Microsc. Microanal. 8 (Suppl. 2), 2002
DOI 10.1017.S1431927602106258

STRUCTURE FACTOR PHASE AND AMPLITUDE MEASUREMENT IN AlN BY QCBED

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Accurate structure factors of AlN crystal have been measured by quantitative electron diffraction (QCBED). The accuracy in the measured electron structure factors is 0.1%, and the standard deviation in phase is ±1.3°. Table 1 lists all the refinement results for (002) and (004) reflections. Table 2 shows the derived x-ray structure factors compared with neutral atomic model and band theory calculations. Figure 1 gives one example of the CBED refinement. The Mott formula[1] was used to transform electron structure factors into X-ray structure factors, resulting in enhanced sensitivity to bonding for low angle scattering[1]. Accuracy for (002) after conversion becomes 0.1% in the X-ray structure factor, while the phase accuracy becomes ± 0.5° (standard deviation. Typical errors in many-beam X-ray phase measurement are 15 degrees). This data is accurate enough to distinguish between ionic, covalent and neutral atom models – the phase accuracy contains new information on bonding.

This project aims to map the charge density of AlN and to determine the role of ***s p*** electrons in the bonding of AlN. The electron diffraction measurement will help to understand the fundmental question of electronic structure of AlN wide-band semiconductor. We will combine electron measurement of low order strong reflections and x-ray measurement of high order structure for accurate charge density refinement and test band theory. Due to large extinction effects, X-ray diffraction cannot measure low order structure factors accurately[2]. The recently developed quantitative CBED method provides extinction-free measurements by using a probe smaller than one mosaic block. Using this method, Zuo et al (1999) [3] directly observed the d-orbital hole and Cu-Cu bonding in Cu_2O crystal.

Experiments were done on a Leo-912 Omega-filter TEM with an LaB_6 gun. The TEM was aligned to best condition and diffraction astigmatism corrected from aluminum powder patterns. High voltage was calibrated as 119.60 KeV using a silicon single crystal. The high quality AlN ceramic sample was used for electron diffraction. The TEM sample was prepared by ion beam thinner. Data was collected at liquid nitrogen temperature to reduce thermal diffuse scattering and contamination. Refinement of CBED patterns used the EXTAL multiple-scattering software of Zuo [4]. Thickness of 70-100nm gave enough fringes in the CBED disks to accurately determine the thickness, an important parameter refined to Angstrom accuracy. Nine measurements were done on (002) (004) reflections.

Due to their high accuracy, these measurements can be used to test many-electron approximations in band theory. FLAWP calculations (Wien2K code) [5] were used to calculate the band structure of AlN. We compare LDA and GGA96 predictions in Table 2. Multipole refinement is in progress.
Supported by NSF award DMR9973894. Thanks to Dr. Yu YD of NTNU (Norway) for supplying the AlN specimen.

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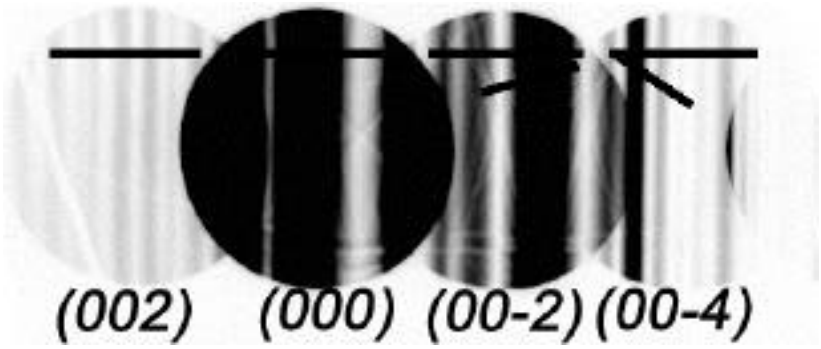
Table 1 The measured electron structure factors for U(002) and U(004) reflections. (The unit is $Å^{-2}$, temperature is 115K).

	U(002)	Phase U(002)	U(004)	Phase U(004)
QCBED	0.05066	30.4°	0.00542	25.6°
Standard deviation σ	±0.00005	±1.3°	±0.00002	±0.4°

Table 2. X-ray structure factors are transformed from electron structure factors. (temperature factor are fully deconvoluted. Unit of x-ray structure factor is electrons/unit cell).

Model	F^x(002)	Phase F^x(002)	F^x(004)	Phase F^x(004)
QCBED measurement	21.48±0.02 0.1%	25.6°±0.53°	8.73±0.02	4.67°±0.13°
Neutral Atom	21.13	25.4°	8.63	6.42°
GGA96	21.70	27.8°	9.00	7.89°
LSDA	21.69	27.76°	8.96	7.93°

Figure 1. An example of QCBED refinement on (002) (004) reflections of AlN. A. Experimental CBED pattern. B. Best fit of the rocking curve indicated in A.



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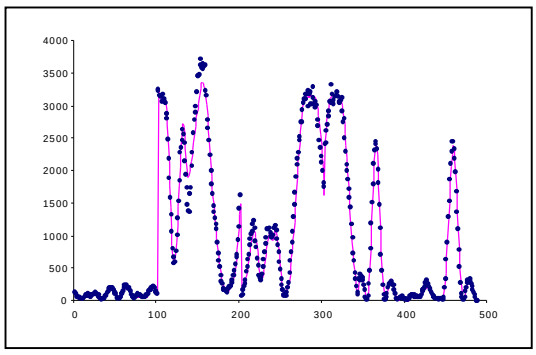

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https://doi.org/10.1017/S1431927602106258 Published online by Cambridge University Press