

## ScAlMgO<sub>4</sub>: an Oxide Substrate for GaN Epitaxy

E. S. Hellman, C. D. Brandle, L. F. Schneemeyer, D. Wiesmann, I. Brener, T. Siegrist, G. W. Berkstresser, D. N. E. Buchanan, E. H. Hartford  
AT&T Bell Laboratories

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### Abstract

We report the use of ScAlMgO<sub>4</sub> as a substrate for the epitaxial growth of wurzitic GaN. The low misfit (+1.8%) allows coherent epitaxy of GaN, as observed by RHEED. The congruent melting of ScAlMgO<sub>4</sub> makes Czochralski growth possible, suggesting that large, high quality substrates can be realized. Boules about 17mm in diameter are reported. We have used nitrogen-plasma molecular beam epitaxy to grow GaN epitaxial films onto ScAlMgO<sub>4</sub> substrates. Band-gap photoluminescence has been observed from some of these films, depending primarily on the deposition conditions. A 3x3 superstructure has been observed by RHEED on the GaN surfaces. Structural analysis by x-ray diffraction indicates very good in-plane alignment of the GaN films. We also report thermal expansion measurements for ScAlMgO<sub>4</sub>.

## 1. Introduction

### 1.1. Sapphire: The Standard of Comparison

The availability of an appropriate substrate for epitaxial growth is crucial to the practical application of new semiconductor materials. The factors determining appropriateness include crystallographic, physical, chemical, and economic parameters. For the wide band-gap semiconductor GaN, the most appropriate substrate for practical applications such as light-emitting-diodes (LED's) has proven to be c-plane sapphire. [1] This fact might be surprising in view of some inappropriate crystallographic parameters, but not so surprising considering its physical, chemical and economic parameters. Sapphire is strong, hard, and inert. It is available in large, inexpensive, high quality wafers from a number of vendors. However, for potential use of GaN in blue lasers, these virtues may prove to be insufficient to counteract the one drawback of sapphire: its huge lattice mismatch with GaN. The -13% misfit [a] results in a very large dislocation density in GaN epitaxial films on sapphire [2]. It is at present not understood why LED's work as well as they do in the presence of such a large dislocation density. For epitaxial growth, 13% is almost the largest misfit that can be tolerated while still getting a well-aligned film. The surprisingly high quality of GaN films that have been obtained on sapphire may be due to an interfacial AlN reaction layer. [3, [3]

### 1.2. Alternatives to Sapphire

A variety of alternatives to sapphire have been investigated over the years, [4] including Si, GaAs, NaCl, GaP, InP, W, TiO<sub>2</sub>, SiC, ZnO, MgAl<sub>2</sub>O<sub>4</sub> (spinel), and MgO. Only the last four can be considered possible improvements over sapphire.

#### 1.2.1. 6H-SiC

The mismatch is only -3.1% to GaN and -1.0% to AlN. SiC has the additional advantage, for many applications, of being conductive. Although excellent results have been obtained with epitaxial GaN films on 6H-SiC, [5] the 6-H SiC substrates are very expensive.

### 1.2.2. ZnO

ZnO has the wurtzite structure, and is only mismatched by +2.3% to GaN. Positive misfits can be compensated by the addition of In to make the (GaIn)N alloy. [6] However, ZnO crystals are not very easy to make, and ZnO is not as thermally stable as would be desired. Zn is considered a troublesome contaminant for many processing techniques, including MBE.

### 1.2.3. MgO

The (111) face of MgO is mismatched by -6.4% to GaN. The main difficulty with MgO is that the (100) face has a much lower surface energy than the (111) face, both in terms of cleavage and crystal growth, which makes the (111) face extremely difficult to prepare. For growth of zincblende GaN, MgO remains an attractive possibility, [7] although substrates larger than 1" may not be practical.

### 1.2.4. Spinel

Spinel is one of the most interesting of the substrates that have been used previously. [8] The (111) face is the low surface energy face, so it is relatively easy to prepare. The oxygen lattice is face-centered cubic, which presents a similar stacking layer along (111) as found in the wurtzite structure. Best of all, the spinel structure and spinel related structures are found in a large number of oxides. Our group has studied two different spinel type materials,  $\text{Fe}_2\text{NiO}_4$ , a -7.1% mismatch and  $\text{LiGa}_5\text{O}_8$ , a -8% mismatch. [9] Despite the diversity of the spinels, there are very few that have really good lattice matches to GaN. The oxides are mostly too small and the sulfides are all too large. The oxides that come closest are  $\text{Na}_2\text{MoO}_4$ ,  $\text{Na}_2\text{WO}_4$ , and  $\text{In}_2\text{CdO}_4$ , with mismatches +1.3%, +1.5%, and +1.3% respectively.

