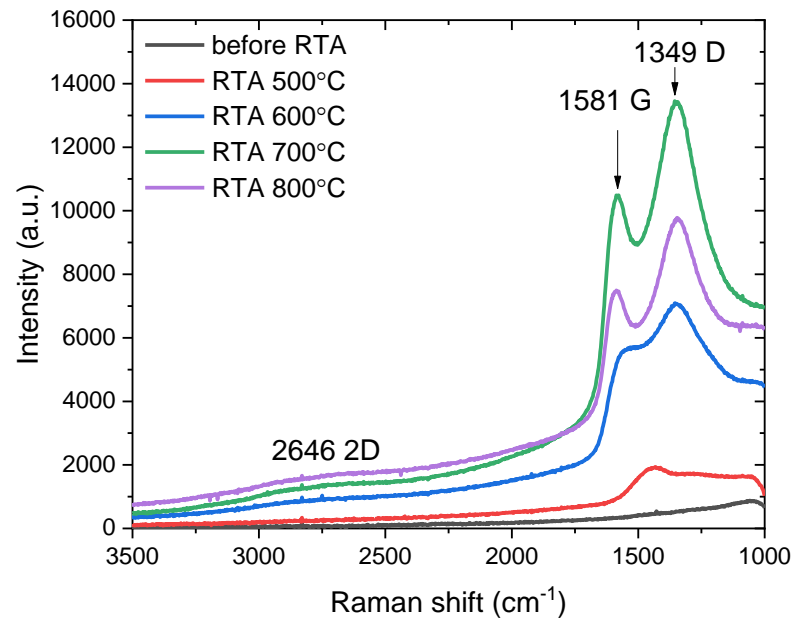


FIG. 6. Raman spectra after rapid temperature annealing (RTA) of BP grown by standard PECVD at 250 °C.

#### **D. Decomposition of TMB**

The origin of carbon contamination is evidently related to the TMB precursor, which contains CH<sub>3</sub> groups. However, we should stress that for the III-V growth of MOVPE, ALD, and PE-ALD using methyl-containing precursors (trimethylgallium,

trimethylindium, and trimethylaluminium), the problem of strong C contamination has never appeared. The TMB decomposition process was studied using optical emission spectroscopy (OES). The optical emission spectra of TMB steps for the time-modulated PECVD process are presented in figures 7 a, b, while the spectra for the standard PECVD process are shown in figures 7 c, d. The signal intensities in the OES spectra are increasing with the increase in plasma power. Main hydrogen atomic lines such as Ha-e are detected and atomic and molecular lines of carbon are detected as well for both spectra with and without Ar addition. Apparently, the methyl groups of TMB are decomposing into hydrogen and carbon in both cases, leading to carbon incorporation into the growing film. The C2 and C3 carbon clusters are a likely source for the formation of carbon phases in the films, which is detected by Raman measurements [19].

For the spectra of TMB/H<sub>2</sub> plasma with Ar addition, a line corresponding to the BH bond could be clearly detected, while its intensity is much lower for the spectra of pure TMB/H<sub>2</sub> plasma. Thus, Ar addition increases the BH bond intensity. The TMB molecule decomposes by dehydrogenation of methyl groups, likely assisted by excited Ar atoms in the plasma, followed by breakage of B–C bonds to form CH radicals. It seems to be a key issue in explaining the observed effect when Ar addition leads to a significant growth rate increase.



