

## Article

# Facile Synthesis of High-Quality Nano-Size $^{10}\text{B}$ -Enriched Fibers of Hexagonal Boron Nitride

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**Abstract:** The interesting properties of hexagonal boron nitride (h-BN) and its potential uses in thermo-structural advanced applications have been limited or restricted by its inherent brittleness, which can easily be eliminated by its fibers (h-BN) in nanoscale dimensions. The current study is based on the synthesis of nanoscale  $^{10}\text{B}$ -enriched fibers of h-BN ( $^{10}\text{BNNFs}$ ) from  $^{10}\text{B}$  in the precursors instead of B in two-hour annealing at  $900\text{ }^{\circ}\text{C}$  and one-hour growth at  $1000\text{ }^{\circ}\text{C}$ . All of the  $^{10}\text{BNNFs}$  are randomly curved and highly condensed or filled from  $^{10}\text{h-BN}$  species with no internal space or crack. XRD peaks reported the  $^{10}\text{h-BN}$  phase and highly crystalline nature of the synthesized  $^{10}\text{BNNFs}$ .  $^{10}\text{h-BN}$  phase and crystalline nature of  $^{10}\text{BNNFs}$  are confirmed from high-intensity peaks at  $1392\text{ (cm}^{-1}\text{)}$  in Raman and FTIR spectroscopes.

**Keywords:** enriched-boron; annealing; growth-duration; synthesis;  $^{10}\text{BNNFs}$

## 1. Introduction

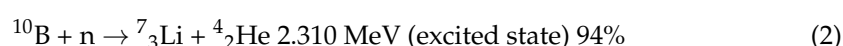
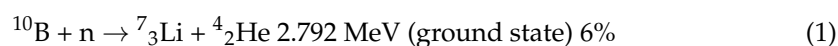
Nanofibers have a large surface area and porous structure due to which it has characteristic properties suitable for a variety application in the field of environmental sciences, automotive industries, smart textile and biomedical, etc. [1].

Boron nitride (BN) is an important material known as an artificial non-oxide ceramic. Hexagonal or cubical are the most commonly available phases of BN. Both of these polymorphs have interesting properties: high-temperature stability, high electric insulation, the

low dielectric constant, large cross-section for thermal neutron and resistance to oxidation at high temperature, etc. [2–4]. Among all polymorphs of BN, hexagonal boron nitride (h-BN) is more interesting due to its stability and ease of synthesis or production. However, the inherent brittleness is the only drawback of h-BN which restricts its use in thermos structural advanced applications [2,5]. The brittleness issue can be resolved by preparing h-BN in fibers. Afterward, it can be used as composite materials. Such composites have all the interesting properties of nanoscale materials of h-BN. However, the first step in making such composites is the synthesis of h-BN fibers (BNFs), which are commercially not available in the market [5]. Because of this goal, many researchers have focused on developing a reliable technique for producing fibers of h-BN. Nanosize fibers of h-BN (BNNFs) were claimed with boron oxide as a precursor. For the increased spin ability, polyvinyl butyral (PVB) was added to the solution. The pyrolyzation of the as-formed product resulted in the synthesis of BNNFs with a diameter under 100 nm [6]. Next time, electrospun polyacrylonitrile (PAN) was coated with boron oxide solutions to form BNNFs [7]. Oxygen contents in the final product was one of the very serious issues related to the above techniques. Polyborazine/polyacrylonitrile blend solutions were also used as precursors for the synthesis of BNNFs [8]. The health-risky nature of the precursors and the complexity of the experimental procedure were some of the main problems associated with the above technique. Boric acid and melamine-based polymeric precursors had been used to synthesize BNFs with lengths in the range of 200–500  $\mu\text{m}$  and diameters of 1–2  $\mu\text{m}$ . The adopted procedure was claimed to be cost-effective, simple, and notified to form pure BNFs [2]. However, unlike the claim, the procedure was found to be complex and lengthy with carbon- and nitrogen-based compounds as impurities in the final product. The as-found impurities were almost impossible to eliminate even at a higher temperature of 1600  $^{\circ}\text{C}$ .

