Mark Hui's difficulties in explaining differences in binding energy data on boron

Professor Leo Lau, Dr. Mark Hui, and Professor Carmel McNaught wrote this teaching/learning case in April 2003 with the expectation of the following learning outcomes from students:

1. *Students appreciate both the complexity and manageability of a practical surface science problem.*
2. *Students identify key issues in a practical problem.*
3. *Students understand the concept of Fermi level.*
4. *Students learn how to interpret an XPS spectrum.*

A. CASE SITUATION

After having a 3-hour long discussion with his research supervisor on his X-ray photoelectron spectroscopic (XPS) studies of a series of samples commonly having boron as their main elemental constituent, Mark Hui was still not too sure if he fully understood Professor Lau's criticisms against the appropriateness of both his measurements and data interpretations. On his research diary, he recorded the following key remarks given by Professor Leo Lau in the meeting:

- Differences among our boron 1s binding energy measurements and those data reported by other research groups in the literature on "pure" elemental boron crystals may not be caused by variations in instruments, as what you assumed. You should check if you and these research groups have followed the standard XPS measurement procedures, particularly those on binding energy calibration and detection of surface charging effects.

- In XPS analysis, binding energy of a photoelectron peak is traditionally defined as the energy difference between the Fermi level of the sample to the spectral peak location of the photoelectrons. When you measure binding energy data from your sample, you need to make sure that the Fermi level of the sample is aligned, within an acceptable experimental error, with the Fermi level of the spectrometer. When you interpret the binding energy data, you need to know if the sample has a bandgap because the binding energy of a sample having a bandgap can vary as much as the bandgap energy. For example, the binding energy of Si $2p_{3/2}$ of n-Si and p-Si can differ by 1eV. You should read my past publications in XPS studies of Fermi level pinning and surface passivation of semiconductors, and surface charge spectroscopy.

- As for your measurements on boron having different degrees of surface oxidation and other boron oxide samples, you need to know the conditions and effects of fixed charge in these samples.

Mark knew that he had to go over the basic principle of XPS and its measurement procedures carefully again, and to conduct more thorough literature search. He planned to start the former task by visiting the website of the Surface Science Course in the department, and the latter task by visiting the Web of Science. But yet he realized that first of all, he had to review his existing experimental results and to define the key issues in his present case situation. He had one week before he would meet with Professor Lau again but the anxiety in him had already begun to rise.

Mark’s current experimental data and interpretations:

I. BACKGROUND

Boron (B) and many boron-rich solids are known to be constructed of different skeletons of icosahedral atomic clusters.[1-2] These solids are hard owing to the presence of strong three-center two-electron bonds in the icosahedra, and strong two-electron bonds interconnecting the icosahedra.[3-5] Incorporation of impurities into the skeletons enhanced the cross-linking between the icosahedral clusters, resulting in even higher hardness.[3,6,7] Addition of oxygen into boron produces boron suboxide (B_xO), which has been reported to be extremely hard in its bulk form, and so is of great scientific and practical interests. [8]

High-temperature high-pressure sintering is the most widely used technique for preparing bulk B_xO and several compounds have been reported. [8-15] It is generally accepted that there exists a stoichiometric phase $B_{12}O_2$. The X-ray diffraction pattern of $B_{12}O_2$ was observed and well documented. In addition to this compound, Badzian reported that $B_{22}O$ solid sintered at 0.7 MPa and 1600 – 2000 ^oC, was hard enough to generate wear grooves on the (111) diamond faces.[8] However, the presence of this compound has never been reported again. Moddeman et al. employed X-ray photoelectron spectroscopy (XPS) to investigate the structure of synthesized boron suboxide, such that the chemical environment surrounding the boron atoms can be revealed. The B 1s spectra were resolved into many components, corresponding to boron atoms in different chemical states.[15]

The above observations have driven further studies of the fabrication of boron suboxide films, and their applicability as surface coatings. The mechanical properties of B_xO films deposited by plasma enhanced evaporation and plasma enhanced sputtering were investigated as a function of the oxygen content.[16-18] However, reliable data on the composition and electronic structure of B_xO thin films are scarce..

In this report, we present the results of XPS analysis on polished boron crystal, boron sputtering target material, crystalline B_xO pellets synthesized by hot isostatic press (HIPS), and dual ion beam deposited (DIBD) B_xO films with various oxygen contents. XPS is used because it probes the chemical bonding environments of the boron atoms. Through the deconvolution of the B 1s photoelectron spectra, the results of the first three groups of specimens were used as references, such that the correlation between the resolved components and various boron states was established. The XPS data of the DIBD films were then analyzed to reveal the composition of these boron states in the film structure. The central goal is to evaluate whether the proposed superhard boron suboxide phases are formed in thin films.

II. EXPERIMENTAL METHODS

A. Sample preparation

The crystalline boron sample (99.5 % purity) was supplied by Johnson Matthey. A chip was polished by using submicron-sized diamond paste to generate a flat surface for XPS measurements. This treatment eliminated the shadowing effect during ion bombardment and photoelectron detection, which influence is significant for a rough surface.

The second sample is a piece of boron sputtering target material provided by Kurt J. Lesker. The purity of the material was claimed to be 99.9 %.

The third group of samples contains three pellets, each 3 mm thick and 1 cm in diameter. HB0 was formed by pressing boron powder (< 44 μm, 99.9 % purity) at a pressure of 0.4 GPa at room temperature. To prepare HB1 and HB2, pellets were first produced with the same procedures from zinc oxide-boron powder mixtures (ZnO, ≈ 100 μm, > 99.5% purity) with molar ratios of 1 : 20 and 1 : 8 respectively. The pellets were then sintered by HIPS method at a pressure of 0.18 GPa in high purity Ar and 1800°C for 4 hour. The route for fabricating HB2 was reported to be successful to create the crystalline $B_{12}O_2$ compound through the reaction[12] :

$$16\,B + 2\,ZnO \rightarrow B_{12}O_2 + 4\,B + 2\,Zn\,(g)\uparrow.$$

The B and B_xO films were deposited in a high vacuum system. The background pressure was 2×10^{-6} Torr. The substrate temperature was set at 750 °C. The system had two Kaufman type ion guns. One ion gun was used to generate a 1200 eV-70 mA argon (Ar^+) ion beam to sputter a 4-inch B target. Another ion gun was used to generate a 300 eV-12 mA ion beam containing different fractions of Ar^+ and oxygen ion flux to bombard the substrate. This is referred as the “ion assist” gun in this report. Only B002 was deposited with no usage of the ion assist gun . From B003 to B015, the O_2/Ar flow ratio admitted into the ion assist gun was increased from 0 to 3.1 successively. The film thickness determined by an α-step surface profiler fell in the range of 381 – 573 nm.

