# Influence of Tilt Angle on Peak Profile in AES Measurement of Insulator

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AES analyses were carried out for insulator samples, to find the proper measurement condition. It was found that the peak shapes are distorted when insulator samples were measured by using a scanned beam under the high tilt angle of specimen. This distortion was disappeared by using a defocused beam. When an insulator sample is measured by AES, defocused beam can be used under the high tilt angles.

## 1. Introduction

Auger Electron Spectroscopy (AES) is commonly used in surface and depth profile analyses being combined with ion etching. A weak point of AES is a measurement for insulator samples. There is a case that cannot be measured in the specimen of insulator because of charging. Therefore, a variety of techniques have been carried out for conditions of, for example, large tilt angles of specimen, low accelerating voltages, small current density, and so on [1,2].

One of the activities on the Inorganic Materials Group of SASJ is to find the appropriate measurement condition of AES analyses for insulator samples. It is found that Auger peak shape would be changed by the measurement conditions. In this paper, we described the relation between peak shapes and measurement conditions.

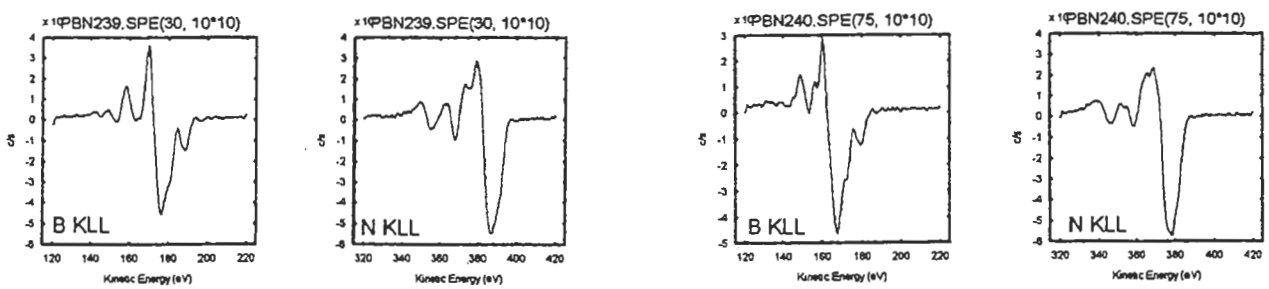
## 2. Experimental

As an insulator sample, a pyrolitic BN is used. This sample has a feature of cleavage, and fresh surface is available easily. Accordingly, it is possible to measure the surface without contamination of C and O, even in a conventional surface analysis apparatus by introducing the specimen immediately into the measurement chamber after cleavage.

Measurement conditions are as follows. In these experiments, a PHI650 Scanning Auger Microprobe was used. Specimen current was fixed about 100nA. In this paper, results only obtained by the PHI650 are shown. Similar results were also obtained by other apparatuses, but not shown here.

Accelerating voltage: 5 kV,
Specimen current: about 100nA,
Tilt angle: 30 and 75 degrees,
Measurement mode: spot and area scan

In these measurement conditions, AES spectra of B KLL and N KLL were measured and their peak shapes were investigated. In addition, Auger spectra measured by conventional XPS were also used as reference spectra that include very little charging effects.




(a) Tilt angle = 30 degrees (b) Tilt angle = 75 degrees

Figure 1. AES spectra of B KLL and N KLL obtained under the tilt angle of 30 and 75 degrees. Measurement mode is area scan (10x10um).

## 3. Results and discussion

### 3.1 Influence of tilt angle

Figure 1 shows the results under area scan mode (scan area: 10x10 um). Comparing the peak shapes of spectra, these are particularly different in the top peak on both B KLL and N KLL. In addition, the peak width of tilt angle=75° is broader than that of tilt angle=30°.

The most commonly used electron energy analyzer for AES is a cylindrical mirror analyzer (CMA). CMA has a characteristic that the depth of focus is small. It is important to place the sample at the focal point of the analyzer exactly during analysis.

When the electron beam is scanned, the actual scan area on the sample surface is different for the tilt angle of 30° and 75 °. Therefore, there would be a considerable deviation from the proper focal point of CMA between tilt angle 30 ° and 75 ° (Fig.2).