Regardless of the pros and cons of the above techniques, a simple and cost-effective technique was needed to synthesize fibers of h-BN. The fibers of h-BN needed to be not only pure and nanoscale but also  $^{10}\text{B}$ -enriched.

$^{10}\text{B}$  is one of the stable isotopes of Boron, which has a thermal neutron cross-section of 3840b. The daughter nuclei of  $^7\text{Li}$  and  $^4\text{He}$  are produced when a thermal neutron is captured by  $^{10}\text{B}$  according to the nuclear reactions given as below [9,10]:



$^{10}\text{B}$ -enriched nanofibers of h-BN ( $^{10}\text{BNNFs}$ ) are large bandgap semiconductors. The emitted daughter's nuclei when accelerated into the semiconductor layer of  $^{10}\text{BNNFs}$  produce electron-hole pairs. These pairs, when detected by their respective electrodes, can be shown in the form of electrical signals. Thus,  $^{10}\text{BNNFs}$  can be a potential element in solid-state neutron detectors like  $^{10}\text{BNNTs}$  [11] and other nuclear feeding activities. In this reference, it can also be a useful material in boron neutron capture therapy (BNCT), bio-medical and drug delivery, etc.

The method for the synthesis of  $^{10}\text{BNNFs}$  in the current study can be traced back to the Boron oxide chemical vapor deposition (BOCVD) technique for the synthesis of BNNTs. In BOCVD, Boron (B) and Magnesium oxides ( $\text{MgO}$ ) were developed as carbon-free precursors for the synthesis of BNNTs in high quality [12]. The BOCVD precursors for the BNNTs synthesis were further developed by the addition of iron oxides ( $\text{FeO}$ ). As a result, the mixture of B,  $\text{MgO}$ , and  $\text{FeO}$  was developed as effective precursors. The precursors resulted in the synthesis of high-quality of BNNTs [13]. The as-introduced precursors were obtained in nano-size with  $\gamma\text{-Fe}_2\text{O}_3$  instead of  $\text{FeO}$ . Such a precursor's mixture has successfully been optimized for the synthesis of nanotubes, nanowires, nanosheets, and microtubes, etc., of h-BN [14]. The optimization also resulted in a simple technique for the synthesis of BNFs. The work on the synthesis of BNNTs showed that the replacement of B by  $^{10}\text{B}$  resulted in enriched boron nitride nanotubes ( $^{10}\text{BNNTs}$ ) [15]. The logics of  $^{10}\text{BNNTs}$

and BNFs lead to the synthesis of enriched boron ( $^{10}\text{B}$ ) nitride nanofibers. The detail of the methodology thus developed for the synthesis of  $^{10}\text{BNNFs}$  is given in the next section.