B. XPS measurements

A PHI Quantum 2000 XPS system (with Al K_α source) was used to collect the B 1s, O 1s and C 1s photoelectron spectra of the samples with a take-off angle of 90°. The relative content of an element is in proportion to the area of the spectrum above the Shirley background divided by the corresponding sensitivity factor. The sensitivity factors of B, O and C are 0.133, 0.733 and 0.314 respectively.

For the crystalline sample, XPS spectra were collected after sputter-cleaning with 2-keV Ar^+ ions for 2.5 min. This step favors the acquisition of an ideal reference spectrum from the freshly cleaned crystal surface. Spectra were collected again after keeping the specimen in ultrahigh vacuum (UHV, 4.1×10^{-8} Torr) for 1000 min. All data were collected without low energy electron flooding except those specially specified.

For the target material sample, the spectra were recorded before sputter-cleaning, and after every one of the two severe sputtering processes (2 and then 4 keV Ar^+ ions, each for 20 min). This scheme examined roughly the severity of oxidation, and the profiles of O and C contents along the direction of depth.

For the HIPS samples, XPS spectra were recorded only before sputter-cleaning in order to prevent from the complicity due to the ion mixing effect caused by ion bombardment. Low energy electron flooding was applied to control sample charging. X-ray diffraction (XRD) was carried out by using a Philips X'PERT X-ray diffractometer (Cu K_α radiation, in θ-2θ mode) for investigating the crystallinity of the samples.

The XPS spectra of the DIBD films were recorded both before and after sputter-cleaning with 500-eV Ar^+ ions for 10 min. Lower sputtering ion energy was used to minimize the ion mixing effect. XRD experiments were also carried out for this group of samples. Measurements were done in the 2θ-scanning mode, where the incident angle of the X-ray beam was fixed at a small glancing angle of 2^o, and 2θ was varied from 15 to 60^o. No characteristic peak was detected, so that all the films were identified to be X-ray amorphous. The same result was obtained for a trial run with the substrate temperature elevated to 850 ^{o}C.

III. RESULTS AND DISCUSSIONS

A. Crystalline boron

For the crystalline sample, after ion sputtering the oxygen content was detected to be lower than 0.6 at. % (O-to-B ratio = 0.006). Furthermore, the carbon and nitrogen contents were also too low to be detected. At this stage, the surface sorbed species as well as the native surface oxide were entirely removed. Therefore, the B 1s spectrum from such a clean surface is considered to be quite ideal to work as a standard reference for pure elemental boron.

This standard B 1s spectrum only contains one component, denoted as B^1, which has a peak energy at 188.1 eV [Fig. 1 (a)]. This value is lower than those reported by Moddeman et al. (187.2 eV) for the elemental boron phase.[15] This discrepancy can partly be due to the difference between the experimental systems used by various groups. However, results of this study suggest that the severity of oxidation of boron is also a factor causing the B^1 peak to shift. This assertion will be elucidated further in the context.

After keeping the specimen in UHV for 1000 min, the oxygen content was detected to increase prominently to 6.5 at. %. At the same time, the B 1s spectrum became slightly asymmetric [Fig. 1 (b)], such that a B^2 component was added in order to get a good fit to the curve. The B^2 component was determined to have a peak energy of 189.3 eV. This feature can be explained by suggesting that an oxide layer was on the freshly cleaned surface. On the freshly cleaned surface, boron atoms were activated and reacted more readily with the residual oxygen remaining in the vacuum chamber. The boron atoms bonded to oxygen are responsible to the generation of the B^2 component. It is also noticed that the B^2 peak is 1.4 eV higher than the B^1 peak, consistent with the hypothesis of surface oxidation.

B. Boron target

For the boron target sample before surface cleaning, the oxygen content was very high, i.e. 44.2 at. %. In addition, the B 1s spectrum is resolvable into three components [Fig. 2 (a)], implying that there are three major chemical states of boron.

The first component lies on the lower energy side and is denoted as the B^1 component. This designation implies implicitly that the component is from the boron atoms not bonded to oxygen. Its peak energy is 187.1 eV, prominently lower than the B^1 peak of the crystalline boron sample at 188.1 eV. Such a deviation is expected to be a result of oxidation, and this hypothesis is explained by the crystal structure in Fig. 3. In this structure, two oxygen atoms are inserted interstitially into a rhombohedral unit cell of α-rhombohedral (α-R) to generate a unit cell of $B_{12}O_2$. A complete transition increases the edge length from 0.5057 to 0.7723 nm. As a consequence, the separation between two adjacent boron atoms (open circles for this component) becomes longer, so that the core electrons experience a weaker potential field to result in a lower binding energy. These boron atoms are not directly bonded to oxygen, and so are responsible for the generation of the B^1 component. The deviation of the peak energy of this component from that of the standard spectrum of crystalline boron is large, suggesting that the oxidation of the target surface is quite severe.

The second component in the B 1s spectrum of the pre-cleaned target surface is denoted by B^2 [Fig. 2 (a)], implying implicitly that it is from the boron atoms bonded to oxygen. The strong intensity of this component further implies that the target surface is severely oxidized. The chemical structure of a boron atom of this type is analogous to that of a boron atom represented by a shaded circle depicted in Fig. 3 for the $B_{12}O_2$ structure. Moddeman et al. designated this local structure as "B_yO_x" with $y/x = 3$. The peak energy of the B^2 component is 188.6 eV, which is lower than the suboxide peak at 189.3 eV of a slightly oxidized boron surface [Fig. 1 (b)], but close to 188.3eV as reported by Moddeman et al. for the boron suboxide phase.[15] The variation of the peak energy of the suboxide-related XPS component from group to group suggests that it is not a constant, but is affected by the severity of oxidation. The third component, B^3, has a peak at 191.9 eV, and is widely acknowledged to come from the B_2O_3 structure.[15]

Subsequent ion sputter sputtering processes induced the following consequences. The oxide layer at the target top surface was stripped. The oxygen content is therefore greatly reduced from 44.2 to a residual value of 8.3 at. %. The cores of the boron grains were exposed, so that the B 1s spectrum [Fig. 2 (b) and (c)] behaved more like that of the crystalline boron sample [Fig. 1 (b)]. On the one hand, the B^2 component became relatively weak as compared to the B^1 component. On the other hand, the respective peak energies of the B^1 and B^2 components rose to 187.9 and 189.3 eV, same like those of the slightly oxidized crystalline boron [Fig. 1 (b)].

There were still many oxygen- and carbon-containing impurities trapped between the boron grains from which the target was hot pressed. They maintained the detected residual oxygen and carbon contents at finite levels of 8.3 and 6.9 at. %. These impurities would be co-sputtered in a DIBD process, and are incorporated into the films deposited.