## 1.3. $\text{YbFe}_2\text{O}_4$ structure

As the cations in spinels get larger, the spinel structure becomes unstable with respect to other structures. One such structure-type is the  $\text{YbFe}_2\text{O}_4$  structure. [10] This structure-type is a rhombohedral layered structure, with hexagonal  $\mathbf{a}$ -lattice constants between 3.236 ( $\text{ScMgAlO}_4$ ) [11] and 3.489Å ( $\text{YFeZnO}_4$ ). [11] The structure can be considered to be a superlattice of rock-salt-like layers and wurtzite-like layers. The oxygen lattice is near to close-packed. The smallest of the known  $\text{YbFe}_2\text{O}_4$ -type materials,  $\text{ScAlMgO}_4$  is well matched (+1.8%) to the hexagonal face of wurtzite-structure GaN. The structure of  $\text{ScAlMgO}_4$  and its relation to that of GaN is depicted in figure 1.

In the rest of this paper, we will describe the preparation of  $\text{ScAlMgO}_4$  crystals, and the epitaxial growth and characterization of GaN films on these crystals.

## 2. Preparation and Characterization of $\text{ScAlMgO}_4$ Crystals

Two types of  $\text{ScAlMgO}_4$  substrate crystals were prepared and used for epitaxial growth.

### 2.1. Micaceous Platelets

Platelets as large as 1 cm across were grown by slow cooling of a stoichiometric melt. A 50g batch prepared from stoichiometric amounts of MgO,  $\text{Sc}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$  was placed in an Ir crucible. The sample was melted using RF induction heating under an  $\text{N}_2$  atmosphere. The sample was then cooled slowly to yield crystals approaching 1 cm diameter and 1 mm thick, although many of these had low angle grain boundaries. Flat flakes were peeled from the micaceous crystal mass and soldered with indium to molybdenum heater blocks for epitaxial growth.

### 2.2. Czochralski Boules

A compelling reason to consider  $\text{ScAlMgO}_4$  as a potentially practical substrate for GaN epitaxial films is that it can be grown by the Czochralski method. Thus, the techniques needed to produce large diameter, high quality crystals on an industrial scale are already well established. A melt of stoichiometric composition was prepared by mixing 44.01g MgO and 75.31g  $\text{Sc}_2\text{O}_3$  forming the mixture into a pellet, placing the pellet into a conventional iridium crucible

MgO and 75.51g Sc<sub>2</sub>O<sub>3</sub>, forming the mixture into a pellet, placing the pellet into a conventional indium crucible together with 55.68g Al<sub>2</sub>O<sub>3</sub>, and heating the charged crucible under N<sub>2</sub> in a conventional RF induction furnace. The starting materials were commercially available, and of at least 99.99% purity.

The charge was completely molten at about 1900°C. At this point, a thin iridium rod was dipped into the liquid and a button of polycrystalline ScAlMgO<sub>4</sub> was formed on the tip of the rod. The rod with solidified ScAlMgO<sub>4</sub> thereon was slowly raised (initially at 2.5 mm/hr) and rotated at 15 rpm. After about 1.5 hours the pull rate was gradually increased to 4 mm/hr. Pull conditions were regulated under computer control by maintaining weight gain to yield a boule having a neck (about 7mm in diameter, about 60mm long) that blended smoothly into the main body of the boule, about 60mm long. This was done to favor the growth of a limited number of relatively large crystals in the boule. Away from the seed end of the boule, the crystallites were large enough to separate with a razor blade into 20mm diameter near-single crystal slices. The slices were then mounted on a polishing block with black wax and polished with emery paper until they were flat on a cleavage plane. The slices were flipped and the process repeated on the reverse side, followed by polishing of the intended growth surface with LINDE A® and LINDE B® polishing compound. The surface chips rather easily, so considerable care is required during this stage, so as not to gouge the surface. After a final polish using Syton® on polishing paper, the slices were soldered to heater blocks with indium.

The substrates are somewhat flexible, but will break into flakes rather than cleave, as expected from a micaceous material. They are clear and insulating. Low angle grain boundaries were visible in some substrates

### 2.3. Thermal Expansion of ScAlMgO<sub>4</sub>

A critical parameter for the compatibility of a substrate and an epitaxial film is the thermal expansion match. If the thermal expansion coefficient of the substrate is too small, the film is liable to crack, while if it is too large, the film can peel or blister on cooling. GaN is a rather hard material and has a relatively small thermal expansion coefficient,  $5.6 \times 10^{-6}/^{\circ}\text{C}$  (in the **a** direction), which is between that of sapphire ( $7.5 \times 10^{-6}/^{\circ}\text{C}$ ) and that of silicon ( $3.6 \times 10^{-6}/^{\circ}\text{C}$ ). Most oxides tend to have larger thermal expansion coefficients. Micaceous materials such as ScAlMgO<sub>4</sub> tend to have smaller thermal expansion coefficients parallel to the cleavage planes and larger thermal expansion coefficients normal to the cleavage planes.

