Dislocation Analysis by means of the Weak-Beam Dark-Field (WBDF) Method

1. Introduction and Motivation:

When analysing dislocations in a crystal, it can be most important to determine the type of each dislocation visible in a TEM image. For this purpose it is necessary to determine the Burgers vector of a dislocation in order to decide if the dislocation is a pure screw, pure edge or a mixed dislocation. The imaging of dislocations in a TEM is predicated on the diffraction contrast by exploiting the DF method under special conditions. By using the WBDF method it is possible to image the dislocation lines and to determine the Burgers vector. In the following the WBDF technique is described.

2. Basics of the WBDF Method:

In a WBDF image dislocations can be visualized as sharp bright lines, whereas the perfect parts of the specimen are not visible and appear dark. Thereto the sample and the incident beam must satisfy specific tilt conditions. The general exposure technique is shown in figure 1.

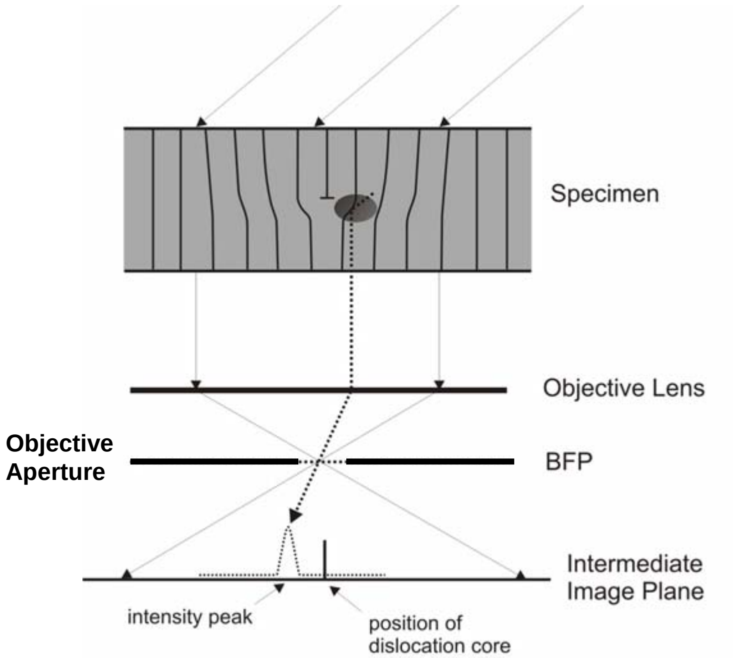


Figure 1: Path of rays in the TEM for the WBDF method using an edge dislocation as an example. For the defect-free area the incident beam is far away from the exact Bragg condition (large excitation error). In the area near the dislocation core (black mark in the specimen) the Bragg condition is exactly satisfied. This is caused by the bending of the hkl plane near the dislocation core. In the WBDF image the dislocation line appears as a bright line to dark background.

In principle, the WBDF technique is an on-axis dark field imaging method by using a diffracted beam with large excitation error for the defect-free sample area. Thus, the defect-free sample area appears dark because of the weak diffraction intensity. However, close to the dislocation core the hkl plane is bended back into the Bragg condition, which gives rise to a bright intensity peak (the dislocation line). The main challenge is to adjust the tilt conditions in the way that the excitation error of the g reflection used is close to zero only near the dislocation core where the bending of the hkl plane is most prominent. Then a very sharp dislocation line near the dislocation core becomes visible in the WBDF image.

Three conditions have to be satisfied to get a high quality WBDF image with well detectable dislocation lines:

- **Beam tilt to on-axis DF and selection of a Bragg spot by using objective aperture**
- **Right adjustment of the excitation error**
- **Two beam conditions to get high contrast**

3. Obtaining appropriate Tilt Conditions

In the following description we refer to a [110] AlN cross-section sample, whereas the principle procedure is the same for other materials. The AlN was grown in the hexagonal phase (2H AlN, wurtzite structure) on a sapphire (Al_2O_3) substrate. We want to use the 0002 reflection for the WBDF image.

1st step: Tilt the sample into an appropriate zone axis, in this case [110], as it is shown in figure 2.

