All-BN distributed Bragg reflectors fabricated in a single MOCVD process

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Abstract
Distributed Bragg Reflectors (DBR) are well-established photonic structures that are used in many photonic applications. However, most of the DBRs are based on different materials or require post-process etching which can hinder integration with other components in the final photonic structure. Here, we demonstrate the fabrication of DBR structures consisting only of undoped boron nitride (BN) layers with high refractive index contrast by using metal–organic chemical vapor deposition (MOCVD). This has been achieved in a single process, without the need for any post-process etching. The difference in the refractive index of the component BN layers stems from different degrees of porosity of the individual BN layers, which is a direct result of a different growth temperature. The fabricated DBR structures consist of 15.5 pairs of BN layers and exhibit a reflectance of 87 ± 1% at the maximum. The wavelength of maximum reflectance can be tuned from 500 nm up to the infrared region (IR), by simply adjusting the growth periods of subsequent BN layers. We also demonstrate that the fabricated structures can be used to create an optical microcavity. The fabricated DBRs are very promising candidates for future applications, for example in combination with single-photon emitters in h-BN, which could allow the building of a cavity-based all-BN single-photon source.

Supplementary material for this article is available online

Keywords: hexagonal boron nitride, Bragg mirror, porosity, DBR, MOVPE, hBN, in situ reflectance

1. Introduction

Multiple thin films with significant refractive index contrast may be stacked on top of each other to create a distributed Bragg reflector (DBR)—a one-dimensional photonic crystal with a photonic bandgap in the direction perpendicular to the layer planes. Such structures exhibit reflectance values approaching 100% for photon energies within the bandgap. Depending on the refractive index values of the incorporated thin films, their thicknesses and the number of layer pairs, the position and width of this maximum can be controlled [1]. DBRs find extensive use primarily in vertical-cavity surface-emitting lasers (VCSEL) [2–6], resonant-cavity light-emitting diodes (RCLED) [7, 8], but also in photovoltaics [9, 10] and diffractive optical components [11]. Metal–organic chemical vapor deposition (MOCVD) is already an established growth method for DBR fabrication, as the first reports date back to the mid-80s [12, 13]. However, DBR structures based on boron compounds have been explored only recently [14, 15].

Boron nitride (BN) crystals, and in particular those which exhibit sp² electronic orbital hybridization—hexagonal and rhombohedral BN are promising candidates to use in thin-film applications, in flexible electronics [16], as substrates for other 2D materials [17, 18], or as single-photon sources [19–21]. These BN polymorphs exhibit a wide optical
bandgap of approximately 6 eV [22, 23]. High-quality $sp^2$-type BN layers are commonly grown using the MOCVD technique with trimethylborane (TMB) or triethylborane (TEB) as the boron source and ammonia as the nitrogen source [24–27]. Recently, flow modulation epitaxy of such films is becoming more common [28]. To investigate many of their properties, it is necessary to insert them into the microcavity of the DBR structures in order to increase the number of photons interacting with the layer. This requires delamination of the $sp^2$-type BN layers from the original substrate onto the DBR. Although there have been successful attempts at delamination of hBN films [29], the process is still far from perfect and induces changes to the fabricated material.

The most widely used DBRs are based on different materials that provide a large refractive index contrast. However, such an approach can have drawbacks in terms of integration with other materials in the final photonic structure, for instance due to the inherent difference in thermal expansion of the different materials, or the requirement of lattice matching for epitaxial structures. Another approach to obtain the needed reflective index contrast is to use porous structures. However, such structures require a selective post-fabrication etching process [30–35].

In this work, we present a different approach. We implemented a low-temperature, low-pressure growth regime [36] to fabricate DBR structures entirely based on BN layers with high reflective index contrast. We achieve this in a single process by only changing the growth temperature of the BN layers. We also show that the fabricated DBRs may easily be used to create a microcavity. This gives hope for future growth of all-BN single-photon sources, where the active area made of hBN is grown directly on top of a BN-based DBR and covered with another BN-based DBR in a single MOCVD process.