The temperature dependence of the unit cell parameters of ScAlMgO<sub>4</sub> was measured using a DISPLEX cryostat with beryllium windows on a diffractometer equipped with graphite monochromated Cu K $\alpha$  radiation. Powder patterns were taken at 50K, with subsequent increments of 50K up to 300K. Lattice parameters were refined using least squares. The data is shown in Figure 2. The refined cell parameters are available in Data File 1. Thermal expansion or contraction of the cryostat resulted in non-uniform shifts that could not be fully accounted for due to the limited 2- $\theta$  range of the data. These shifts particularly affected the refined **a**-lattice parameters. The cell parameters at 300K, **a**=3.2405 and **c**=25.106 are quite close to the published cell parameters, **a**=3.236 and **c**=25.15; the small differences may be due to slight differences in the stoichiometry of the melt-cooled ScAlMgO<sub>4</sub>.

Using the refined unit cell parameters for 200 and 250K, we obtain thermal expansion coefficients of  $6.2 \times 10^{-6}/^{\circ}\text{C}$  for the **a** direction and  $12.2 \times 10^{-6}/^{\circ}\text{C}$  for the **c** direction, roughly what we expected.

## 3. GaN Epitaxial Growth

Nitride growth was done in a Riber molecular beam epitaxy (MBE) system with a custom nitrogen plasma source. The plasma source is described in Reference [12] The substrates were heated to 700°C in vacuum, then exposed to the nitrogen plasma at a temperature between 600 and 650°C briefly before starting the GaN deposition. The nitrogen plasma is excited with 10-20 watts of RF power in nitrogen at 65-85 mTorr. The RHEED (reflection high energy electron diffraction) pattern was observed through the whole process. Figure 3a shows the RHEED pattern of a ScAlMgO<sub>4</sub> substrate before growth. Although somewhat blurry, it shows the streaks indicative of a surface with atomically smooth areas. Exposure to nitrogen resulted in a marked improvement in the RHEED pattern of the substrate, as seen in figure 3b. The streaks are still blurry, but they are significantly brighter. The patterns in 3a and 3b are too similar to determine whether the surface has been nitridized, or merely cleaned of contaminants. The substrate was then exposed to a Ga beam by opening the Ga furnace shutter, starting the growth. The Ga furnace temperature was set to obtain GaN growth rates between 350 and 5000 Å/hr. The RHEED pattern remains bright at the start of GaN growth, in contrast to growth on Al<sub>2</sub>O<sub>3</sub>, for which the RHEED pattern is sharply reduced in intensity at the start of growth. Figure 3c shows the pattern after growth of GaN for 50 seconds at 350Å/hr. Almost no qualitative change in the RHEED pattern is observed, instead, the pattern grows brighter and more distinct. This is

characteristic of coherent epitaxy. For higher growth rates, the streaky pattern is sustained for only a short time, while for low growth rates, the streaks become brighter and sharper to the end of the growth, as seen in Figure 3d. Figure 3e shows the RHEED pattern we observe in films of high quality after cooling to 250°C in vacuum. This 3x3 reconstruction, which we have confirmed by low energy electron diffraction (LEED) and also observe in the best of our films on sapphire, may be a result of nitrogen loss at the surface. We have not been able to find reports of the 3x3 reconstruction on GaN in the literature; the (3x3) reconstructions known for InSb ( $\bar{1} \bar{1} \bar{1}$ ) [13] and 6H-SiC (0 0 0 1) [14] surfaces could be related.

## 4. GaN Film Characterization

After the films are removed from the growth chamber they are un-soldered from the heater block. The films are smooth as seen under a Nomarski microscope, although under some growth conditions gallium droplets can form. No peeling is observed, although our first film had some surface chipping remaining from the surface preparation. The films are further characterized by x-ray diffraction, optical transmittance, and by photoluminescence.

### 4.1. X-ray Diffraction

The film crystallinity was examined using a 4-circle x-ray diffractometer using monochromated Cu K $\alpha$  radiation. In addition to the usual  $\theta$ -2 $\theta$  and  $\omega$  scans, scans on in-plane diffraction peaks of the film and substrate were used to measure the azimuthal order. This measurement is particularly relevant to large misfit epitaxial systems, as discussed in Reference [15]. The  $\theta$ -2 $\theta$  scan for a 0.22  $\mu\text{m}$  thick film on ScAlMgO $_4$  is shown in figure 4. Prominent are the substrate (0 0 3l) peaks and the GaN (0 0 2l) peaks. The diffractometer resolution was insufficient to resolve the  $\theta$ -2 $\theta$  peak widths.  $\omega$  (rocking) scans on the substrate (0 0 18) peak revealed two crystallites in the measured region with a 0.3° difference in orientation, each with  $\omega$  peak widths of 0.5°. [b] The (0 0 4) rocking curve for the film showed 0.8° wide peaks. These numbers are to be compared with the 0.37° rocking curve width measured for GaN grown concurrently on sapphire, which had an  $\omega$  peak width limited instrumentally to 0.22°. The  $\Phi$  scans of substrate and GaN azimuthal peaks indicated only the 3 peaks spaced at 120° intervals for the rhombohedral sapphire and ScAlMgO $_4$ , and the 6 expected peaks in the hexagonal GaN. Figure 5 shows the peaks in detail. The ScAlMgO $_4$  substrate again reveals two crystallites, misoriented in  $\Phi$  by 0.4degree the sapphire peak again exhibits the instrumental resolution. The azimuthal broadening of the GaN peaks is clearly apparent, and is similar for growth on sapphire and growth on ScAlMgO $_4$ . The Matthews theory of island rotations for large misfit systems [16] predicts that the  $\Phi$  width on sapphire should be a factor of 3 larger than that on ScAlMgO $_4$ , which we do not see in the data. This suggests that island nucleation is not the principle source of the azimuthal broadening in this film.