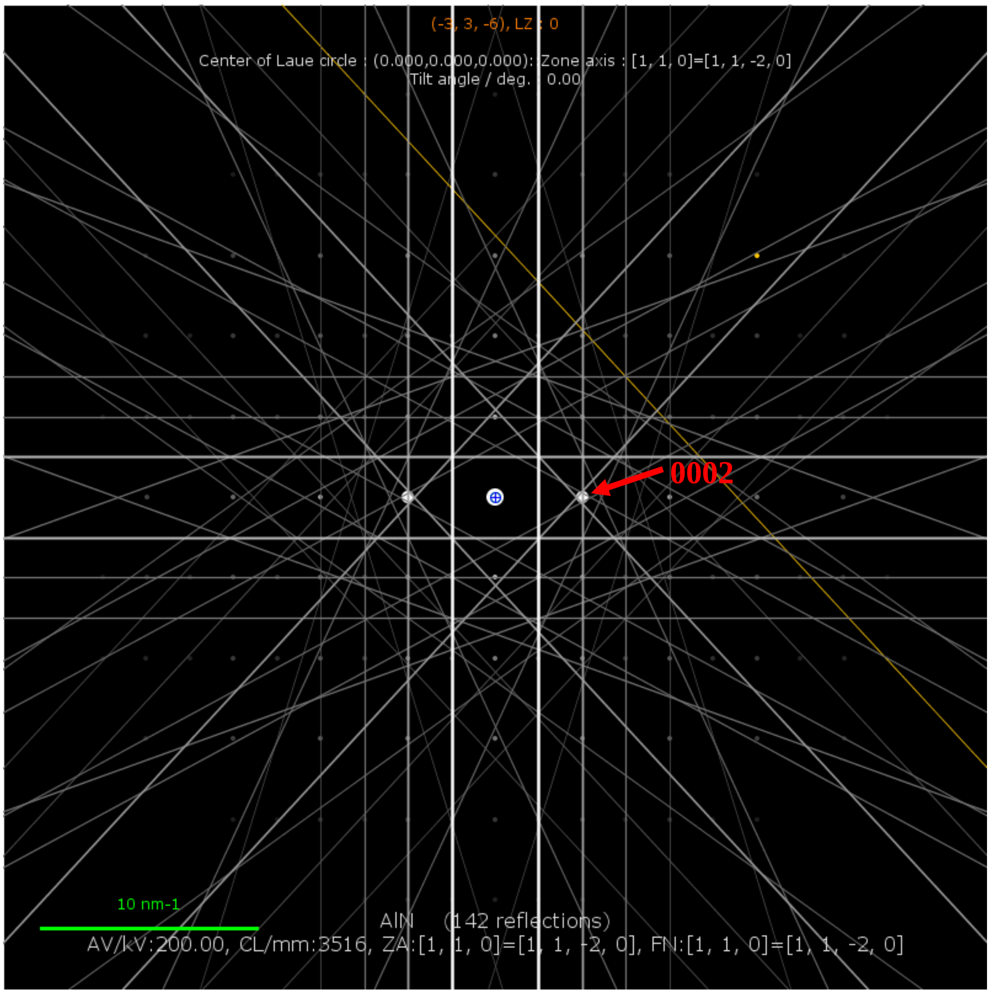


Figure 2: Calculated spot pattern of 2H AlN from the [110] zone axis. The Kikuchi lines are also shown.

2nd step: The next step is to get rid of almost all other reflections with the exception of the transmitted beam and in this case the 0002 reflection. This means we want to approach the two beam condition. For this purpose tilt the sample that way, that only that line of reflections is visible, which contains the 0002 reflection. The tilting condition is shown in Figure 3.
3rd step: Tilting to the so-called 3g condition. Normally the 3g condition is used to get high quality WBDF images. 3g condition means, that the g reflection you want to use for your WBDF image (in this case 0002) is in the optical axis, whereas the Bragg condition for the 3g refection (in this case 0006) is exactly satisfied. Then we get a relatively large excitation error for the g reflection. This condition is shown in figure 4. To reach this condition we have to proceed after step 2. First tilt the sample again, however in the opposite direction so that the g reflection satisfies the Bragg condition exactly. This means, that the g-Kikuchi line intersects the g-reflection exactly and the –g-Kikuchi line passes the zero spot. Then tilt the incident beam that way, that the g refection appears in the optical axis. The 3g condition as it is drawn in Figure 4 is then satisfied. Now the 3g-Kikuchi line intersects the 3g refection exactly and the -3g-Kikuchi line passes the zero spot.