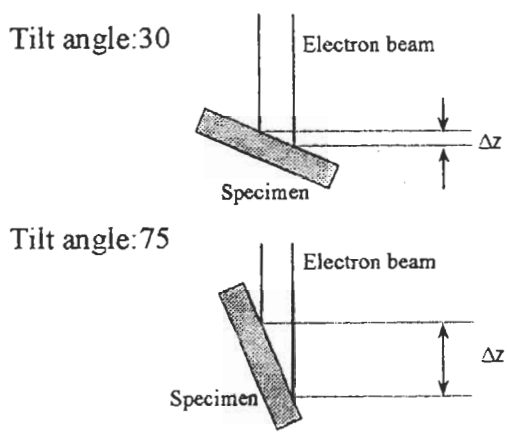


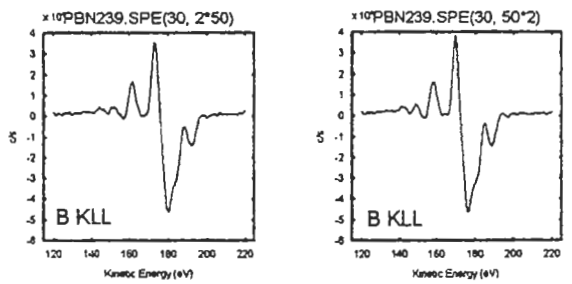

Figure 2. Deviation from the focal point of CMA between the tilt angle 30 and 75 degrees. Deviation at tilt angle of 75 degrees is larger than that of 30 degrees.

Next, influence of deviation from the focal point of CMA was investigated.

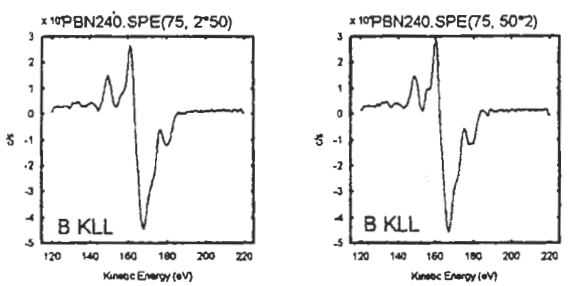
### 3.2 Influence of scan area

AES measurements were carried out on two kinds of area scan mode. One is scanned a long parallel to the tilt axis, the other is scanned a long perpendicular to the tilt axis. In these cases, each scan area was equal 2x50 and 50x2 um, respectively. Other conditions are the same.

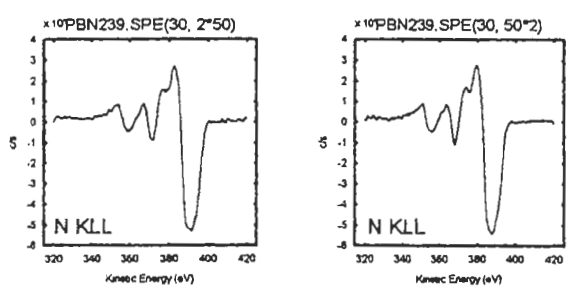
Figure 3 shows these measurement results. Either result is the same. Accordingly, there is no problem in measurement whichever scan mode may be used. It is necessary to consider the other reason of peak shape change.



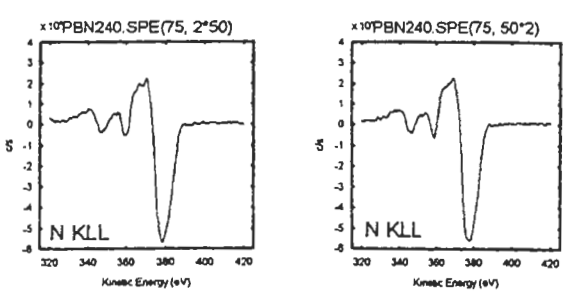

(a) B KLL spectra under the tilt angle = 30 degrees, scan area = 2x50um(left), 50x2um(right)




(b) B KLL spectra under the tilt angle = 75 degrees, scan area = 2x50um(left), 50x2um(right)




(c) N KLL spectra under the tilt angle = 30 degrees, scan area = 2x50um(left), 50x2um(right)