C. HIPS samples

The XRD pattern of HB0 shows some diffraction peaks of the boron powder (Fig. 4). The signal-to-noise ratio is low because the grain size is as small as 44 μm. The oxygen content is high (20.4 at.%), mainly because the specimen is very porous so that great amount of oxygen-containing impurities is trapped. The B 1s spectrum [Fig. 5 (a)] shows the following features to indicate the presence of oxide phases. The B^1 peak energy (186.7 eV) is lower than that of the sputter-cleaned boron crystal. A strong B^2 component associated with the suboxide structure, and a B^3 component associated with the B_2O_3 structure are observed.

Two sets of diffraction peaks were found in the XRD pattern of HB1 [Fig. 4 (b)]. One set is identified to that of boron powder, and the other is from the $B_{12}O_2$ powder.[11] This result indicates that HB1 is a solid admixture of boron and $B_{12}O_2$ grains. The intensity of the peaks from the $B_{12}O_2$ phase is relatively higher, indicating that this process favors the growth of $B_{12}O_2$ crystallites. The B 1s spectrum of HB1 [Fig. 5 (b)] shows a strong B^2 component, consistent with the growth of the $B_{12}O_2$ grains as reflected by the XRD data.

The XRD pattern of HB2 matches very well with the powder diffraction pattern of crystalline $B_{12}O_2$,[11] verifying that the sample is dominated by the $B_{12}O_2$ phase. Correspondingly, the B 1s spectrum [Fig. 5 (c)] has three components. The B^1 component has a relatively low peak energy (186.9 eV), consistent with the highly oxidized status of the specimen. The B^2 component is highly resolvable with a sharp peak at 188.7 eV, and is attributed to the boron atoms in the suboxide phase as presented by the shaded circles in Fig. 3. The B^3 component has the maximum intensity at 192.1 eV, and should come from the B_2O_3 structure.

E. DIBD films

No characteristic peaks are found in the XRD spectra of all the films, verifying that all of them are X-ray amorphous.

The B 1s spectra of the DIBD films before and after ion sputter-cleaning were shown in Figs. 6 and 7, respectively. Although ion sputter-cleaning reduced the amounts of carbon and oxygen substantially, the changes in the B 1s spectral profile shape were not so drastic. This suggests that the reduced amounts of oxygen and carbon by ion sputter-cleaning might simply be physisorbed in the as-deposited films.

The changes in B 1s spectra among different films (B002 – B015 with a numbering order reflecting an increasing amount of oxygen) show the concomitant changes in the B 1s spectra with increasing oxygen content. First, the B 1 spectra of all the films have a B^1 component, which is consistently to be the strongest. Its appearance represents the boron atoms which are not directly bonded to oxygen. Moreover, the peak energy of this component is insensitive to the variation of the oxygen content, and lies in a narrow region of 187.5 – 188.3 eV.

A B^2 component is resolved to correspond to the formation of the suboxide phase. Its intensity is stronger with increasing oxygen content, indicating a concurrent growth of the suboxide phase. The peak energy lies in the range of 188.8 - 189.6 eV, which is higher than that of the $B_{12}O_2$-dominating HB2 sample, but is closer to 189.3 eV of the slightly oxidized crystalline boron [Fig. 1 (b)]. This suggests that the suboxide phase in the film samples still deviates from the stoichiometric $B_{12}O_2$ structure.

Interestingly, a new component B^4 is required in order to get good fits to the B 1s spectra. Its peak energy lies in the range of 190.7 - 191.4 eV, which approximately matches with that of a similar component reported by Moddeman et al. at. 190.5 eV.[15] They assigned the component to a second suboxide structure denoted as "B_yO_x" with $1.5 < y/x < 3$, corresponding to the boron atoms bonded with the oxygen atoms having coordination number less than 3. Indeed, the mechanism of DIBD makes the film structure to be more distinctive from the specimens of other groups in the study. This is because in a DIBD process, the species arriving the substrate are in a rather dynamic state. Destruction and reconstruction of bonds proceed frequently, so that more new atomic combinations can be achieved. The intensity of the B^4 component increases with increasing oxygen content, because of the concomitant growth of the related suboxide phase. Finally, a relatively weak B^3 component is observed only for B015 containing the highest oxygen content, indicating that the B_2O_3 phase has rather limited contribution to the DIBD films.

IV. CONCLUSIONS

In conclusion, the XPS data of polished crystalline boron, boron sputtering target material, boron suboxide pellets formed by HIPS technique, and DIBD deposited boron and BO_x films before and after sputter-cleaning were analyzed and compared for monitoring the change in the chemical structure of boron under different oxidation states. Special attention was paid to identify the formation of suboxide phase in the film samples, since boron suboxide was claimed to exhibit extremely high hardness.

Ion sputtering on polished crystalline boron produced a very clean surface, giving an ideal reference B 1s spectrum for pure boron. The spectrum was characteristic of containing only one component with a peak energy at 188.1 eV. The cleaned boron surface was partially oxidized by the residual oxygen in the UHV environment after a period of time, causing a B^2 component to emerge at a higher energy region.

The pre-cleaned boron target was severely oxidized at the top surface. Oxygen-containing species were trapped between the boron grains. Native oxide layers were also formed to cover the boron grain surface. Sequential ion sputtering removed most of these oxygen atoms to expose the core of the boron grains. One result is to reduce the detected oxygen content drastically. The next consequence is to make the B 1s spectrum like that of the crystalline sample. The suboxide-related B^2 component and the B_2O_3-related B^3

component became weaker with respect to the B^1 component. Residual oxygen- and carbon-containing species trapped in the gaps between the grains were difficult to completely remove. They distributed throughout the bulk and maintained minimum background levels of the two contaminants.

A HIPS sample was dominated by the $B_{12}O_2$ structure, giving a good reference B 1s spectrum of this structure. Three components were clearly resolved and correlated to the boron atoms not directly bonded to oxygen, bonded to oxygen in the $B_{12}O_2$ structure, and in the B_2O_3 structure respectively.

All the DIBD films were identified to be X-ray amorphous, even the substrate temperature was as high as 850^oC. Some oxygen and carbon contaminants in the target were co-sputtered during deposition, resulting in a residual oxygen content of 4.2 - 4.3 at, %, and residual carbon content in the range of 13.2 – 22.3 at. %. The oxygen content in the films was successfully adjusted from 4.2 to 19.6 at. % by increasing the assist oxygen ion flux. A great portion of the boron atoms is not bonded to oxygen, and is responsible to the appearance of the B^1 component. The increase in the oxygen content enhanced the growth of two suboxide phases, as illustrated by the behavior of the B^2 and B^4 components. The DIBD films are distinctive to show the emergence of the B^4 component, which is related to the generation of a suboxide structure in which the boron atoms are bonded to the oxygen atoms having coordination number less than 3. The mechanism of DIBD process favors the formation of this phase.