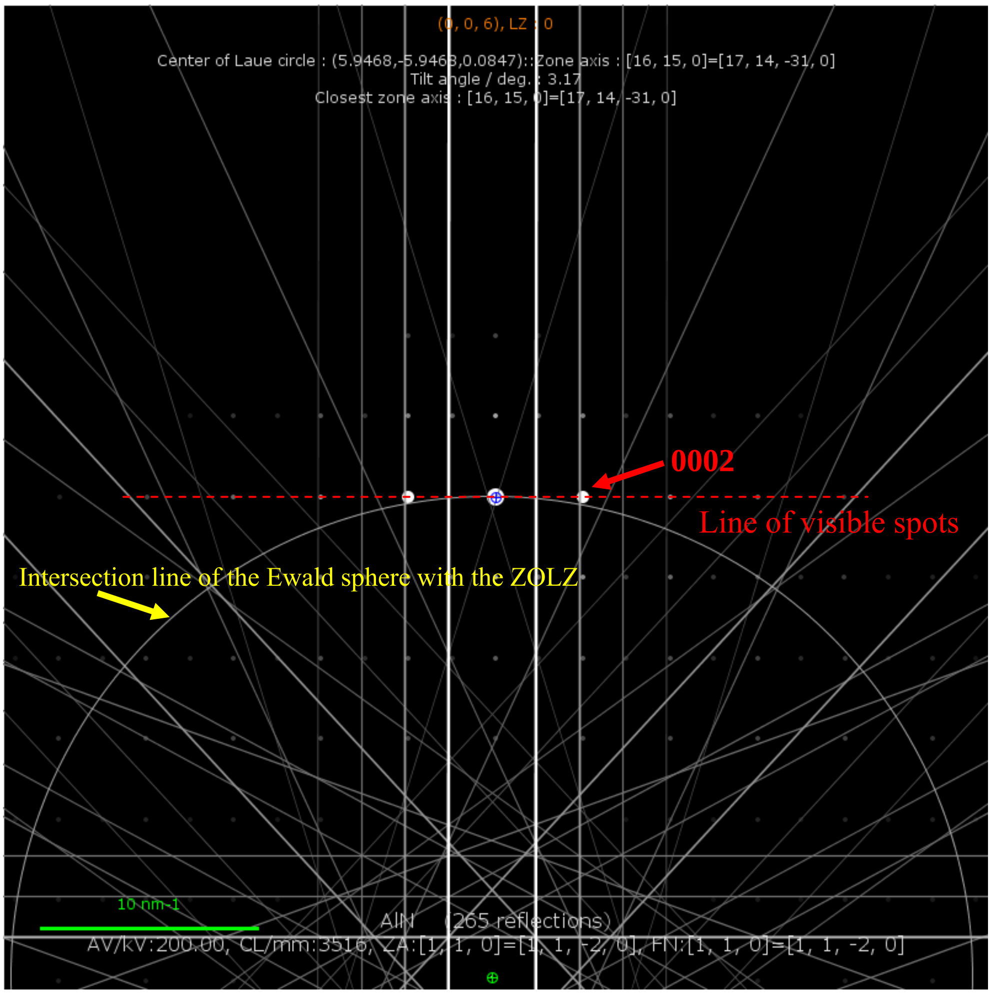


Figure 3: Tilt condition after sample tilt out of the zone axis to approach the two beam condition.

4th step: Select the g reflection with the objective aperture and put the image on the screen.

4. Dislocation Analysis and g*b Criterion

As mentioned above, a dislocation bends the lattice planes near the dislocation core. This can be described by a displacement field $\vec{R}$. The displacement field generates an additional phase shift $\sim \exp\left(2\pi i \vec{g}\vec{R}\right)$ of the Bragg diffracted beam, which leads to a diffraction contrast in the image. As a rough rule of thumb the displacement vector $\vec{R}$ is parallel to the Burgers vector $\vec{b}$.

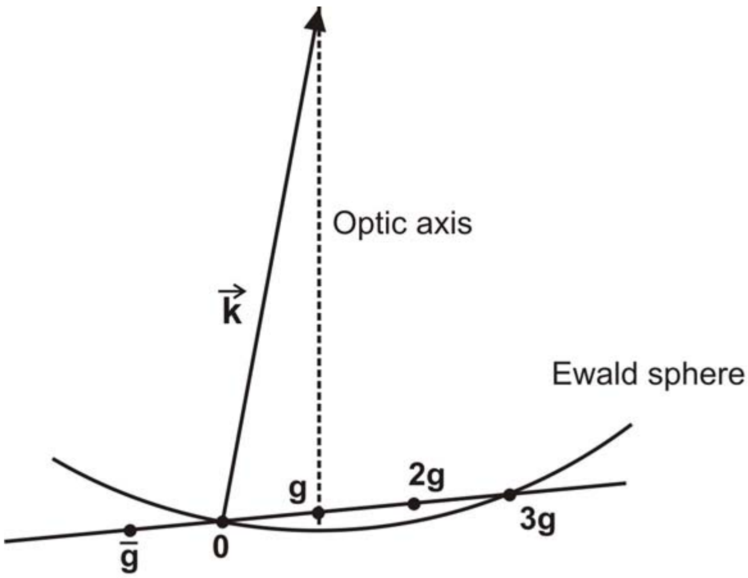


Figure 4: 3g condition for the WBDF image. The g refection is in the optical axis with a large excitation error.