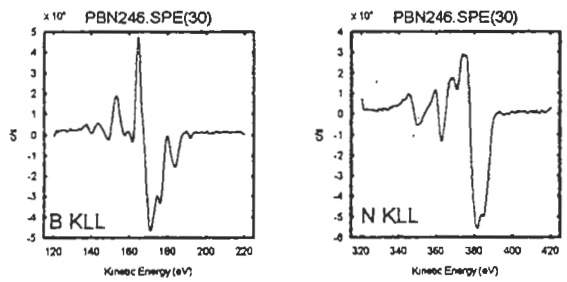



(d) N KLL spectra under the tilt angle = 75 degrees, scan area = 2x50um(left), 50x2um(right)

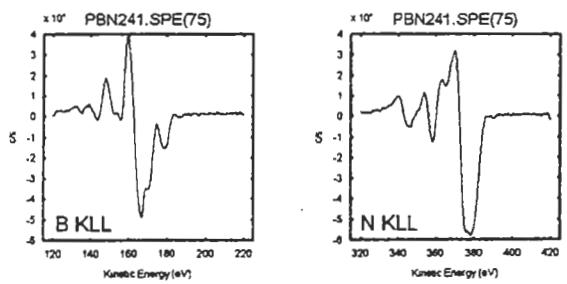
Figure 3. Comparison of AES spectra measured under the two types of scan area (2x50um and 50x2um).

### 3.3 Comparison of spot mode and scan mode

In order to lower the current density, the electron beam is usually scanned. When the electron beam is scanned, the beam pauses at both ends of the scan area (PHI650). Therefore, charging is different between the intermediate and the end of the scan area in an insulator specimen. Then, AES spectra were obtained under the defocused beam on spot mode and focused beam on scan mode, and these spectra were compared. These spectra are shown in Fig.4. The spectra obtained under the defocused beam are similar for the both tilt angles of 30° and 75°. While the spectra for focused scan mode are different as mentioned above.




(a) Tilt angle = 30 degrees




(b) Tilt angle = 75 degrees

Figure 4. AES spectra of B KLL and N KLL obtained under the defocused beam on spot mode.

### 3.4 Comparison of Auger spectra between from X-ray excitation and electric excitation

Auger spectra with conventional XPS are shown in Fig. 5. These spectra can be considered to be typical ones that include very little charging effects. These peak shapes are similar to ones obtained under the tilt angle of 30° and 75° with the defocused beam. From these results, when an insulator sample is measured by AES, it can be feasible to use defocused beam at the tilt angle of 75°.

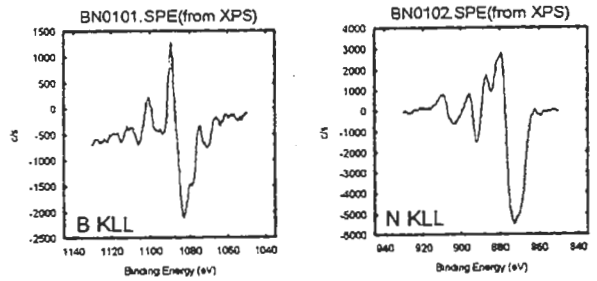



Figure 5. Auger spectra with conventional XPS shown as differential spectra.

### 4. Conclusion

As a result it was confirmed that the peak shapes are distorted when insulator samples were measured by using scanned beam under the tilt angle of 75°. This distortion was not observed by using defocused beam. It may be concluded that when an insulator sample is measured by AES, defocused beam at high tilt angles is advisable.

### References

[1] Handbook of Auger Electron Spectroscopy (Third Edition), edited by C.L.Hedberg p.10, Physical Electronics, Inc. (1995)

[2] Jitsuyou Auger Denshi-Bunkoho (in Japanese), edited by R. Shimizu and K. Yoshihara, Kyoritsu Shuppan, Tokyo, (1989)

査読者との質疑

査読者：後藤敬典氏(名古屋工業大学)
荻原俊弥氏(ジャパンエナジー ARC)

後藤、荻原：
ピークシフト量に関しては、具体的な数値を記述すべきでしょう。

著者：
今回の測定に用いた試料では、本来のピーク位置が不明ですのでピークシフトに関しては言及しないことにして、ピーク形状の変化のみの記載に変更いたしました。

後藤：
ＸＰＳ測定では、チャージアップ対策を行っているでしょうか。
著者：
非単色Ｘ線を用いての測定ですので、特に行っておりません。

後藤：
微分スペクトルはどの方法で得たものでしょうか。
著者：
装置付属のデータ処理ソフトを用いた数値微分により微分スペクトルを得ています。