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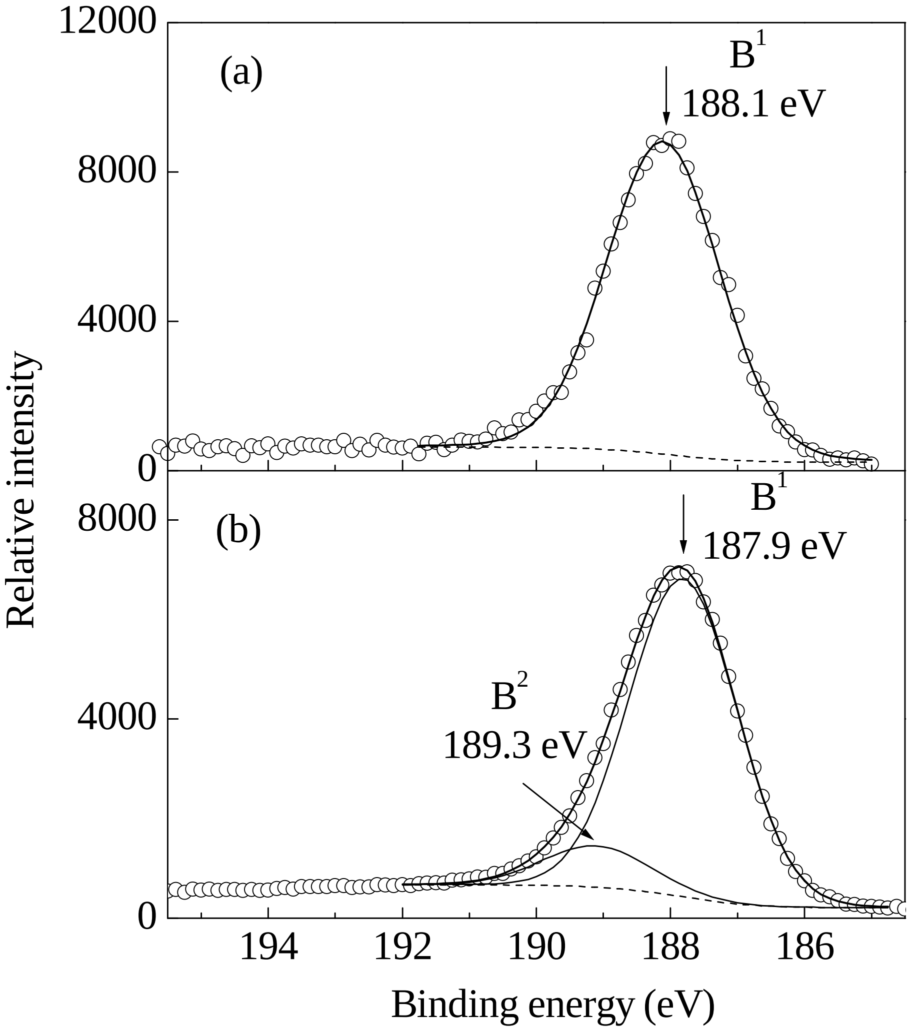


Fig.1. B 1s photoelectron spectra of polished crystalline boron collected (a) just after and (b) at 1000 min after sputter-cleaning.

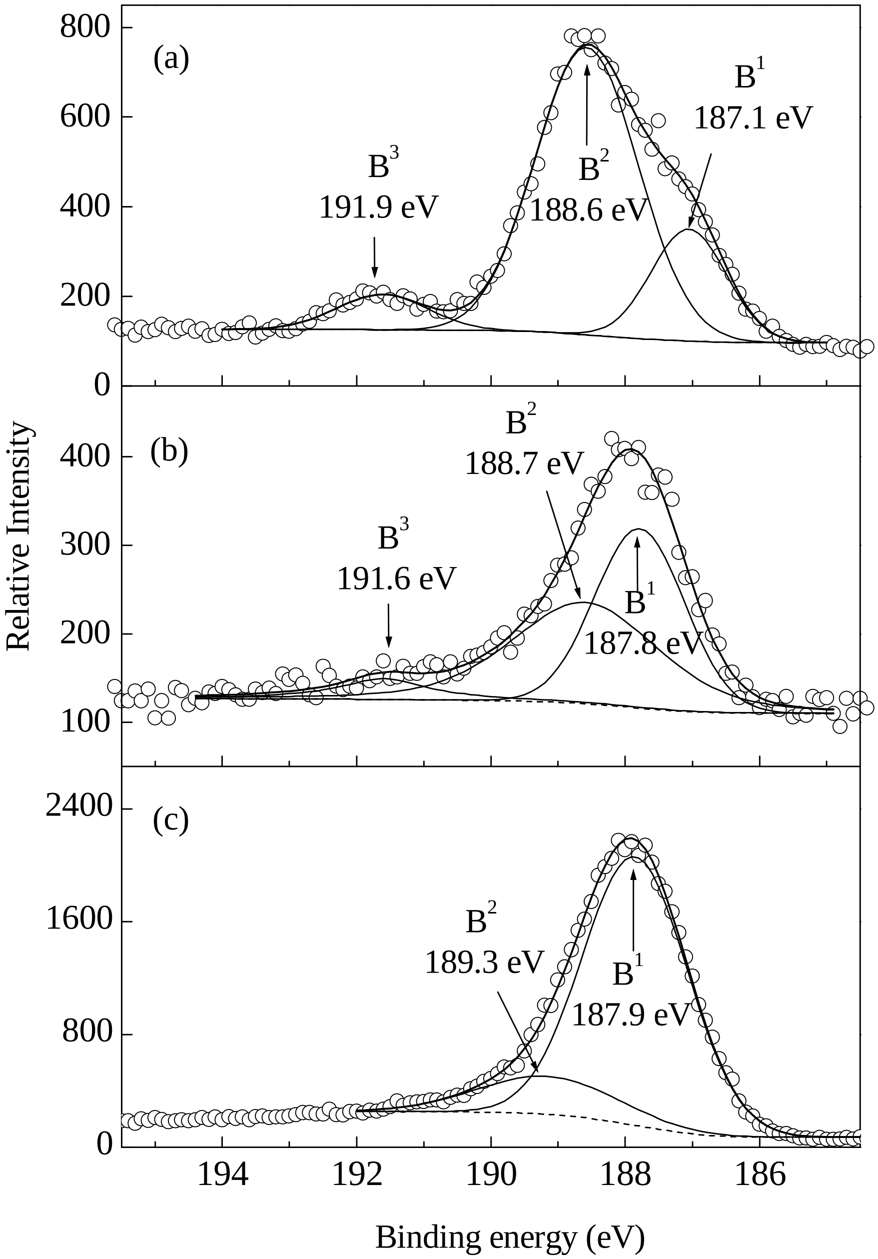


Fig. 2. B 1s spectra of the boron target material recorded
(a) before, (b) after sputtering by 2 keV Ar+ for 20 min, and
(c) after further sputtering with 4 keV Ar+ for 20 min.