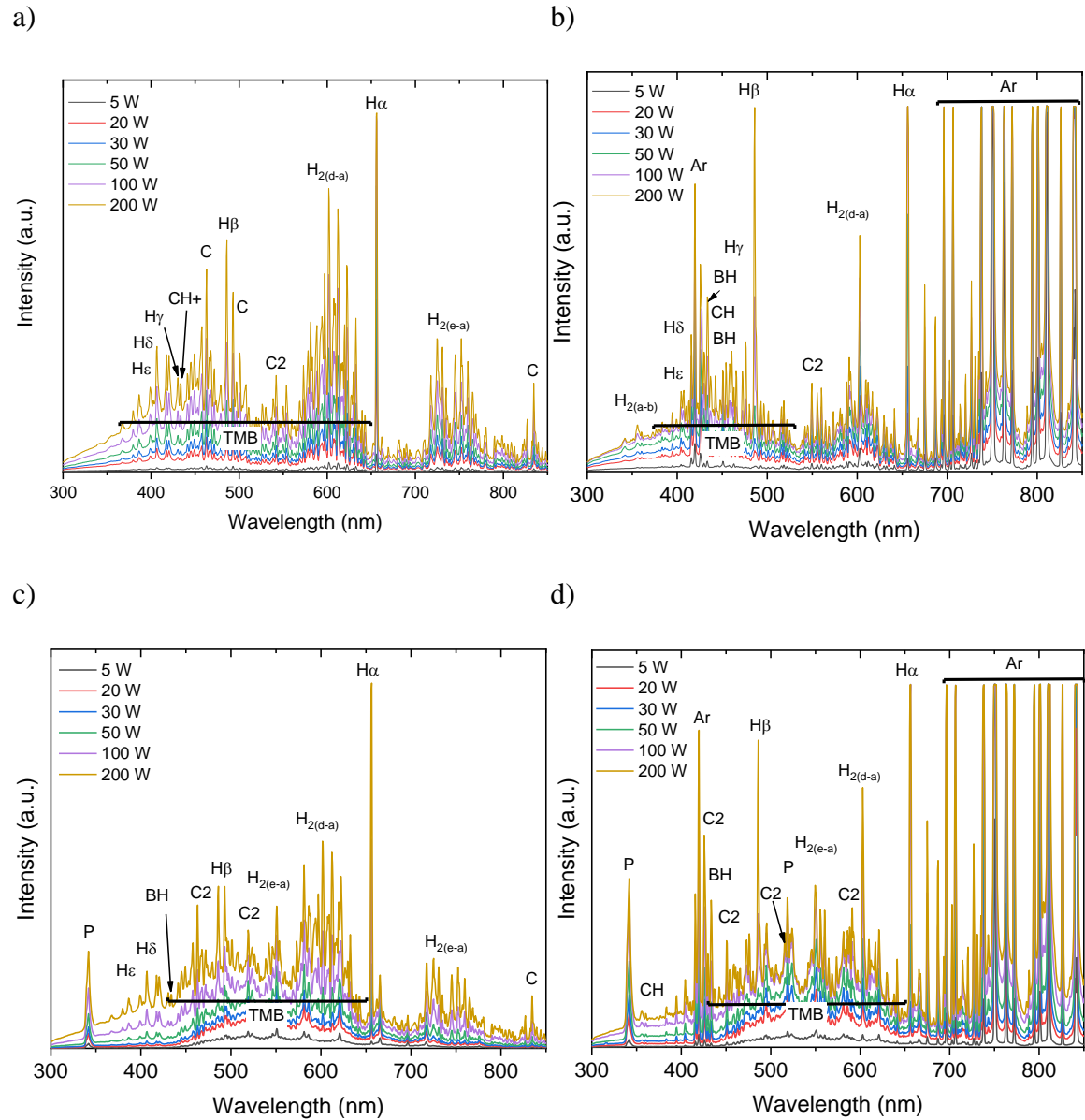


FIG. 7. OES spectra of a) TMB/H<sub>2</sub> step of time-modulated PECVD process; b) TMB/H<sub>2</sub> step of time-modulated PECVD process with Ar addition; c) standard PECVD process (TMB+PH<sub>3</sub>); d) standard PECVD process (TMB+PH<sub>3</sub>) with Ar addition.

#### IV. SUMMARY AND CONCLUSIONS

The peculiarities of growing thin layers of boron phosphide using the method of plasma-chemical deposition in both standard PECVD and time-modulated PECVD

modes with trimethylboron (TMB) and phosphine (PH<sub>3</sub>) precursors on Si substrates at the temperature of 250 °C were shown. TEM indicates the amorphous structure of the films, while EDX reveals a high carbon content. EELS proves the presence of boron in the film. According to Raman spectroscopy, thermal annealing up to 800 °C does not lead to crystallization of boron phosphide. Moreover, Raman spectroscopy of annealed samples emphasizes carbon peaks indicating its high concentration in agreement with the EDX data. OES spectra showed that Ar addition is required to provide better decomposition of TMB. Thus, TMB is not suitable as a precursor for BP growth due to the high carbon contamination in the grown BP film. Plasma deposition of BP films using diborane as the boron precursor will be explored in the future.

## ACKNOWLEDGMENTS

The reported study was supported by Ministry of Science and Higher Education of the Russian Federation (research project 0791-2023-0007). TEM studies were performed using equipment from the Federal Joint Research Center “Material Science and Characterization in Advanced Technology,” supported by the Ministry of Science and Higher Education of the Russian Federation.

## AUTHOR DECLARATIONS

### **Conflicts of Interest**

The authors have no conflicts to disclose.

## DATA AVAILABILITY

The data that supports the findings of this study are available within the article.

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PLEASE CITE THIS ARTICLE AS DOI: 10.1116/6.0004065

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