You can see this relation qualitatively for the pure edge dislocation in figure 1. In this illustration the main part of the displacement near the dislocation core points in the direction of $\vec{b}$. The phase shift $\exp(2\pi i\vec{g}\vec{R})$ only comes into effect if $\vec{g}\cdot\vec{R}\neq 0$. This means, if $\vec{g}\cdot\vec{b}=0$ => $\vec{g}\cdot\vec{R}=0$ and the dislocation line is not visible, then the displacement field is parallel to the exploited hkl plane and does not change the phase. This relation is called $\vec{g}\cdot\vec{b}$ criterion. If $\vec{g}\cdot\vec{b}=0$, the dislocation line is not visible, otherwise the dislocation is visible in the WBDF image. This enables the Burgers vector determination and identification of the dislocation type by taking WBDF images of the same sample area by exploiting different g reflections.

5. Example: WBDF analysis of wurtzite 2H AlN grown on c-plane Sapphire

a) Crystal structure and growth:

AlN can be grown in the hexagonal wurtzite structure on c-plane Sapphire. This structure can be described by means of two hcp lattices, shifted along the c direction by $\frac{3}{8}c$, as it is shown in figure 5. The a-parameter of bulk AlN is 3.112Å, the c-parameter is 4.982Å.

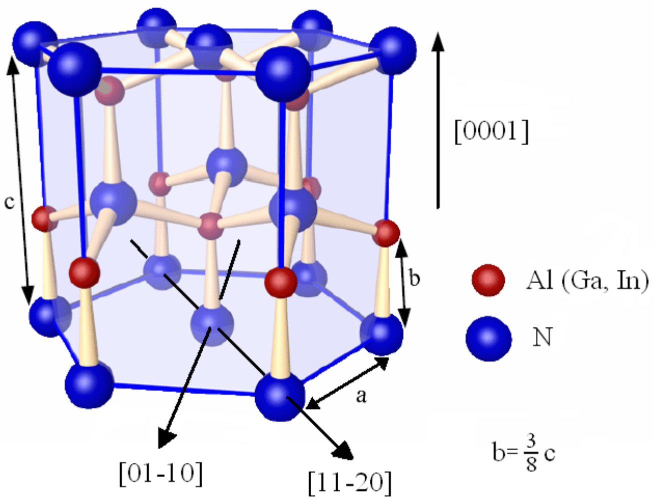


Figure 5: Hexagonal wurtzite structure of 2H AlN (α-phase)

Figure 6 shows the growth conditions of 2H AlN on c-plane sapphire from the [001] direction. The inner cell (a=2.747Å) of Al_2O_3 determines the AlN growth. This gives rise to a large lattice mismatch of -11.7%, and the AlN receives compressive stress at the AlN/Al_2O_3 interface. A strain energy in the AlN lattice is reduced by the formation of strain induced misfit dislocations, which are normally partial dislocations. These misfit dislocations can interact with each other, which leads to the formation of threading dislocations (TDs). The growth of the TDs along the epitaxial growth direction of the material is not strain induced but growth induced. Thus it is still a big challenge to grow AlN directly on sapphire.

b) Burgers vector determination in 2H group III-nitrides and g*b criterion

Depending on the crystal structure and therewith depending on the basal plane, only specific Burgers vectors are possible. The construction of the possible Burgers vectors results from the displacement of the basal plane along the Burgers vector namely that way, that we still obtain a valid stacking order after the displacement. Figure 7 shows the possible Burgers vectors in 2H group III-nitrides and their notation.

