Marked advancement in sapphire crystal quality from improved process control

John Ciraldo of Rubicon Technology discusses how sapphire substrate producers need to continually improve quality to keep up with advances in LED technology.

Synthesized sapphire material has been widely adopted by industry for many applications due to its broad range of favorable properties. For example, as one of nature's hardest materials, sapphire has been frequently utilized for optical applications in environments where abrasion and subsequent wear have proven problematic for softer materials, e.g. glass. However, by far the largest adoption of sapphire has been for the LED market, particularly for the application of gallium nitride (GaN)-based devices.

While sapphire generally presents the best lattice match to GaN of any widely available and optically transparent substrate material, achieving a very high-quality GaN film at the GaN/sapphire interface still presents challenges. This is due in part to the lattice mismatch between the two materials, but is exacerbated by defects in the sapphire crystal that directly impact the quality of the epitaxial layers in LED devices. Defects such as surface bubbles, dislocations, and impurities are widely known within the industry to be problematic in LED applications where, for example dislocations in the substrate can be replicated in the epitaxial overgrowth. Moreover, in many LED applications sapphire is part of the structure of the final LED device, with the consequence that the optical properties of the sapphire affect the LED efficiency.

Although the quality of available sapphire material has improved over time, it has struggled to keep up with the advances in LED technology. As LED producers continue to push the limits of power and efficiency in their devices, substrate quality becomes an increasingly important consideration. As a result, substrate producers need to continue to innovate and find new ways to enhance their material.

Rubicon Technology takes a holistic approach to improving the quality of its sapphire (Raja Parvez, 'Vertical integration streamlines sapphire production', Compound Semiconductor March 2013, p50–55). For example, rather than relying on outside vendors for high-quality sapphire precursors, Rubicon has brought much of the refinement in-house, providing tighter control of purity levels. Vertical integration extends through proprietary furnace technology and crystal growth methodology to patented tools for precise crystal orientation, a high-precision polishing platform, and large-diameter custom patterning capability. By controlling every aspect of the process, Rubicon maintains greater consistency and uniformity and has earned a reputation for overall sapphire material quality.

The use of x-ray diffraction (XRD) rocking curves is often employed to evaluate the quality of single-crystal materials. This technique is highly sensitive to strain, particularly in the case of single-crystal material, which is represented by a broadening in the rocking curve peak. Common causes of strain within the crystal include dislocations, vacancies, and bubbles (i.e. macro-scale vacancies within the bulk crystal). Thus, by evaluating the full width half maximum (FWHM) value of a rocking curve, one can obtain detailed information about the quality of a crystal. With the help of Dr Albert Macrander and Dr Naresh Kujala, rocking curve data was obtained at The Advanced Photon Source at Argonne National Laboratories for multiple sapphire samples. Included in the study were standard Rubicon sapphire, as well as commercially available sapphire material from other vendors. The results of this study can be found in Figure 1. As can be seen, material from Rubicon shows a greater overall intensity with a significantly narrower peak, both of which are indicators of superior crystal quality. Moreover, the peaks from Rubicon's material show a higher symmetry, indicative