### 4.2. Optical Transmittance

One of the easiest ways to measure the GaN film thickness for the thicker films is to measure the optical transmittance. The Fabry-Perot fringes can be counted to determine the film thickness, and the position of the absorption edge can be verified. The amplitude of the fringes can be used to give a rough estimate of the substrate index of refraction. It is roughly 0.3 lower than GaN over the visible range, or about 2.

### 4.3. Photoluminescence

Figure 6 shows the comparison of the photoluminescence (PL) spectra of 0.16 $\mu\text{m}$  thick GaN films grown on ScAlMgO $_4$  and on sapphire in the same growth run. A low growth rate and a low plasma power were used for this run. A He-Cd laser was used as an excitation source; the emitted light was dispersed by a 0.5 m monochromator and detected by a CCD camera. The emission spectra of both samples look very similar for both temperatures and the intensity is roughly the same. The spectra measured at 5 K show a rather small donor bound exciton peak with a slightly red shifted [17] maximum at 3.43 eV (Fig. 6a). These spectra are dominated by the donor-acceptor pair transition at 3.26 eV and its phonon replicas. The broad band in the yellow spectral region with maximum at 2.25 eV is commonly believed to result from defects. [18] The intensity of this band is low in these samples, a sign of good crystal quality. At room temperature we observe a band edge peak at 3.37 eV although the main emission features are broad bands with their maximums around 2.0 and 1.7 eV.

The PL results show that the band edge emission from thin GaN films grown on ScAlMgO $_4$  and on sapphire is roughly comparable. Films of superior quality have been grown on sapphire by MOCVD; these films are typically an order of magnitude thicker than our MBE films. We find that, between sapphire and ScAlMgO $_4$ , the luminescence intensity is much more dependent on growth conditions than on the substrate type. Considering the crystal quality

of the substrate, which has to be improved, the luminescent properties of GaN films grown on ScAlMgO<sub>4</sub> look very promising.

## 5. Discussion

What should be clear from our results is that ScAlMgO<sub>4</sub> is a very promising substrate material for epitaxial growth. What should also be clear is that ScAlMgO<sub>4</sub> substrates are not the magic solution to all problems facing the realization of practical high performance GaN based devices. Although we can expect further improvement in film properties when truly single crystal ScAlMgO<sub>4</sub> substrates are available, we do not observe the dramatic improvements that we might naively expect from an order of magnitude reduction of misfit. This could indicate either that misfit dislocations are not the primary limitation to, for example, luminescence properties in our films, or that new types of defects are introduced by the substrate.

### 5.1. Stacking faults

Since the **c**-axis lattice constant of ScAlMgO<sub>4</sub> is 4 1/2 times that of GaN, we expect stacking faults to occur at surface steps on ScAlMgO<sub>4</sub> (which should be **c**/3 (0.75nm) high) A similar situation holds for the sapphire (0 0 0 1) surface. If this sort of stacking fault is important in GaN, then film quality should be sensitive to the surface mis-orientation. Stacking faults of this type should not occur for growth on isostructural substrates, such as ZnO. On the other hand, the insensitivity of the photoluminescence results to the choice of substrate suggests that misfit *per se* is not limiting the film quality of films on sapphire.

### 5.2. Lattice Matching to Alloys

We note that since a small fraction of InN will increase the film lattice constant, substrates with positive misfits to GaN can be perfectly lattice matched to alloys in the (In,Ga,Al)N system. Thus the +1.8% misfit for ScAlMgO<sub>4</sub> should be perfectly lattice matched to In<sub>0.16</sub>Ga<sub>0.84</sub>N and In<sub>0.30</sub>Al<sub>0.70</sub>N, while the -3% misfit to 6H-SiC cannot be eliminated in this manner.

## 6. Conclusion

The initial results reported here indicate that ScAlMgO<sub>4</sub> is a promising substrate material for GaN epitaxial growth. Although growth conditions for GaN on ScAlMgO<sub>4</sub> may have to be re-optimized to benefit from the order-of-magnitude reduction in the misfit, we anticipate that when large, high quality single crystals are available, growth on ScAlMgO<sub>4</sub> will result in epitaxial films surpassing the quality of those currently produced on sapphire. This may be a crucial step towards the realization of high-performance GaN-based opto-electronic devices.