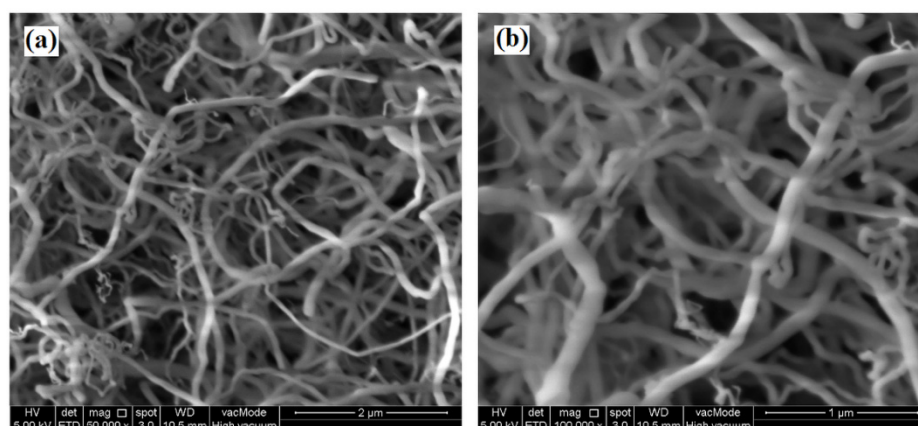
## 2. Materials and Methods

A 100 mg mixture of Magnesium oxides ( $\text{MgO}$ ) and Iron Oxides in a 1:1 ratio is uniformly mixed with enriched Boron ( $^{10}\text{B}$ ) powder of 100 mg in weight. The mixture is put in an alumina boat and annealed in the Argon atmosphere at  $900\text{ }^{\circ}\text{C}$  for 2-hours. Afterward, the annealed mixture in the boat is partially covered via a few Silicon ( $\text{Si}$ ) substrates and pushed inside one end closed quartz tube already placed in the quartz tube chamber (beneath the heating filament) of the horizontal tube furnace. The furnace is seal closed and afterward flushed with Argon gas with a flow rate of 100 sccm. Consequently, the furnace is programmed to heat the precursor's mixture with a heating rate of  $10\text{ }^{\circ}\text{C}/\text{min}$ . During heat-up, Argon gas flow (100 sccm) is maintained as a source of an inert atmosphere. In such a condition, the precursor's mixture is heated up to  $800\text{ }^{\circ}\text{C}$  [16]. At  $800\text{ }^{\circ}\text{C}$ ,  $\text{NH}_3$  (200 sccm) is introduced into the system as a reactive gas. Under such a condition, the precursor's mixture is further heated up to  $1000\text{ }^{\circ}\text{C}$ . Growth of the final material was assumed to occur at  $1000\text{ }^{\circ}\text{C}$ . Therefore,  $1000\text{ }^{\circ}\text{C}$  is fixed as the final temperature. At the final temperature, the system is maintained for 1-hour to complete the growth. Subsequently, the reactive gas ( $\text{NH}_3$ ) flow is stopped whereas the inert (Argon) atmosphere was maintained till the system was brought to room temperature.