Burgers-Vectors	**Notation**	**Dislocation type**
AB,AC,BC	1/3[2-1-10]	perfect dislocation, a-type
DE	[0001]	perfect dislocation, c-type
AB+DE	1/3[2-1-13]	perfect dislocation, (a+c)-type
AF	1/3[1-100]	Shockley partial
AE	1/6[2-203]	Frank partial
FE	1/2[0001]	Frank partial

Figure 8 shows the diffraction patterns from the AlN/Al_2O_3 interface of the AlN [110] and the AlN [120] zone axis. The charts below show g*b values for the applicable g reflections. Referring to the AlN [120] zone axis pattern, only c- and (a+c)-type dislocations will be visible when using the 0002 reflection. When using the 2-1-10 refection only a- and (a+c)-type dislocations will be observable in the WBDF image. By imaging the same sample area exploiting two different refections for the WBDF image, e.g. 0002 and 2-1-10, the Burgers vectors can be determined by means of the exclusion principle.

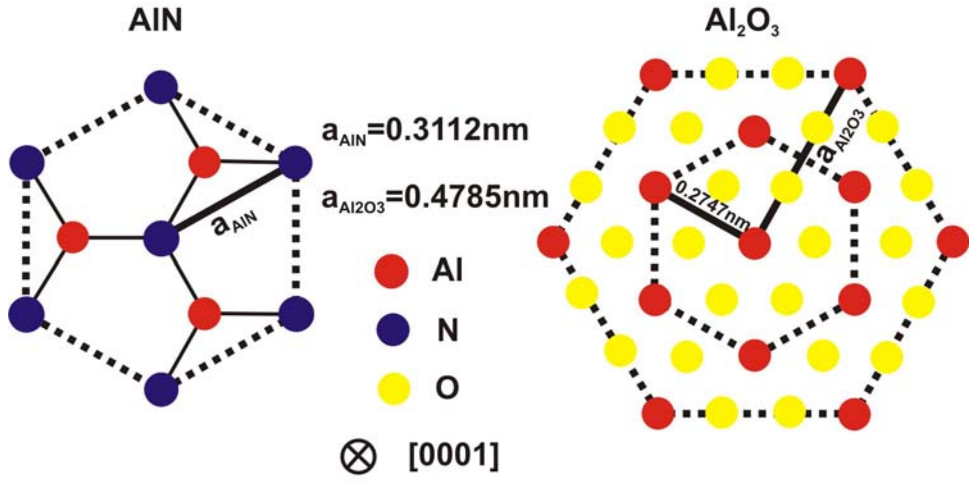
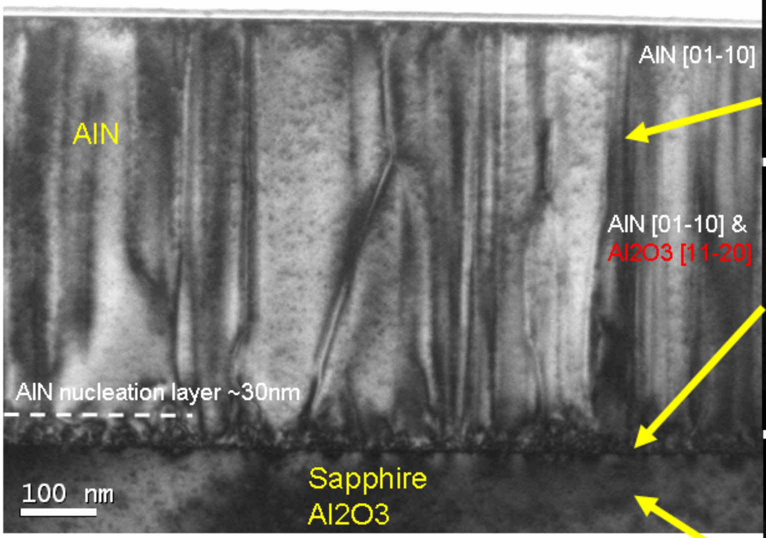


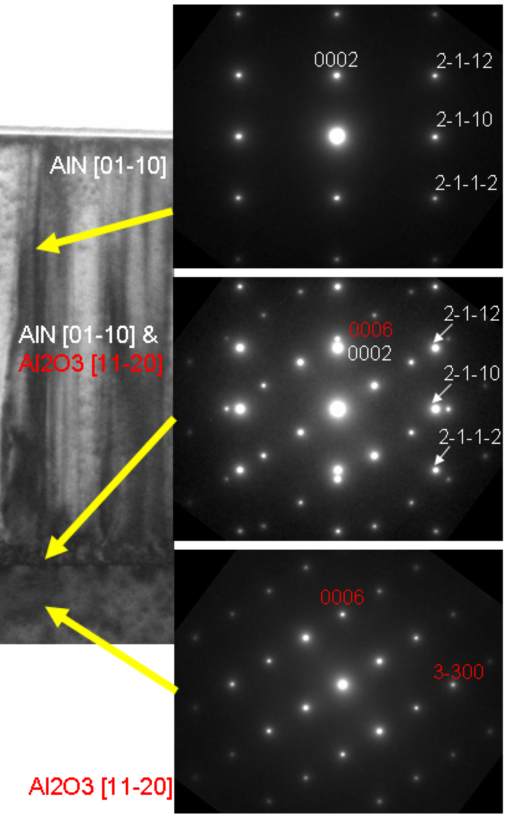
Figure 6: Unit cells of AlN and Al_2O_3 shown in the epitaxial growth direction of [0001]

