

# Analysis of Wide-gap Semiconductors with Superconducting XAFS Apparatus

S. Shiki<sup>\*a</sup>, N. Zen<sup>a</sup>, N. Matsubayashi<sup>a</sup>, M. Koike<sup>a</sup>, M. Ukibe<sup>a</sup>, Y. Kitajima<sup>b</sup>, S. Nagamachi<sup>c</sup>, and M. Ohkubo<sup>a</sup>

<sup>a</sup> Advanced Industrial Science and Technology (AIST), Japan

<sup>b</sup> High Energy Accelerator Research Organization (KEK), Japan

<sup>c</sup> Ion Technology Center Co. Ltd., Japan

(Received 17 December 2012; accepted 1 24 December 2012)

## Abstract

Fluorescent yield X-ray absorption fine structure (XAFS) spectroscopy is useful for analyzing local structure of specific elements in matrices. We developed an XAFS apparatus with a 100-pixel superconducting tunnel junction (STJ) detector array with a high sensitivity and a high resolution for light-element dopants in wide-gap semiconductors. An STJ detector has a pixel size of 100  $\mu\text{m}$  square, and an asymmetric layer structure of Nb(300 nm)-Al(70 nm)/AlOx/Al(70 nm)-Nb(50 nm). The 100-pixel STJ array has an effective area of 1  $\text{mm}^2$ . The XAFS apparatus with the STJ array detector was installed in BL-11A of High Energy Accelerator Research Organization, Photon Factory (KEK PF). Fluorescent X-ray spectrum for boron nitride showed that the average energy resolution of the 100-pixels is 12 eV in full width half maximum for the N-K line, and The C-K and N-K lines are separated without peak tail overlap. We analyzed the N dopant atoms implanted into 4H-SiC substrates at a dose of 300 ppm in a 200 nm-thick surface layer. From a comparison between measured X-ray Absorption Near Edge Structure (XANES) spectra and ab initio FEFF calculations, it has been revealed that the N atoms substitute for the C site of the SiC lattice.

*Keywords* : superconducting tunnel junction detector, soft X-ray, x-ray absorption spectroscopy, XANES, synchrotron radiation

## I. Introduction

Silicon carbide is a wide-gap semiconductor that is promising as the next generation power electronics because of its high mobility and high breakdown voltage. Hot ion implementation is necessary for light element doping, but electrical activity activation is still insufficient for high-dose nitrogen doping [1]. Although reason for the low electrical activation rate is

not unknown well, it was pointed out that nitrogen atom occupy silicon site instead of carbon site in SiC crystal.

Fluorescent yield X-ray absorption fine structure (XAFS) spectroscopy is useful for analyzing local structure of specific elements in matrices. Soft X-ray region below 3 keV is important, because absorption edges of all elements are in this soft X-ray region. The fluorescent yield technique is necessary for low density impurity atoms, however the performance of conventional semiconductor X-ray detectors is insufficient because of low detection efficiency and characteristic X-ray line overwrap. Superconducting

\*Corresponding author. Fax : +81 29 861 5881

e-mail : s-shiki@aist.go.jp

tunnel junction (STJ) detector is promising for XAFS apparatuses in the soft X-ray region [2, 3].

We developed an XAFS apparatus with an STJ array detector with 100-pixels [4]. In this paper, current status of the XAFS spectrometer using an STJ detector array, and the sensitivity to low density light elements in wide-gap semiconductor are reported.

## II. Experiments

The XAFS apparatus consists of the STJ array detector, 100-channel analog/digital electronics, software for both data acquisition and monochromator controller to scan the synchrotron radiation X-ray energy. Each STJ pixel has a size of 100  $\mu\text{m}$  square and an asymmetric layer structure of Nb(300 nm)-Al(70 nm)/AlOx/Al(70 nm)-Nb(50 nm), which is effective to avoid double peak response to monochromatic X-rays in pulse height spectra [5]. The 100-pixel STJ array has an effective area of 1  $\text{mm}^2$ . The XAFS apparatus is installed in BL-11A of High Energy Accelerator Research Organization, Photon Factory (KEK-PF). Energy resolution of STJ detector was evaluated using peak width in fluorescent X-ray spectrum. Fluorescent yield XAFS spectra were obtained by measuring X-ray count for a particular energy window corresponding to an element of interest as a function of incident X-ray energies.

## III. Results and Discussion

Figure 1 shows a pulse height spectrum averaged over the entire pixels. The sample was BN powder. The incident X-ray energy was 430 eV. The spectrum shows characteristic X-ray lines of B-K, C-K, and N-K. These lines are separated clearly without overlap. The energy resolution is 12 eV in full width half maximum (FWHM) for these lines. We reported energy resolution of 12-30 eV FWHM using the same detector chip [5, 6]. The improvement of resolution is due to reduction of external electronic

noise. The measured energy resolution of this study is similar to intrinsic resolution of the STJ detector in previous study [6].

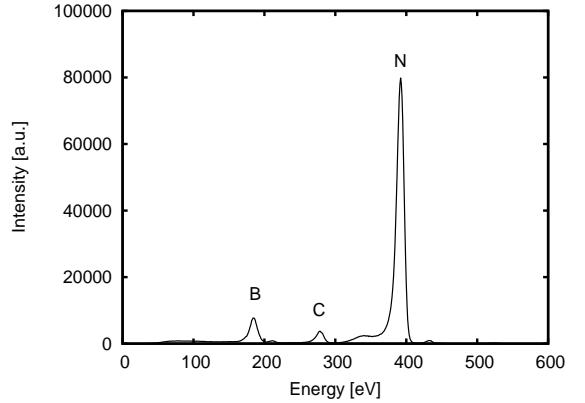


Fig. 1. Pulse height spectrum of STJ array detector for BN powder sample at incident X-ray energy of 430 eV.