## 3. Results and Discussions

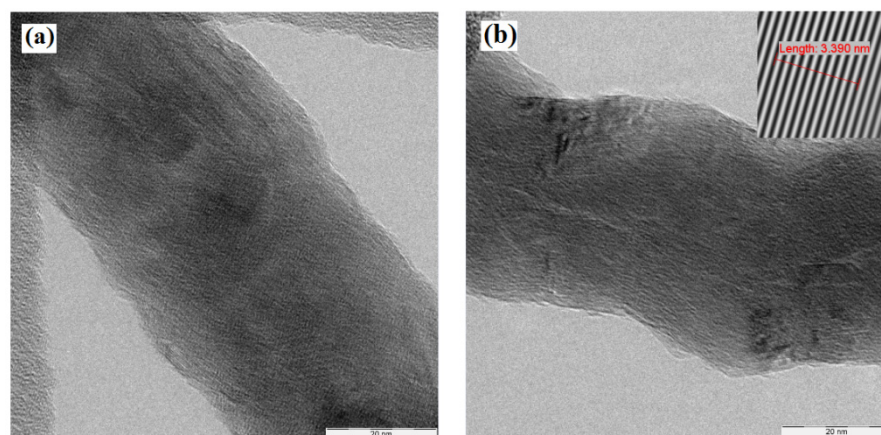
B,  $\text{MgO}$ , and  $\text{FeO}$  were introduced as effective precursors for the synthesis of BNNTs [13]. The replacement of enriched Boron ( $^{10}\text{B}$ ) in the precursors with  $\text{MgO}$  and  $\gamma\text{-Fe}_2\text{O}_3$  in the precursors resulted in the synthesis of enriched Boron nitride nanotubes ( $^{10}\text{BNNTs}$ ) [15]. Along with the precursors, variations in the experimental parameters have also been found to play a crucial role in the synthesis of different nano ( $10^{-9}$ ) or micro ( $10^{-6}$ ) structures of hexagonal boron nitride (h-BN) [14,17]. As a strategy, experimental procedures were successfully optimized in two stages of annealing and growth duration. As a result, a technique was developed for the synthesis of micron-scale fibers of h-BN [18]. In that technique, the precursor's mixture (B,  $\text{MgO}$ , and  $\gamma\text{-Fe}_2\text{O}_3$ ) was first annealed (in the inert atmosphere) for two hours at  $900\text{ }^{\circ}\text{C}$ . Afterward, the annealed mixture was first softened in the Argon atmosphere up to  $800\text{ }^{\circ}\text{C}$ . The growth was initiated at  $800\text{ }^{\circ}\text{C}$  with  $\text{NH}_3$  as a reactive gas and then continued for two hours at  $1000\text{ }^{\circ}\text{C}$  [18]. In the present work, the goal was to synthesize not only fibers of h-BN in nano-size but also  $^{10}\text{B}$ -enriched. Therefore, changes had to be brought not only in precursors but also in the experimental parameters. As for precursors,  $^{10}\text{B}$  was introduced as a precursor with  $\text{MgO}$  and  $\gamma\text{-Fe}_2\text{O}_3$  instead of Natural B whereas, for the reduction of size, the growth duration was reduced to one hour instead of two. The rest of the procedure remains the same as for the synthesis of micron size BNFs [18]. The changes result in the synthesis of high-quality nano-size  $^{10}\text{B}$ -enriched fibers of h-BN ( $^{10}\text{BNNFs}$ ). Figure 1 shows the as-synthesized  $^{10}\text{BNNFs}$  characterized by Field emission scanning electron microscopy (FESEM). Figure 1a shows a relatively lower magnification micrograph of the as-synthesized  $^{10}\text{BNNFs}$ . All of the  $^{10}\text{BNNFs}$  are randomly curved and dispersed at the top of the Si-substrate. In general, the  $^{10}\text{BNNFs}$  create a view of cooked noodles from the top. All the  $^{10}\text{BNNFs}$  are of variable size or diameter in the range of greater than 20 nm and smaller than 100 nm. The smaller size  $^{10}\text{BNNFs}$  lay among or beside larger size  $^{10}\text{BNNFs}$ . Some of them seem to stick with others and form clusters. The region with a cluster-like appearance is further magnified and viewed in higher magnification as shown in Figure 1b. In a higher magnification micrograph, most of the  $^{10}\text{BNNFs}$  seem isolated. A cluster-like appearance is a place in the micrograph where a higher density of smaller size  $^{10}\text{BNNFs}$  is present. Both smaller and larger size  $^{10}\text{BNNFs}$  have a variable diameter and randomly curved morphology with an average length of greater than five microns. No other species or morphologies can be seen in higher

magnification. To internally look into the structure, the  $^{10}\text{B}$ NNFs sample is analyzed with the help of a high-resolution transmission electron microscope (HRTEM).



**Figure 1.** FESEM shows the apparent structure and morphology of the as-synthesized  $^{10}\text{B}$ -enriched fibers of h-BN ( $^{10}\text{B}$ NNFs) in (a) low and (b) higher magnification.