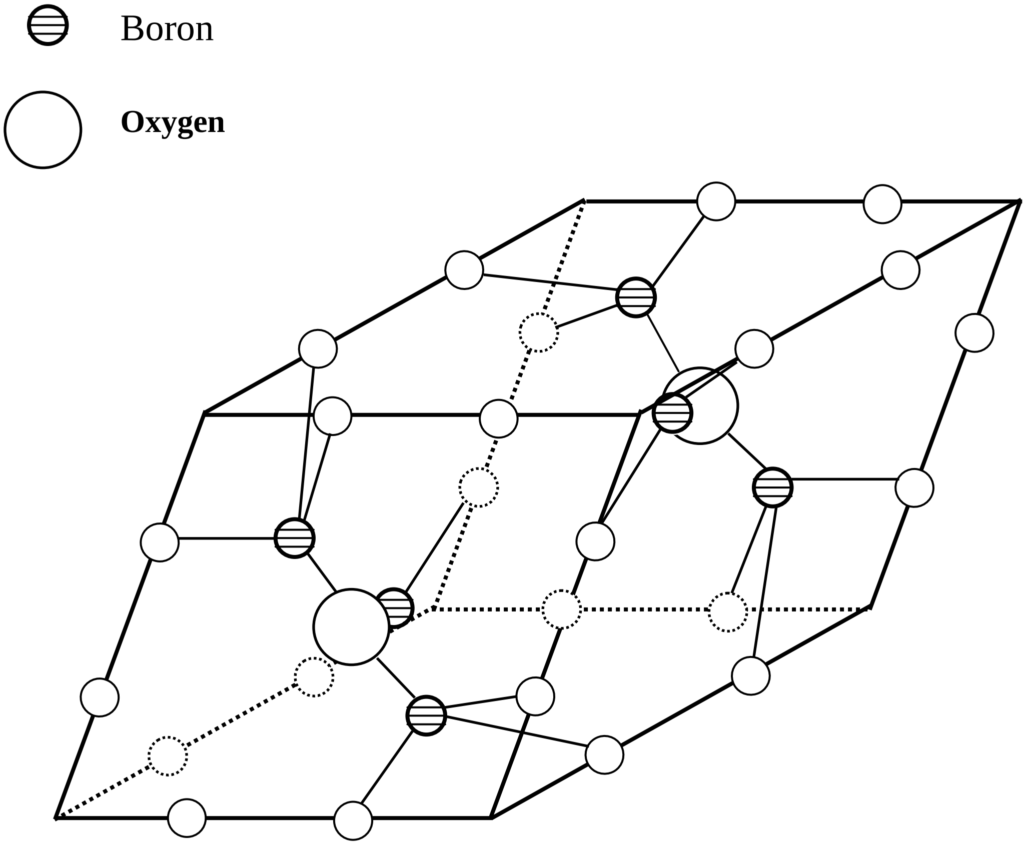


Fig. 3. The unit cell of $B_{12}O_2$.

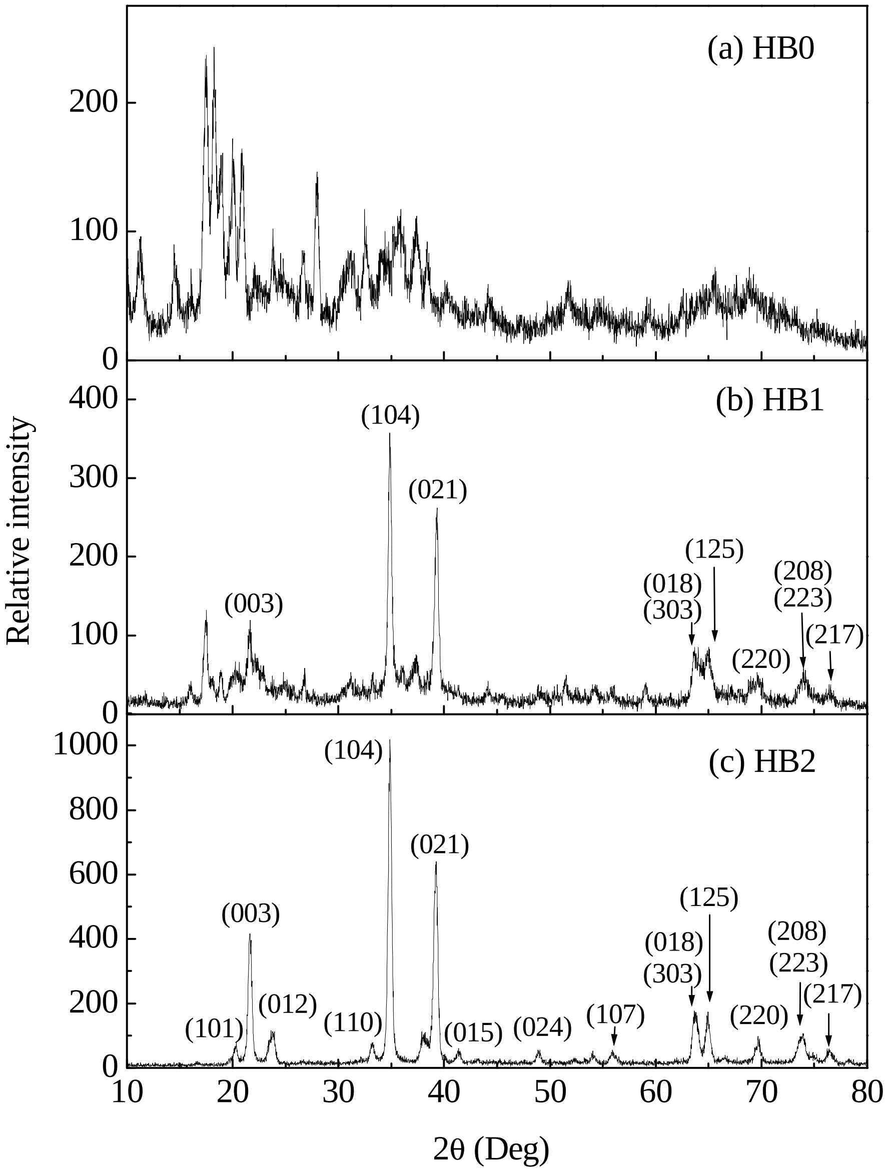


Fig.4. XRD patterns of (a) HB0, (b) HB1 and (c) HB2.