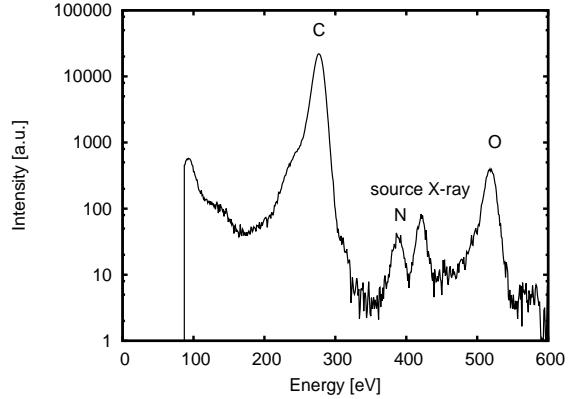


Fig. 2. Fluorescent X-ray spectrum of SiC sample with nitrogen density of 300 ppm using an STJ array detector.

Sensitivity of the XAFS apparatus is tested by measuring a SiC sample containing the nitrogen impurity at 300 ppm. Fluorescent X-ray spectrum at incident X-ray energy of 420 eV is shown in Fig. 2. The N-K line is clearly separated from the C-K line. This clear separation is impossible in semiconductor X-ray detectors because of line overlap. The N-edge spectrum of the nitrogen dopant in SiC was successfully obtained for the first time (Fig. 3). By comparing with FEFF calculations, it has been revealed that the N atoms implemented at 500 °C

substitute for the C sites in SiC even before annealing [7].

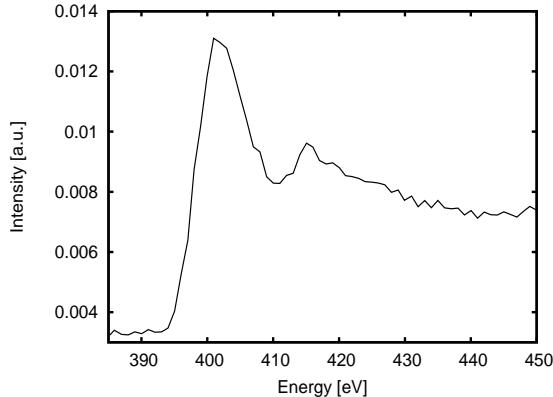


Fig. 3. N-edge spectrum of SiC sample with nitrogen density of 300 ppm acquired using an STJ array detector.

#### IV. Conclusion

We constructed XAFS apparatus using a 100-pixel STJ array detector. The STJ array detector showed an energy resolution of 12 eV in FWHM, which is enough to separate the K lines of light elements. We succeeded in measuring the N-edge XAFS spectrum of SiC at a nitrogen concentration of 300 ppm. The STJ XAFS apparatus is also useful for oxides, magnetic materials, etc. which contain light trace elements.

#### Acknowledgments

We express our sincere thanks to our group members for their continuous help. This study was supported by the Budget for Nuclear Research of the Ministry of Education, Culture, Sports, Science and Technology. The XAFS apparatus is open to users

through the nanotechnology platform program of MEXT. The measurements at KEK-PF were performed under the approval by the Photon Factory Program Advisory Committee (2009G686 and 2011G678).

#### References

- [1] T. Kimoto, and N. Inoue, “Nitrogen ion implantation into  $\alpha$ -SiC epitaxial layers”, *Phys. Stat. Sol. A* **162**, 263-276 (1997).
- [2] S. Friedrich, T. Funk, O. Dury and S. E. Labov, and S. P. Cramer, “A multichannel superconducting soft x-ray spectrometer for high-resolution spectroscopy of dilute samples”, *Rev. Sci. Instrum.* **73**, 1629-1631 (2001).
- [3] P. Fons, H. Tampo, A. V. Kolobov, M. Ohkubo, S. Niki, and J. Tominaga, “Direct observation of nitrogen location in molecular beam epitaxy grown nitrogen-doped ZnO”, *Phys. Rev. Lett.* **96**, 045504 (2006).
- [4] S. Shiki, N. Zen, M. Ukibe, and M. Ohkubo, “Soft X-ray spectrometer using 100-pixel STJ detectors for synchrotron radiation”, *AIP Conf. Ser.* **1185**, 409-412 (2009).
- [5] M. Ukibe, S. Shiki, Y. Kitajima, and M. Ohkubo, “Soft X-ray detection performance of superconducting tunnel junction arrays with asymmetric tunnel junction layer structure”, *Jpn. J. Appl. Phys.* **51**, 010115 (2012).
- [6] S. Shiki, M. Ukibe, Y. Kitajima, and M. Ohkubo, “X-ray detection performance of 100-pixel superconducting tunnel junction array detector in the soft X-ray region”, *J. Low Temp. Phys.* **167**, 748–753 (2012).
- [7] M. Ohkubo, S. Shiki, M. Ukibe, N. Matsabayashi, Y. Kitajima, S. Nagamachi, “X-ray absorption near edge spectroscopy with a superconducting detector for nitrogen dopants in SiC”, *Sci. Rep.* **2**, 831 (2012).