## Acknowledgments

**The authors would like to thank A. J. Valentino for assistance in the substrate growth.**

Tac

12 3.2372 25.0349

100 3.2382 25.0381

150 3.2394 25.0486

200 3.2373 25.0636

250 3.2383 25.0789

298 3.2405 25.1061

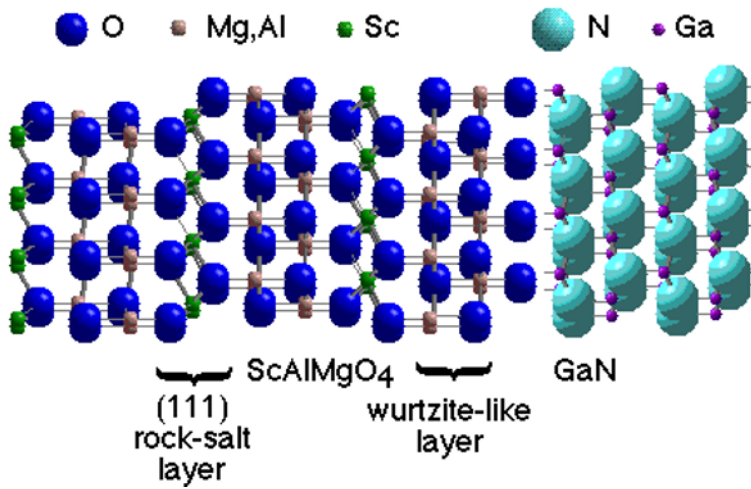
## Footnotes

[a] The appropriate convention for computing the misfit is  $(a_s - a_f)/a_f$ , where  $a_f$  is the film lattice constant,  $a_s$  is the substrate lattice constant. This convention gives the misfit relevant to the misfit dislocation density.

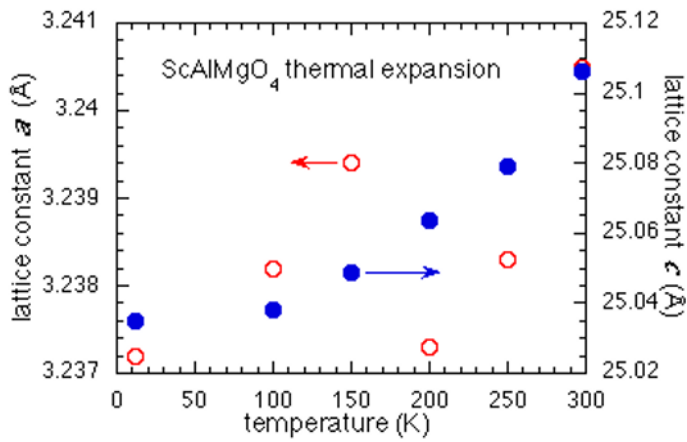
[b] Some authors in the GaN field seem to confuse the  $\omega$  rocking curve with the  $\theta$ - $2\theta$  peak width, which is usually much smaller.

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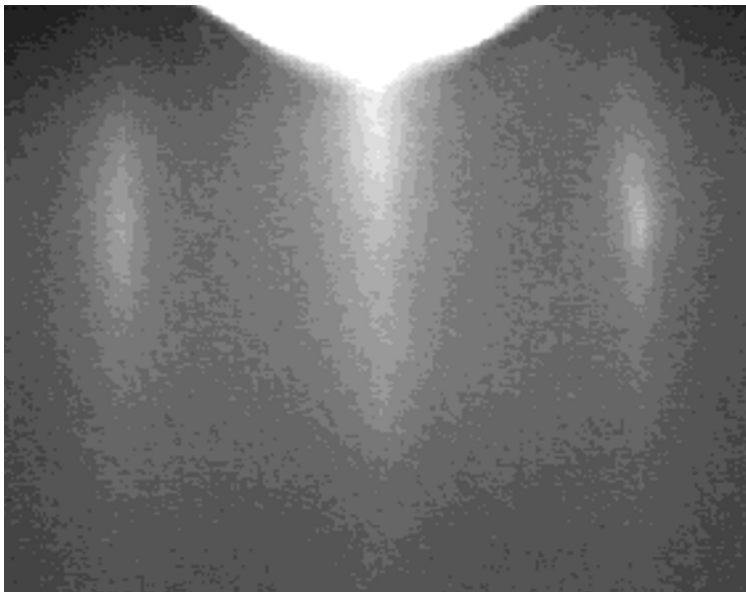
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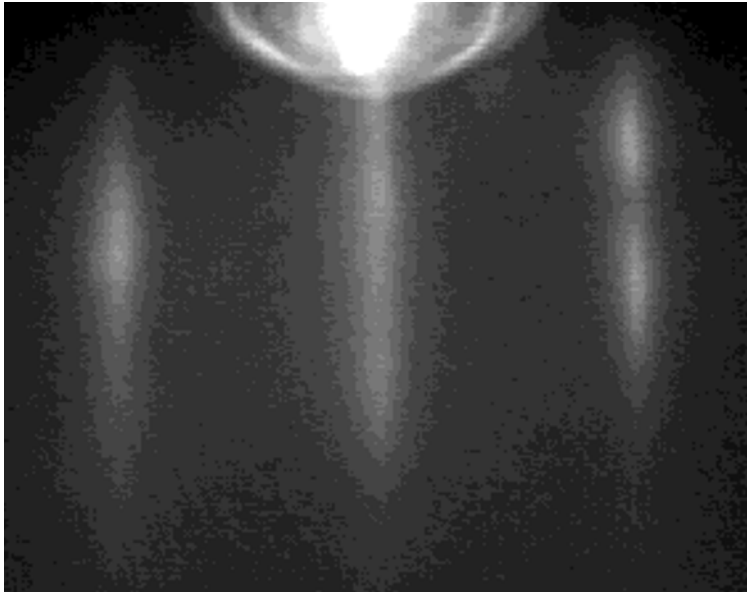
**Figure 1.** Comparison of the crystal structure of ScAlMgO<sub>4</sub> to that of GaN.