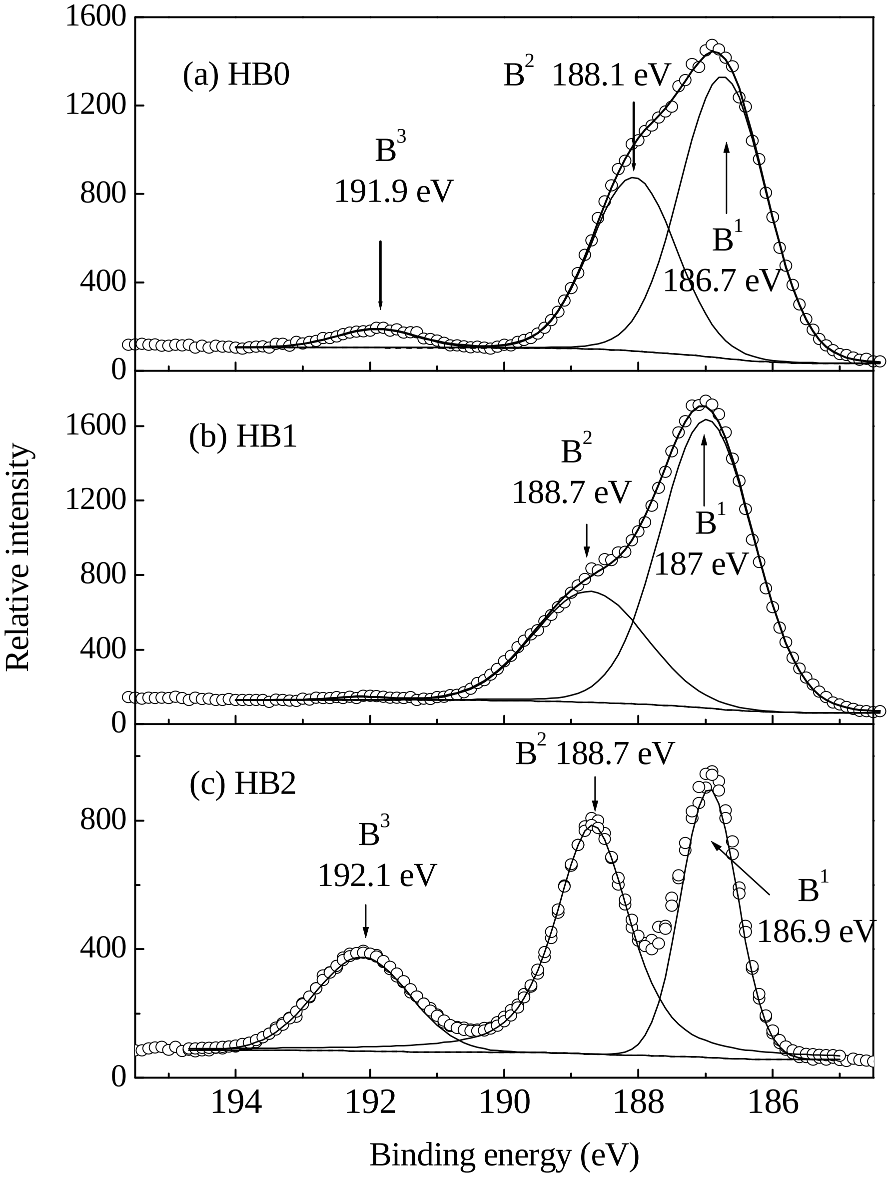


Fig. 5. B 1s spectra of (a) HB0, (b) HB1, and (c) HB2.

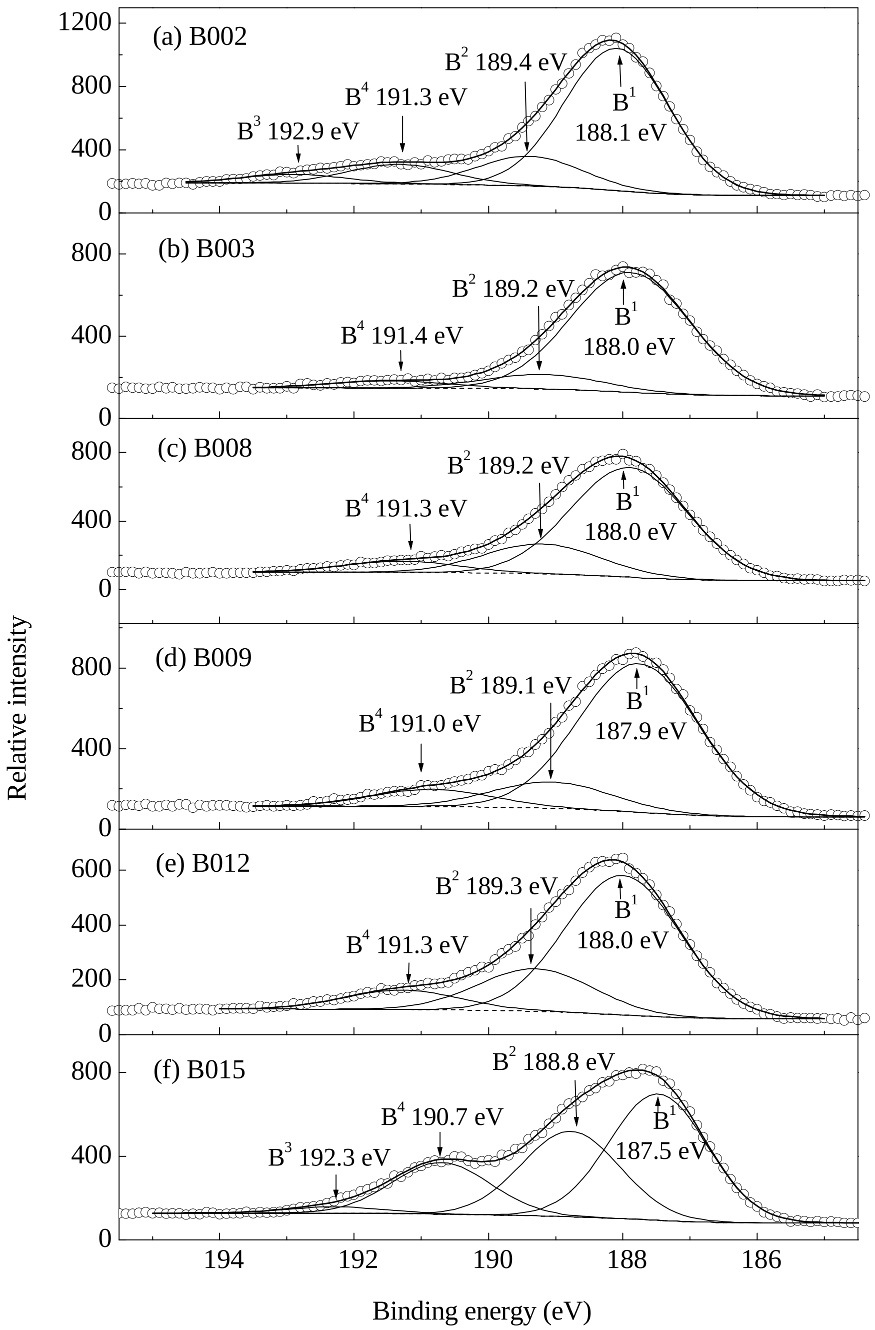


Fig. 6. B 1s spectra of the DIBD B and BxO films before sputter-cleaning.