2. Materials and methods

Boron nitride layers were obtained by MOCVD using an Aixtron CCS 3 × 2” reactor. Growth was performed on single-side polished, 430 μm thick, two-inch sapphire c-plane wafers (with up to 0.2° misorientation) as substrates, except for the sample discussed in section 3.3, where a 330 μm thick double-side polished substrate was used. Ammonia and triethylborane (TEB) were used as precursors for nitrogen and boron, respectively. The carrier gas was set to hydrogen for most samples, but for selected samples, nitrogen was used. We note that in the text where it is the case, the system temperature was controlled via the ARGUS multipoint pyrometer. Since these two measurement techniques produce slightly different results, the values from the ARGUS pyrometer will be reported as the growth temperature. The system is also equipped with a monochromatic optical reflectometer, capable of measuring normal reflectance from the sample’s surface at the wavelength of 635 nm. This allows to estimate the thickness and refractive index (at 635 nm) of the fabricated BN layers.

All of the BN layers have been fabricated at a chamber pressure of 100 mbar, TEB flow of 80 ccm liters per minute and ammonia flow of 100 ccm. The only free parameter was the system temperature. In the case of the DBR samples, where two types of BN layers were grown at different temperatures, the flow of precursor gases was interrupted as the system was ramping or cooling to the desired temperature.

Ellipsometric measurements were carried out with the use of the Woollam RC2 dual rotating compensator ellipsometer with a vertical auto angle stage. This architecture allows for the measurement of the full Mueller Matrix as well as the depolarization factor. The samples were prepared on roughened sapphire substrates that ensure a lack of depolarization due to reflection from the bottom side of the substrate and simplify ellipsometric analysis since in this case, the measurement is sensitive only to the in-plane component of the substrate’s refractive index. Samples were measured in reflection mode in the wavelength range from 190 to 1700 nm for several angles of incidence in the range from 40° to 75° to improve precision. The warmed and calibrated system allows for Psi and Delta measurements with the following accuracy: Psi $+/- 0.02°$, Delta $+/- 0.05°$, and % depolarization $+/- 0.5%$. However one has to bear in mind that ellipsometry is an indirect method and the accuracy of the extracted parameters like complex refractive index or thickness of the sample mainly depends on the developed model and its fitting to the ellipsometric data. Modeling and fittings were performed with the use of CompleteEASE v6.61 software. The optical model took into account the semi-infinite sapphire substrate of refractive index measured prior to the main experiment, and the BN layer was parametrized with a generalized oscillator approach with the use of one or more Tauc-Lorentz and Lorentz oscillator models [37]. To retrieve optical constants of the BN layers multisample analysis was carried out where the fitting of the model was done to several samples simultaneously while geometrical parameters like thickness or roughness were extracted for subsequent samples. A detailed discussion of the modeling procedure and extraction of the uncertainty of optical constants can be found in a recent paper [38] and its supporting information.

The normal reflectance spectra were recorded using Ocean Optics HR-4000CG UV-NIR Spectrometer with a mercury lamp as a light source. Light from the mercury lamp was injected into an optical fiber and then beamsplitted. One of the beams was then reflected from the measured samples surface, then interfered with the non-reflected beam and then led into the detector. Each time before measurements were performed, the spectrometer was calibrated using two samples: one with a close to zero normal reflectance and another with a known, high reflectance value (e.g. a silver or gold film). All reflectance measurements made after removing the sample from the reactor chamber have been performed using this method. The presented spectra are an average result over 25 acquisitions.

AFM measurements were performed in the tapping mode using a Multimode AFM (MMAFM-2) with a Nanoscope IIIa controller from Bruker (formerly Digital Instruments). The
probes used were NT-MDT NSG30 with a guaranteed tip radius of 10 nm.

Specimens for cross-sectional transmission electron microscopy (TEM) measurements were prepared using a standard method of mechanical pre-thinning followed by Ar ion milling at 4 kV and 500 V acceleration voltage of Ar ions. TEM characterization was performed using an image-corrected Titan Cubed 80–300 microscope operating at 300 kV. The scanning transmission electron microscopy was performed using an annular dark-field (HAADF) Fischione 3000 detector. The elemental composition determination was performed by energy-dispersive x-ray spectroscopy (EDX) using EDAX 30 mm2 Si(Li) detector with a collection angle of 0.13 sr.