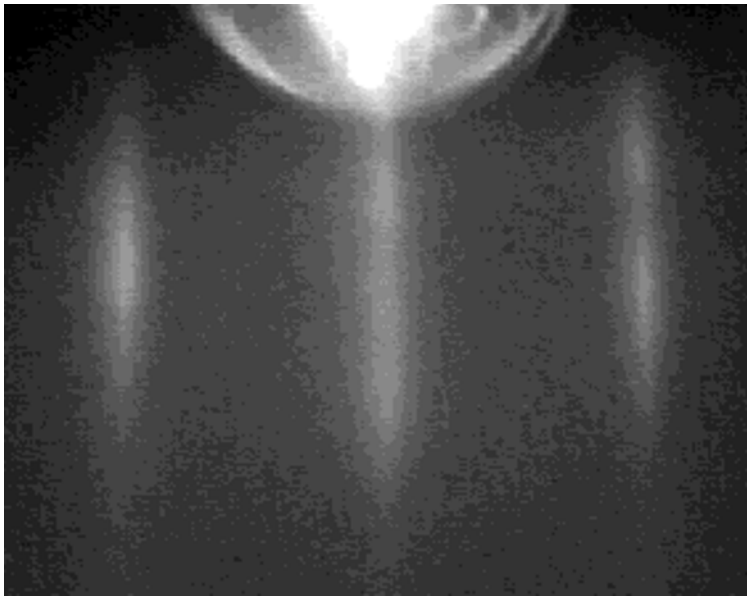
**Figure 2.** Lattice constants of ScAlMgO<sub>4</sub> measured at different temperatures.



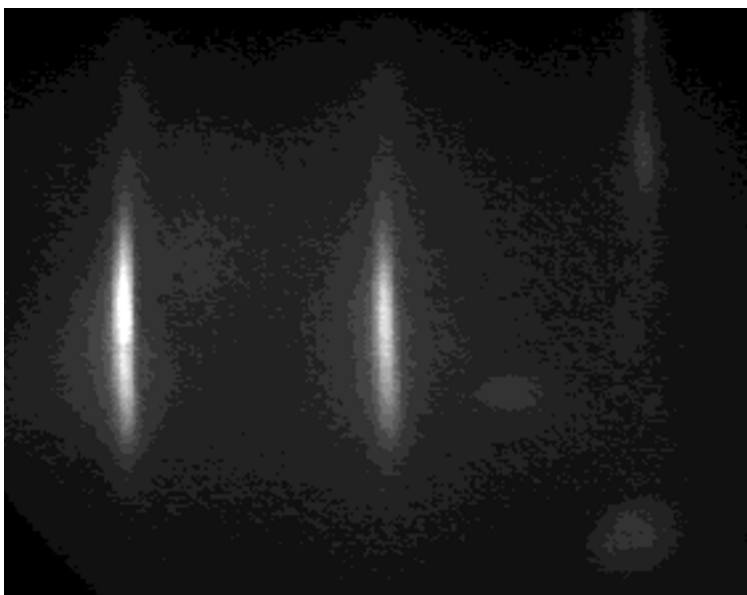
**Figure 3a.** Reflection high energy electron diffraction (RHEED) pattern of ScAlMgO<sub>4</sub> along the  $\langle 11\bar{2}0 \rangle$  azimuth. The plane of the substrate is at top. The bloom at the top is caused by the insulating surface.