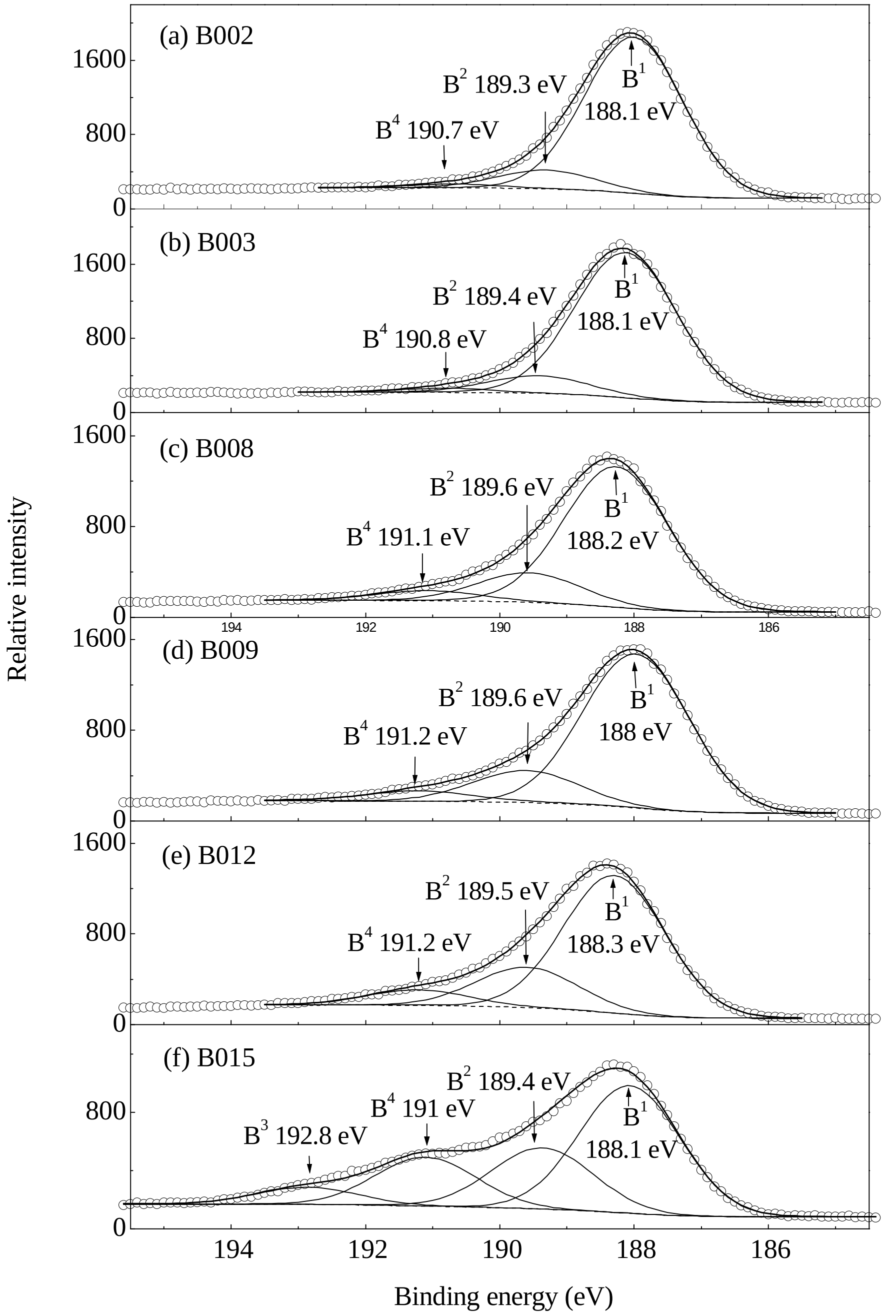


Fig. 7. B 1s spectra of the DIBD B and BxO films after sputter-cleaning.

B. APPENDICES

Appendix 1   Most relevant data in the literature and insights from them

After his meeting with Professor Lau, Mark reviewed again the relevant data in the literature on boron and boron oxides and found the following two articles most useful:

Moddeman W.E., Burke A.R., Bowlin W.C., and Foose, D.S. "Surface oxides of boron and $B_{12}O_2$ as determined by XPS", *Surface and interface analysis* **14**: 224-232, 1989.

Foo W.C., Ozcomert J.S., and Trenary M. "The oxidation of the β-rhombohedral boron (111) surface", *Surface Science* **255**: 245-258, 1991.

In addition, from an article by K. Kimura (Mater. Sci. Eng. **B19**, 67, 1993), Mark confirmed that indeed β-rhombohedral boron is a semiconductor with a bandgap of 1.6eV and in the bandgap there are commonly an acceptor band at 0.2eV and donor bands at 0.8, 1.0, 1.2, and 1.4eV above the valence band maximum.

By reading some articles of Professor Lau (Surface Science 245, 345(1991); Appl. Phys. Lett. 60, 1108(1992)), he learned the proper procedures for XPS studies of semiconductors with different Fermi level positions, band bending, and surface charging conditions. He knew that he had to repeat some of his experiments, with a special attention to having good electrical connection between his sample and the spectrometer and to paying attention on intentional and unintentional surface charging during XPS measurements.

Appendix 2 Mark's results by repeating some of his experiments

In this revision of his experiment, Mark first conducted Fermi level position measurements of his samples. To do this, he collected the experimental valence band spectrum of a gold reference sample, with a spectrometer pass energy of 60eV. The specification of spectrometer pass energy is very important because this fixed the spectrometer spectral resolution and thus the onset position of the valence band photoemission. With this experimental condition, the onset of the valence band of gold was located at -0.49eV, with the zero point of the energy scale always calibrated by Au $4f_{7/2}$ at 84.0eV. Then he recorded the following spectra (Fig. 1 Data from sputter-cleaned boron crystal; and Fig. 2 Data from the boron crystal after surface oxidation):