Numerical modeling of the optical parameters of the fabricated structures was performed using T-matrix formulation of the transfer matrix method (TMM) implemented as MATLAB code, similarly as in other works [39]. In the calculations, the refractive index values for sapphire were taken from the literature [40]. Effective Medium Approximation calculations have been performed using the Bruggeman approach [41]. A detailed description can be found in the supplementary material.

3. Results and discussion

3.1. Growing BN layers with high refractive index contrast

We have fabricated two kinds of BN layers to serve as component layers for the distributed Bragg reflector. One of them has been grown at 640 °C system temperature, the other at 820 °C. We kept all other growth parameters constant. Figure 1 shows the in situ evolution of the sample reflectance at 635 nm, during the deposition of BN layers using hydrogen as the carrier gas, grown at 640 °C (figure 1(a)) and 820 °C (figure 1(b)). For the layer grown at the lower temperature, the reflectance increases at first, reaching a maximum of 11.5%, then drops to the level of pure sapphire. For the layer grown at the higher temperature, it is the reverse—at first, the reflectance decreases, reaches 5.9%, then increases to the level of pure sapphire. This means that both of the fabricated BN layers are transparent at a wavelength of 635 nm. However, since we observed opposite extrema for these two samples, it means that one of them has a higher refractive index than sapphire, and the other one has lower. A simple TMM model of a single BN layer on a sapphire substrate allows to plot the reflectance as a function of layer thickness for a given refractive index value. This is represented by blue curves in figure 1. Best fits to the in situ measurement were achieved assuming the refractive indices of the grown BN layers are 1.890 and 1.702 for the samples grown at 640 °C and 820 °C, respectively. The small discrepancies between the measured and simulated curves result most likely from the slightly non-uniform growth rate of the BN layers. Apart from the refractive index values, the same calculation allows to determine the thickness of the fabricated BN layers. The full Fabry–Perot cycles for refractive indices of 1.890 and 1.702 should occur at 168 nm and 186.5 nm, respectively. From our simulations, we get thickness \( d = 167 \) nm for the sample grown at 640 °C and \( d = 181 \) nm for the sample grown at 820 °C. This means, that for both layers, a nearly full Fabry–Perot cycle has occurred.

More information about the optical properties of the samples can be acquired using spectroscopic ellipsometry. Optical constants were extracted from ellipsometric azimuths \( \Psi \) and \( \Delta \) measured in the experiment with the use of the model described in the Materials and Methods section. Since the measurement is performed in a broad range of wavelengths, we can determine the refractive index dispersion of the investigated layers. This is presented in figure 2. At 635 nm, the refractive index values of the fabricated layers determined using spectroscopic ellipsometry are in good agreement with the values calculated from the in situ reflectance profile—discrepancies do not exceed 0.7%. For both BN layers, the refractive index for wavelengths above 500 nm may be treated as slowly varying, therefore the refractive...
index contrast is almost constant, at an average of 0.17. This value is on a similar level as the refractive index contrast of porous and non-porous GaN layers used in DBRs [34, 35]. Furthermore, for this wavelength range, the imaginary part of the refractive index is negligible, therefore the layers may be treated as non-absorbing. As such, these layers are suitable for the construction of DBR structures optimized for wavelengths greater than 500 nm. Below that value, the imaginary part of the refractive index becomes significant. In the case of the BN grown at 640 °C, the imaginary part of the refractive index rises rapidly with decreasing wavelength, reaching 0.8 at 200 nm. The main absorption resonance in the permittivity spectrum occurs at wavelengths around 200 nm in the case of the BN layer grown at 640 °C, which is in good agreement with results in [42] for hBN and tBN. In the case of the layer grown at 820 °C, the main absorption resonance occurs for wavelengths below 200 nm, which is indicated by the monotonic behavior of the real part of the permittivity within the whole 200–900 nm wavelength range. Moreover, an additional resonance at 5.28 eV occurs, which was not observed in [42].