**Figure 3b.** RHEED pattern of  $\text{ScAlMgO}_4$  along the same azimuth, after exposure to the nitrogen plasma.

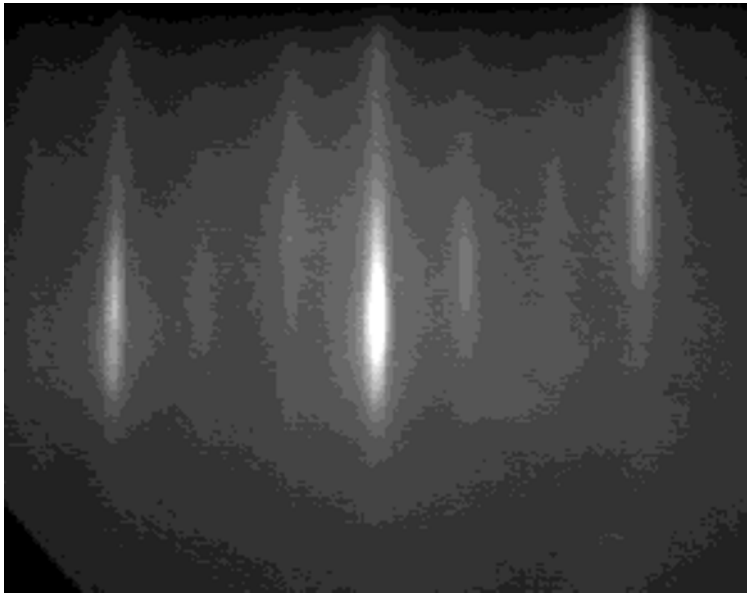


**Figure 3c.** RHEED Pattern after 50s (2 monolayers) of GaN growth. Note that little change has occurred in the pattern.