Fig. 1

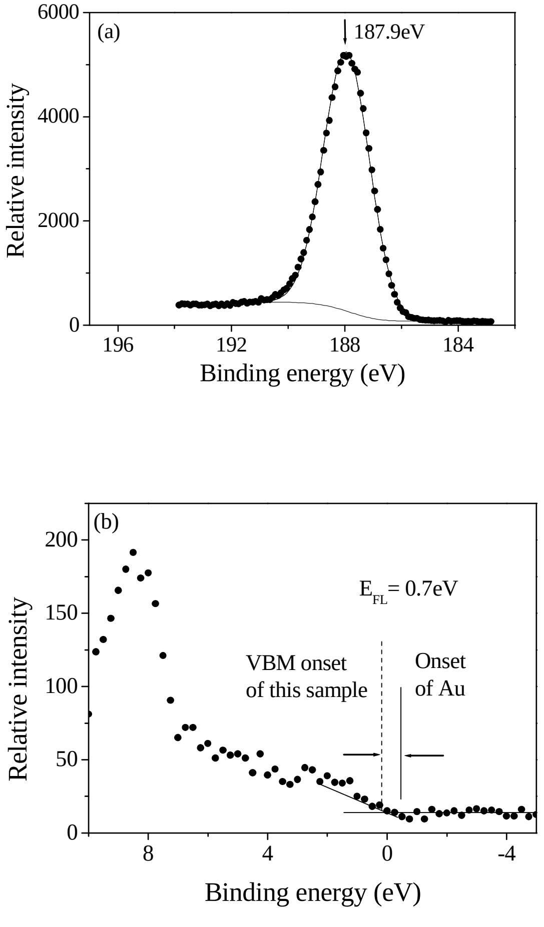
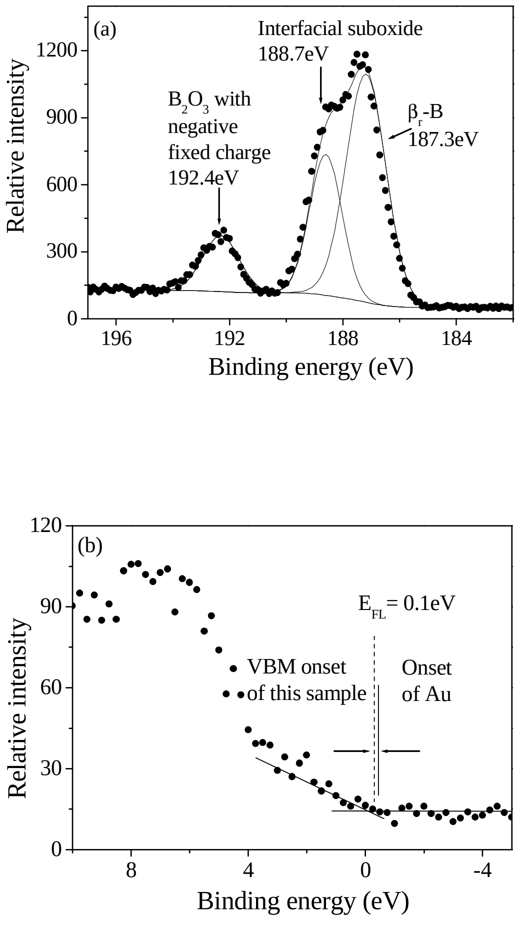


Fig. 2



C. CASE QUESTIONS

Assuming that you are Mark Hui, prepare a Power-Point presentation to explain to Professor Lau your mistakes in the last meeting and your new set of results and interpretations. Particularly, you should expect Professor Lau to keep drilling you on the following XPS concepts:

1. What are the physical meanings of binding energy in XPS and how are they measured?

2. Why do the B 1s binding energy data of elemental boron from your samples vary by so much even the data are all measured with the same spectrometer? Why do the data for pure boron crystals reported by different research groups differ by so much?

3. Is there a universal measurement method by which these discrepancies can be prevented?

4. Can you explain all the B 1s data in the case in a consistent manner?

Your small discussion group is required to answer these four questions in class. Perhaps each member presents the answer for one of the four questions. However, all members should fully understand the answers of the group. When a group does its presentation, other students will act as Professor Lau, and are expected to make relevant and intelligent comments.

HINTS FOR SELF-PREPARATION

A. From the data in Appendix 2 and the bandgap information in Appendix 1, estimate the Fermi level positions of the samples in Figs. 1 and 2.

B. With your answer from Question A, explain the difference in B 1s binding energy between the two samples

C. Given that the B 1s binding energy for B_2O_3 with its Fermi level in midgap and without any fixed charge is 193.6eV, explain why the B 1s binding energy of B_2O_3 in Fig. 2 of Appendix 2 is much lower than 193.6eV.

D. With your answer from Question C, explain why the Fermi level positions for the two boron samples in the near-surface region probed by XPS are different.

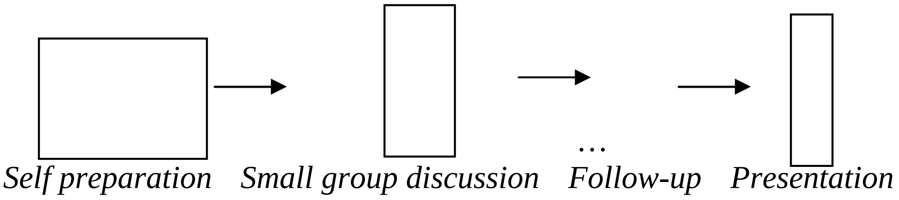
E. Read the article by Moddeman *et al*. and judge if their binding energy calibration approach by fixing B 1s of B_2O_3 at a constant is appropriate.

F. In the first paragraph on p. 227 of the article by Moddeman *et al.*, they mentioned that they decreased the X-ray power and measured the B1s spectra. Why did they decrease the X-ray power?

G. Read the article by Foo *et al*. and find out what binding energy calibration approach did they use. Taking this into account, present a consistent picture of all B 1s binding energy data in these two articles, and Mark's old and new reports.

D. PROCEDURE

Each group is required to give a presentation with less than seven PowerPoint slides for this case.



Individual preparation (60 minutes)

The assignment helps you understand the case situation. You are required to do them and submit your draft to the TA or instructor prior to your small group meeting. Please mark the submission date on your draft.

Advanced reading

- **X-ray photoelectron spectroscopy (XPS)**

The principle of XPS can been found from: http://www.phy.cuhk.edu.hk/course/surfacesci/index2.html

Small group discussion (60minutes)

After the discussion, each group is required to prepare for a presentation.

How to do a presentation:

- **Individual study-group presentation in class**. Each member should answer one of the four case questions in the presentation but the presentation should be coherent. Every member needs to fully understand the entire presentation of the group. Remember the presentation skills which we covered in class earlier. The PowerPoint presentation should be well designed and clearly explained, so that the class can understand it. The presentation should not take for more than 10 minutes.

- **Q&A sessions**. Every member in the same group may be called up to answer questions. Their individual performance will add up to the score of the group.

- **Good floor responses.** All students are expected to listen attentively to other's presentations, questions and opinions, and to make their own opinions known.