The real part of the refractive index of bulk BN is close to 2.3 for visible wavelengths [43]. In the case of both of our BN layers, this value is much smaller. This suggests that both types of layers are at least partially amorphous or perhaps porous, and to a different extent. Figure 3 presents STEM micrographs of both investigated BN layers. The scans show areas of distinctively higher (brighter parts of the graph) and lower (darker parts of the graph) density in both types of BN layers. This indicates that both layers are indeed porous. The lower density areas in the case of the BN layer grown at 640 °C (figure 3(a)) are smaller and more finely distributed than in the case of the BN layer grown at 820 °C (figure 3(b)). This implies that the latter exhibits higher porosity than the former. Since the pores are filled with either vacuum or air (with refractive index close to 1), the layer with higher
porosity should exhibit a lower refractive index, which is exactly what we observe.

3.2. Fabrication and characterization of distributed Bragg reflectors

DBRs with the best performance (i.e. highest and broadest reflectance maximum for a given number of layer pairs) are achieved when the component layers have a thickness \( d = \lambda / 4n \) where \( \lambda \) is the wavelength for which the reflectance maximum has to occur and \( n \) is the real part of refractive index \([1]\). For DBRs component layers with refractive indices of 1.89 and 1.702, and the wavelength of 635 nm, the optimal layer thickness should be 84.0 and 93.3 nm, respectively. However, since the refractive index contrast between the constituent BN layers is so high, small changes in individual layer thickness should result only in the shift of wavelength for which the maximum reflectance is observed. The value of maximum reflectance should not change drastically with the thickness of each type of layer, as long as the position of the maximum is at a wavelength of 500 nm or greater. Detailed simulations regarding this matter can be found in the supplementary material.

We fabricated four DBR structures using two approaches. Samples A and B have been fabricated with constant growth times of individual BN layers to ensure similar thicknesses among BN layers grown at the same temperature. In the case of samples C and D, growth times have been manually adjusted during the MOCVD process, so that switching between the growth of high and low refractive index BN layers occurs exactly at the reflectance extremum for 635 nm, thus ensuring that the maximum reflectance will be reached at this particular wavelength. Variable process parameters for all DBRs are specified in Table 1. For each DBR, we have chosen to fabricate 15.5 layer pairs to achieve a reflectance peak in the vicinity of 90% with a clearly defined maximum (see figure F1 in the supplementary material for more details). Schematically, such a structure is presented in Figure 4(a). Figure 4(b) presents an AFM amplitude image from a cross-section of the DBR A. A clear layered structure of the sample is observed. This proves that we have indeed fabricated DBR-like layered structures. The pseudocolor contrast between the two types of BN layers on the AFM scan results from the fact, that in the case of the more porous BN layers, the AFM tip would make a deeper recess than in the case of a less porous layer, before the same force acts upon it. It is also worth noting, that for improved visibility of the layer borders, the AFM scan was conducted at a slight angle with respect to the cross-section.

Figure 5(a) presents the in situ reflectance evolution of the growing DBRs A and C. In the case of DBR A, for which the growth times of each individual BN layer have been fixed, not much information can be extracted from this graph—during the growth of the DBR, it is unclear what is the wavelength for which the reflectance maximum will occur. In the case of DBR C, manual control of the growth times allowed for switching between growing high and low refractive index BN layers exactly at the reflectance extrema. This ensured that the reflectance maximum will occur close to 635 and allowed for monitoring the maximum reflectance value after growing each individual layer pair. Such an in situ optimized growth process is in principle possible for different wavelengths provided the right equipment is available.