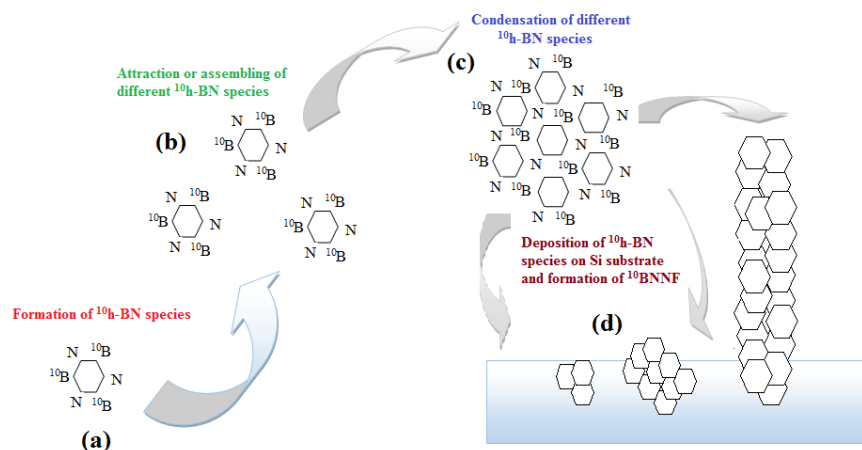
Figure 2a shows an individual  $^{10}\text{B}$ NNF from the as-synthesized  $^{10}\text{B}$ NNF sample. The  $^{10}\text{B}$ NNF looks like a round and solid concrete pillar, which is highly condensed or filled with no internal space or crack. The HRTEM shows a non-uniform or rough outer surface of the as-synthesized  $^{10}\text{B}$ NNF. The rough or non-uniform outer surface confirms the non-uniform or variable diameter of  $^{10}\text{B}$ NNF in the sample. To further confirm this fact and others, another  $^{10}\text{B}$ NNF is arranged on the TEM copper grid and characterized via HRTEM. The micrograph of this  $^{10}\text{B}$ NNF is shown in Figure 2b. It shows that the  $^{10}\text{B}$ NNF is curved from various points. These curves in the structure confirm the curved morphology of the  $^{10}\text{B}$ NNFs shown in Figure 1 via FESEM. Besides this, the  $^{10}\text{B}$ NNF has a solid or condensed structure. However, the condensation of the  $^{10}\text{h}$ -BN species in the current  $^{10}\text{B}$ NNF is different from the one shown in Figure 2a. Unlike the previous, the  $^{10}\text{h}$ -BN species in the current  $^{10}\text{B}$ NNF seems to be condensed from out to inside. As a result of this condensation, the  $^{10}\text{B}$ NNF attains a rope-like morphology. Some crack-like spots can also be seen near the center. However, these are not the cracks. These are the points where  $^{10}\text{h}$ -BN species from different directions meet and construct the final morphology. All the condensed species have visible lattice fringes with an interlayer spacing of  $\sim 0.34$  nm, as shown in the inset on the upper right hand of Figure 2b. According to the available literature, these layer spacings are the characteristics of the h-BN lattice with a highly crystalline nature [19].



**Figure 2.** HRTEM micrographs show (a) a highly condensed or filled  $^{10}\text{B}$ NNF with no internal space or crack, (b) curved  $^{10}\text{B}$ NNF from various points with  $^{10}\text{h}$ -BN species condensed from out to inside. The inset on the upper right-hand corner shows the interlayer spacing.