c) Experimental WBDF Images and Dislocation Analysis

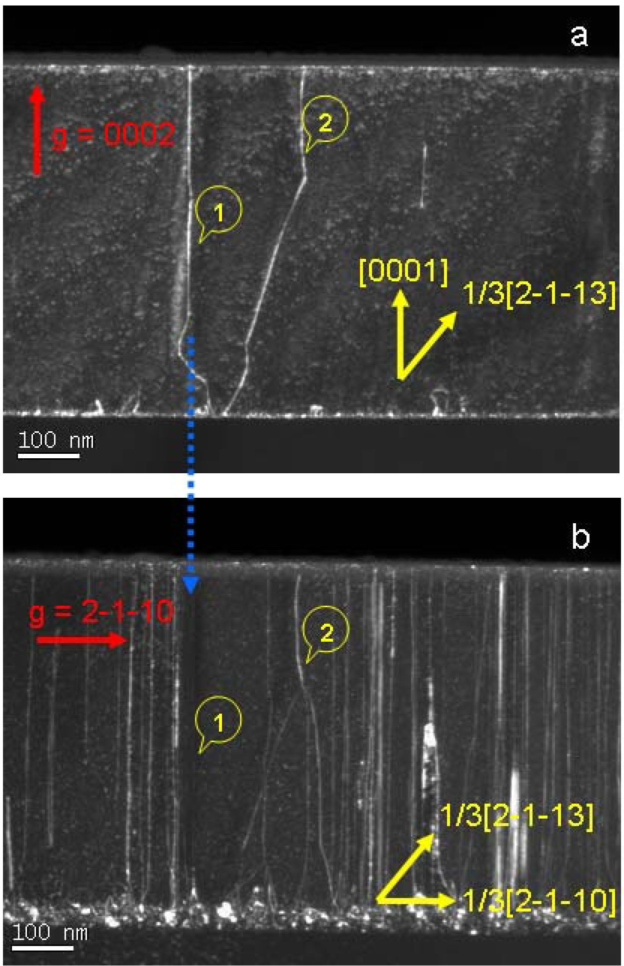
BF-Image of Sample Y1153:

AlN (~500nm) on Sapphire

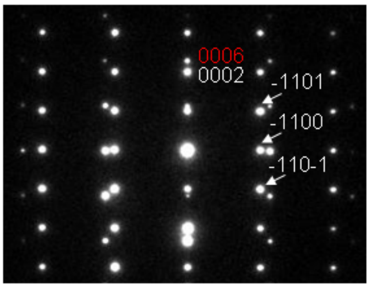




Nucleation layer ~30nm, oxygen doped

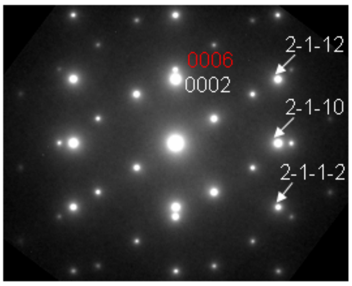


g*b – criterion: AlN [110] / [11-20] & Al2O3 [01-10]



g*b	(0002)	(-1101)	(-1100)	(-110-1)
1/3 [2-1-10]	0	-1	-1	-1
1/3 [2-1-13]	2	0	-1	-2
[0001]	2	1	0	-1

g*b – criterion: AlN [120] / [01-10] & Al2O3 [11-20]



g*b	(0002)	(2-1-12)	(2-1-10)	(2-1-1-2)
1/3 [2-1-10]	0	2	2	2
1/3 [2-1-13]	2	4	2	0
[0001]	2	2	0	-2

Figure 8: Spot patterns from the AlN/Al2O3 interface of the AlN [110] and AlN [120] zone axis. The g*b values for the applicable g reflections are also shown.