Table 1. Variable process parameters of the fabricated DBR structures. Values for samples C and D are average and may vary depending on the individual BN layer. Growth times of all individual component BN layers of samples C and D are available in Table T1 in the supplementary material.

| DBR sample | Time of BN growth at 640 °C [min] | Time of BN growth at 820 °C [min] | Number of BN layer pairs | Carrier gas |
|------------|----------------------------------|----------------------------------|-------------------------|------------|
| A          | 27.5                             | 14.5                             | 15.5                    | Hydrogen   |
| B          | 26.5                             | 16.25                            | 15.5                    | Hydrogen   |
| C          | 24.2 ± 0.9                       | 15 ± 0.4                         | 15.5                    | Hydrogen   |
| D          | 6 ± 0.2                          | 5.5 ± 0.2                        | 15.5                    | Nitrogen   |

Figure 4. (a) Schematic of the DBR; (b) AFM amplitude image of a cross-section of DBR A, collected in tapping mode.
Figure 5. (a) In situ-reflectance spectra of selected DBR structures, measured for 635 nm at the center of the wafer. (b) Reflectance spectra of the fabricated DBR structures, measured at the center of the wafer after the deposition process.

Figure 6. Peak reflectance level measured at different points of the 2″ DBR samples. The upper parts of the wafers were located closer to the center of the showerhead in the MOCVD process. The anomalies in the lower part of the graphs are due to the presence of serial numbers on the substrate wafers.
Figure 5 presents the post-process reflectance spectra of the fabricated DBR samples recorded at the center of the 2″ wafer on which the DBR was deposited, while figure 6 depicts the mapped peak reflectance level for the whole 2″ DBR wafer in the case of all fabricated DBRs. 15.5 layer pairs were enough to achieve 87 ± 1% reflectance at the maximum, measured at the center of the wafer. For some areas of the wafer that value was greater, exceeding even 90% in the case of DBR C. The spectral width of the high reflectance area for which the reflectance is greater than 80% is about 30 nm.

Figure 5(b) presents the post-process reflectance spectra of the fabricated DBR samples recorded at the center of the 2″ wafer on which the DBR was deposited, while figure 6 depicts the mapped peak reflectance level for the whole 2″ DBR wafer in the case of all fabricated DBRs. 15.5 layer pairs were enough to achieve 87 ± 1% reflectance at the maximum, measured at the center of the wafer. For some areas of the wafer that value was greater, exceeding even 90% in the case of DBR C. The spectral width of the high reflectance area for which the reflectance is greater than 80% is about 30 nm.

Figures 7(a), (b) presents STEM micrographs of a cross-section of DBR A. The sample clearly exhibits a layered structure with alternating high-index BN layers (bright parts of the scan) and low-index BN layers (dark parts of the scan). However, the individual layers exhibit waviness and slight variation in their thickness depending on the spot in the sample. These fluctuations are in the sub-micrometer scale and should not affect DBR properties, as is confirmed by further simulations (see figure 8). The porosity contrast between neighboring BN layers grown at different temperatures is clearly noticeable. More porous, less dense layers appear darker in the STEM image. The black curve in figure 7(c) presents the intensity of the STEM signal extracted across horizontal lines from figure 7(a), averaged over a 500 nm wide area. The high-intensity areas correspond to the high-index BN layers and the low-intensity areas correspond to the low-Index BN layer, therefore, this figure allows to estimate the thickness of each individual BN layer. The red and blue curves in figure 7(c) represent the relative EDS signal from boron and nitrogen atoms, respectively. Higher
levels of the boron signal coincide with the high level of the STEM signal, while high levels of the nitrogen signal coincide with low levels of the STEM signal. Therefore, we can conclude that the stoichiometry of the BN is different in the layers grown at different temperatures, which may also contribute to the observed differences in the refractive index values.

We have implemented the thickness values determined from figure 7 into the TMM model to simulate the reflectance profiles of DBRs A and B. The refractive index values of both types of BN layers were taken from figure 2. The exact simulation procedure is described in the supplementary material. The results for DBR A are presented in figure 8(a). Although there are some discrepancies between the measured and simulated curves, the fit reproduces the shape of the reflectance curve very well. Similar calculations were performed in the case of DBR B (figure 8(b)). In this case, the exact thicknesses of the BN layers were not known. However, a very good fit was achieved by assuming that the ratio of individual layer thickness between DBRs A and B is equal to the ratio of the growth times for these layers.