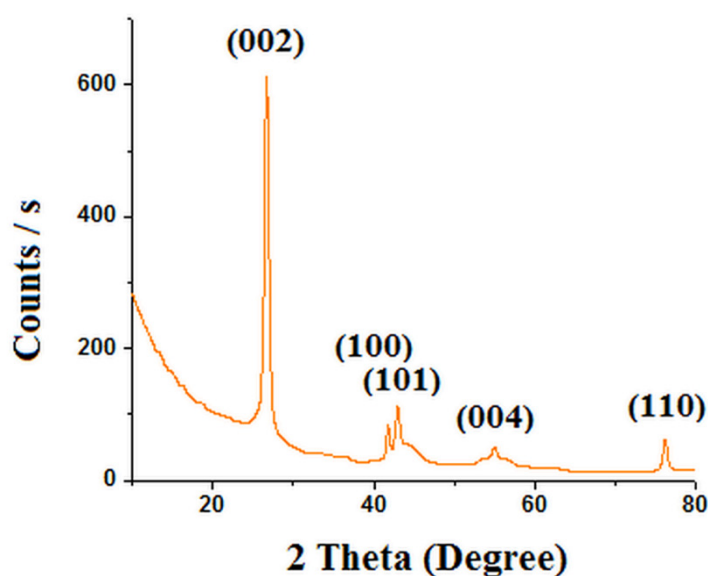


The possible plan for the formation (during different growth stages) of  $^{10}\text{BNNF}$  due to condensation of  $^{10}\text{h-BN}$  species is shown via a sketch in Figure 3. The sketch shows (a) the formation, (b) attraction or assembling (c) condensation, and finally (d) deposition of condensed  $^{10}\text{h-BN}$  species on Si substrate forming  $^{10}\text{BNNF}$ .



**Figure 3.** A sketch showing different growth stages (a–d) of the as-synthesized  $^{10}\text{BNNF}$ s.

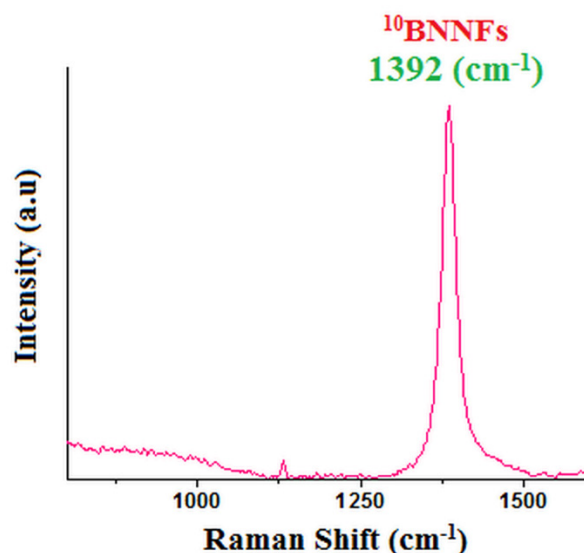
The compositions, crystalline nature, and phase of the as-synthesized  $^{10}\text{BNNF}$  sample are checked with X-ray diffraction (XRD). The as-obtained XRD pattern is shown in Figure 4. The peaks in the XRD pattern are spotted at different 2-theta values of  $26.7^\circ$ ,  $41.8^\circ$ ,  $43^\circ$ ,  $55^\circ$ ,  $76.2^\circ$ . The sharp and high-intensity peak at  $26.7^\circ$  stands for (002) planes in  $^{10}\text{h-BN}$  with a highly crystalline nature. The peaks with lower intensities reported at  $41.8^\circ$ ,  $43^\circ$ ,  $55^\circ$ ,  $76.2^\circ$  correspond to (100), (101), (004) and (110) planes in  $^{10}\text{h-BN}$  [13,20]. The intensities of the peaks on one side indicate the crystalline nature of the sample and on the other side reflect the higher number of planes in that particular sample. Based on this logic, the majority of planes present in the current  $^{10}\text{BNNF}$  sample are (002).



**Figure 4.** The as-obtained XRD pattern shows peaks for  $^{10}\text{h-BN}$  contents of the as-synthesized  $^{10}\text{BNNF}$ s.