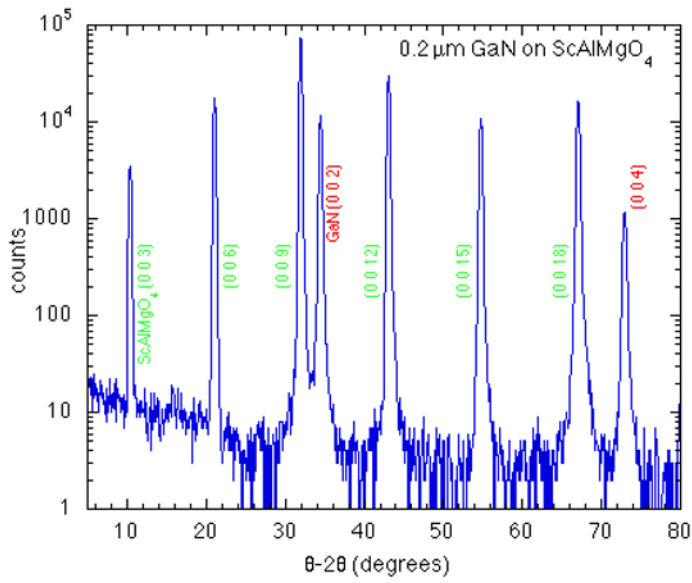


**Figure 3d.** RHEED pattern after  $.15\mu\text{m}$  of GaN growth.

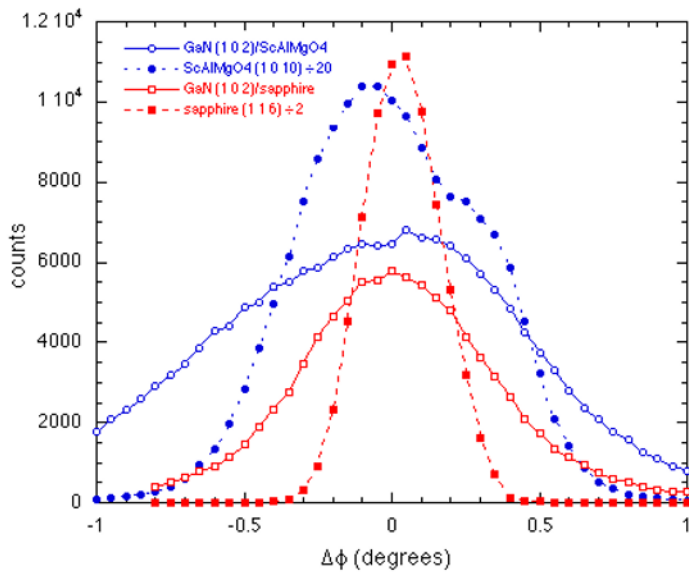




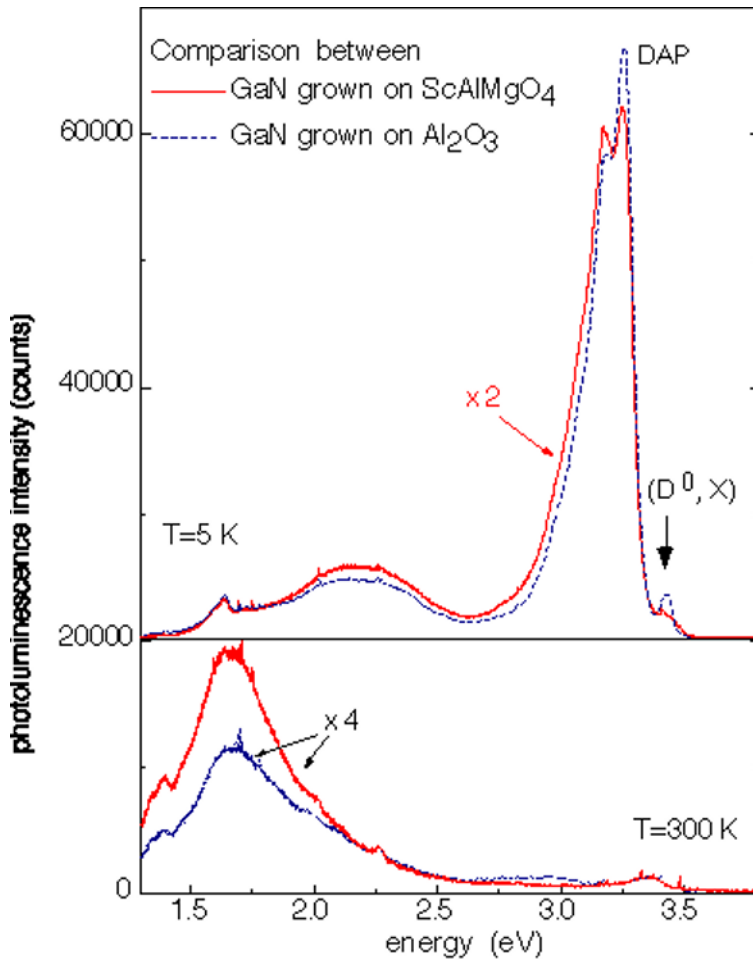
**Figure 3e.** RHEED pattern along same azimuth, showing the 3x3 superstructure observed after cooling in vacuum to 250°C.



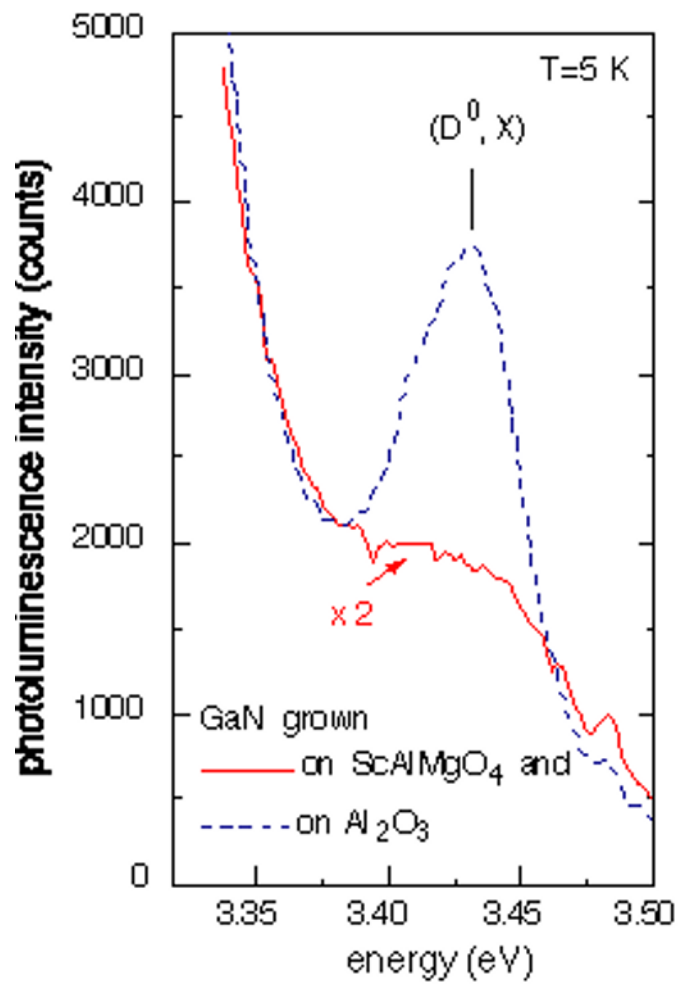
**Figure 4.**  $\theta$ -2 $\theta$  x-ray diffraction scan for a 0.22  $\mu\text{m}$  thick GaN film on ScAlMgO<sub>4</sub>.



**Figure 5.** Azimuthal scans on in-plane diffraction peaks for GaN films on sapphire and on ScAlMgO<sub>4</sub>. The peak width for the sapphire (1 1 6) peak is essentially the instrumental resolution. GaN peaks rotated 60° from the substrate peaks are used to avoid interference.



**Figure 6.** Comparison of the photoluminescence (PL) spectra, at room temperature and at 5K, of 0.16 $\mu$ m thick GaN films grown on ScAlMgO<sub>4</sub> and on sapphire in the same growth run.



**Figure 6a.** Enlarged view of the band-edge region of the photoluminescence spectra of Figure 6.

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