3.3. Creating microcavities from the BN-based DBRs

To construct a microcavity, we have fabricated yet another DBR structure (DBR E), however this time, it was deposition onto a double-side polished sapphire substrate instead of a single-side polished one. The growth times of both types of BN layers have been adjusted so that the reflectance maximum at the center of the wafer would occur at around 650–660 nm. This spectral region is particularly interesting in case of 2D materials such as MoS2 since its exciton energy corresponds to this spectral range. The growth details for DBR E are listed in table 2. Reflectance maps for this DBR are available in the supplementary material.

An optical microcavity is a space inserted between two DBR structures. To achieve the construction of such a cavity, we have deposited a non-transparent layer of aluminum at the edge of the wafer with the DBR E, then cut the wafer in half. The resulting structure is presented in figure 9(a). We have then placed one half of the wafer on top of the other in such a way that the aluminum layers are in direct contact. Lastly, we have placed additional weights to press down the edges on opposite sides of the wafer (figure 9(b)). That way, the aluminum serves as a spacer and allows to create a wedge-like air-filled microcavity between two identical DBRs. Figure 9(c) depicts a schematic drawing of the mapped area, with selected pixels marked with different colors. Figure 9(d) presents the reflectance profiles measured at the marked points. Movie V1 in the supplementary material presents the reflectance measured for all points on this line. Constructing even such a simple system allows observing the wedge-like microcavity. Near the aluminum spacer, the cavity is too large to observe any dip in the reflectance at the high-reflectance plateau. As the distance from the spacer increases, the width of the air cavity decreases. In turn, two dips in the reflectance spectrum arise, then transition into one dip, then back to two. The position of these dips also varies depending on the spot, which is also a result of the different width of the air cavity.

The Q-factor of the cavity measured at the spot marked in black in figure 9(c) equals 220. Using TMM calculations, we simulated the spectral reflectance curves of the DBR optimized for the wavelength of 650 nm with an air cavity of varying thickness (see video V2 in the supplementary material). Based on these simulations, we estimate the thickness of the acquired cavity to be in the range of 3–7 microns.
Due to the easy construction, many 2D materials such as transition metal dichalcogenides can be inserted in such microcavities. In theory, this provides means to measure weak optical signals from these materials or even single-photon phenomena. Another interesting approach would be to fabricate an all-BN single-photon source, by depositing an appropriate hBN layer [19, 20], possibly with intentional carbon defects [21] on top of a BN-based DBR followed by depositing another BN-based DBR in a single MOCVD process.

4. Conclusions

We demonstrate that it is possible to grow DBRs made of a single material in a single process. By using MOCVD, we fabricated DBR structures consisting purely of BN. To this end, we grow two types of BN layers with high refractive index contrast by only modifying the growth temperature during the process. STEM results show that both types of fabricated layers are porous, but to a different extent. This variation in porosity explains the difference in the refractive indices of these layers. We also show that the growth process can be controlled by in situ reflectometry, allowing to fine tune the growth of subsequent layers. By implementing experimentally measured quantities, the layer thicknesses (extracted from STEM results) and the refractive index values (from spectroscopic ellipsometry), into a Transfer Matrix Model, we were able to achieve a very good numerical fit to the experimental normal reflectance data. Furthermore, we demonstrate that the fabricated DBRs can be used to construct an air-filled optical microcavity. The presented method of fabrication of DBRs entirely made of BN is very promising for future integration with BN based single photon emitters, opening up the possibility to produce dedicated all BN single photon sources.

Figure 9. (a) photograph of DBR E with aluminum spacer deposited near the edge of the wafer, (b) DBR microcavity set up as for measurement, (c) schematic of the mapped area, (d) reflectance profiles measured at the points marked in (c). The colors of the curves correspond to the colors of the points. Each curve has been offset by 100% with respect to the previous one—i.e. for the red curve 100% should be read as 0% and 200% as 100%, for the black curve 200% should be read as 0% and 300% as 100% and so on.
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Data availability statement

The data that support the findings of this study are openly available at the following URL: DOI: 10.18150/BS7WBK.

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