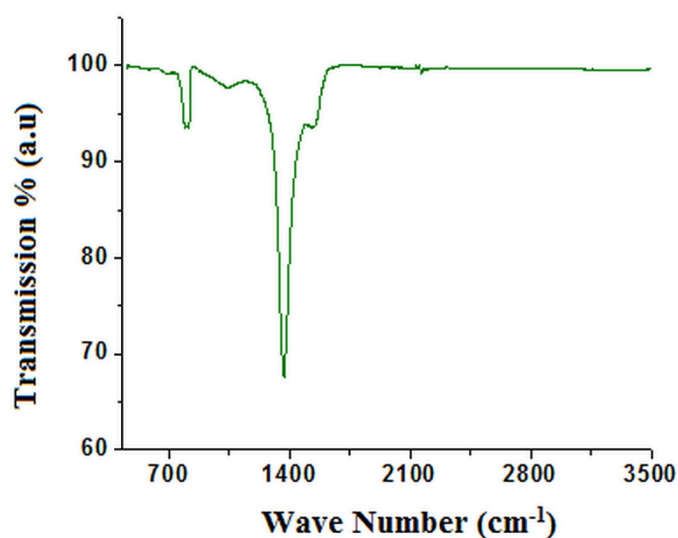
Non-destructive Raman spectroscopy was used to further check the contents, phase, and crystalline nature of the synthesized  $^{10}\text{BNNF}$ s. Raman spectroscopy of the sample is carried out in the spectral range of  $800\text{--}1600\text{ cm}^{-1}$  with a laser beam with a wavelength of  $514\text{ nm}$ . The obtained Raman spectrum of the sample is displayed in Figure 5. The spectrum shows a high-intensity peak in the displayed spectrum in  $1392\text{ cm}^{-1}$ . This

high-intensity peak according to the available literature corresponds to the  $E_{2g}$  mode of vibrations in  $^{10}h\text{-BN}$  (with B–10 content) or  $^{10}\text{BNNFs}$  [15,21]. During Raman spectroscopy of the sample, a small peak can also be seen in the spectrum at  $1150\text{ (cm}^{-1}\text{)}$ . Such a lower intensity peak in the spectrum can be assigned to the formation of boric acid. It might have been formed due to sample exposure to oxygen in the air or  $^{10}\text{B}_2\text{O}_3$  contents in the sample with laser interaction during Raman spectroscopy [22].



**Figure 5.** The spectrum shows a high-intensity peak in the displayed Raman spectrum at  $1392\text{ (cm}^{-1}\text{)}$  for the synthesized  $^{10}\text{BNNFs}$  sample.

Fourier transformed infrared (FTIR) spectroscopy of the  $^{10}\text{BNNF}$  sample is performed in the spectral range of  $400\text{--}3500\text{ (cm}^{-1}\text{)}$ . The as-obtained FTIR spectrum is shown in Figure 6. The FTIR spectrum shows a lower intensity peak at  $800\text{ (cm}^{-1}\text{)}$ . It is a weak absorption and corresponds to an out-of-plane R-mode. In this mode of vibrations, Boron and Nitrogen atoms move radially in or outward. Along with lower intensity, a higher intensity peak was also spotted in the FTIR spectrum at  $1392\text{ (cm}^{-1}\text{)}$ . It is a strong absorption corresponds to in-plane stretching in  $^{10}h\text{-BN}$ . In this mode, the atoms vibrate along longitudinal or tube axis called L-mode [21].



**Figure 6.** FTIR spectrum shows peaks correspond to “R” and L-mode of vibrations in  $^{10}h\text{-BN}$ .

#### 4. Conclusions

As the use of B, MgO, and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> as precursors mixture worked for the synthesis of nanotubes, nanowires, nanosheets, and microtubes, etc., of hexagonal boron nitride with different experimental parameters. Likewise, the same precursor's mixture with two hours annealing and two hours growth duration resulted in micron size fibers of hexagonal boron nitride. This is because the <sup>10</sup>B-enriched boron nitride nanotubes can be synthesized by using <sup>10</sup>B instead of B in precursors with MgO and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>. Similarly, the same precursor's mixture (<sup>10</sup>B, MgO, and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>), with a growth duration of one hour instead of two, resulted in enriched nanoscale fibers of h-BN. <sup>10</sup>B enrichment, nanoscale properties, and semiconductor nature enable <sup>10</sup>BNNFs a potential material to be used as a sensing element in solid-state neutron detectors, BNCT and biomedical applications, amongst others

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