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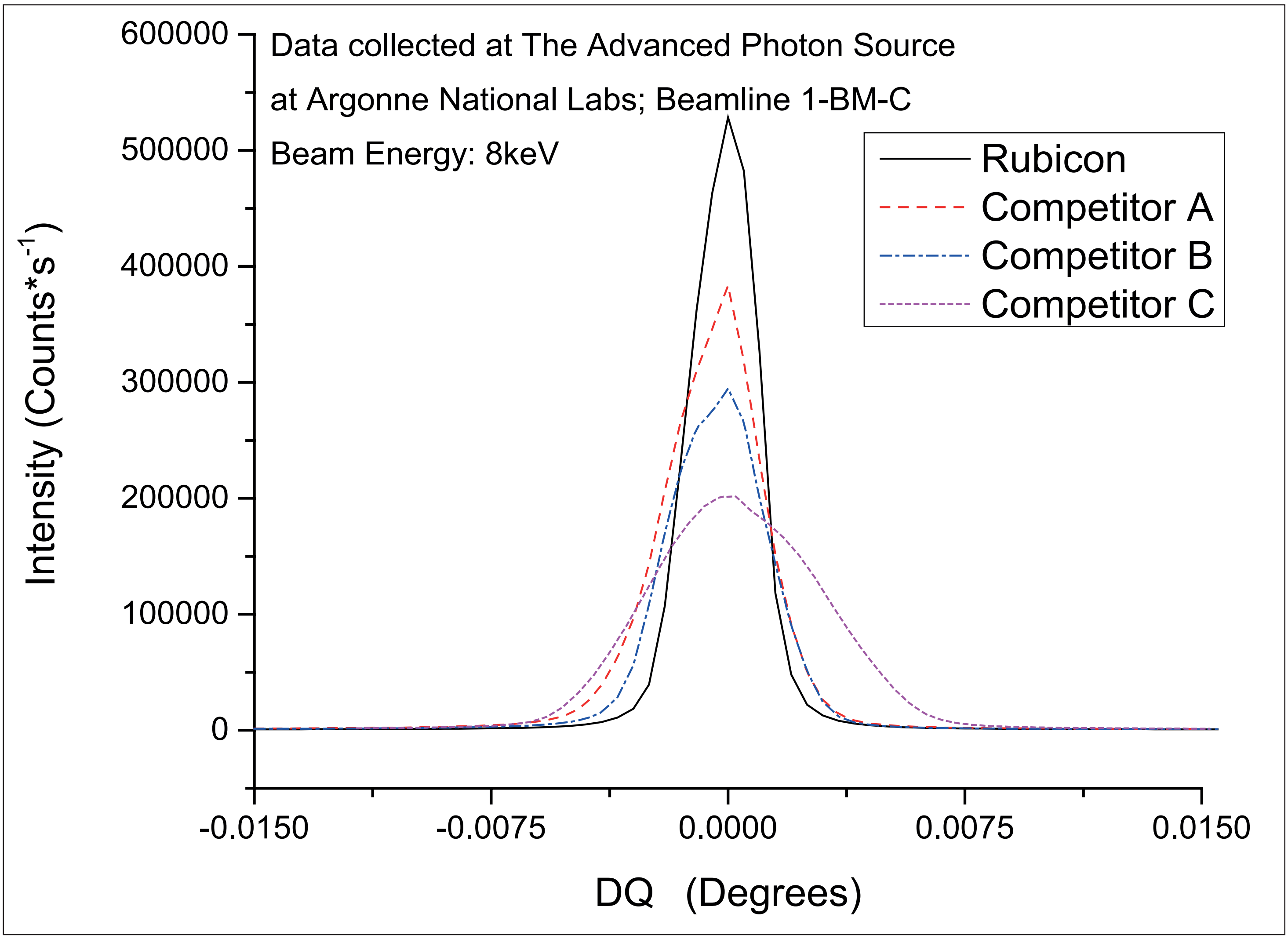

Figure 1: X-ray rocking curve of c-plane sapphire material. The Bragg reflection of the sapphire was for the (0006) reflection, which occurred at a Bragg angle of 21º. The synchrotron x-ray beam had been preconditioned with a Si(111) x Si(111) double crystal monochromator. Intensity recorded via pin-diode.

of a very low stress gradient within the material. FWHM values for each sample can be seen in Table 1.

In addition to collecting rocking curve data, the unique capabilities of Argonne's Advanced Photon Source also allows for x-ray topography data to be collected on the single-crystal material. X-ray topography is used for the imaging of lattice defects, which can be seen as streaks in the topography images. Thus, x-ray topography produces data that is comparable to traditional etch pit density (EPD) techniques via wet etch. However, unlike EPD, x-ray topography has the advantages of being non-destructive, and sensitive to sub-surface defects as well as purely surface defects.

Again with the help of Dr Albert Macrander and Dr Naresh Kujala, x-ray topography images were taken on each of the three competitor materials used for rocking curve data, as well as the same standard Rubicon wafer used for the collection of rocking curve data. Each topographic image represents an area of approximately 5.5mm wide x 3.5mm high. Streaks observed in topographic images represent lattice defects. Dark regions represent high densities of lattice defects (i.e. 'tangles'). Dark or light spots within the images are artifacts from the system setup and are not related to the samples themselves. The sample from competitor C shows a very high density of lattice defects, including a large band of extremely high defect density. Competitor sample A is comparatively much better, but still demonstrates a significant amount of defects. Competitor sample B is similar in defect density to Competitor sample A. The final image is of the Rubicon material. While, as is to be expected, some lattice defects are present in the Rubicon sample,

Table 1: FWHM values for Gaussian fits of x-ray rocking curve data from Figure 1.

Sample	FWHM (arc-seconds)
Rubicon	8.712
Competitor A	12.881
Competitor B	14.110
Competitor C	22.710

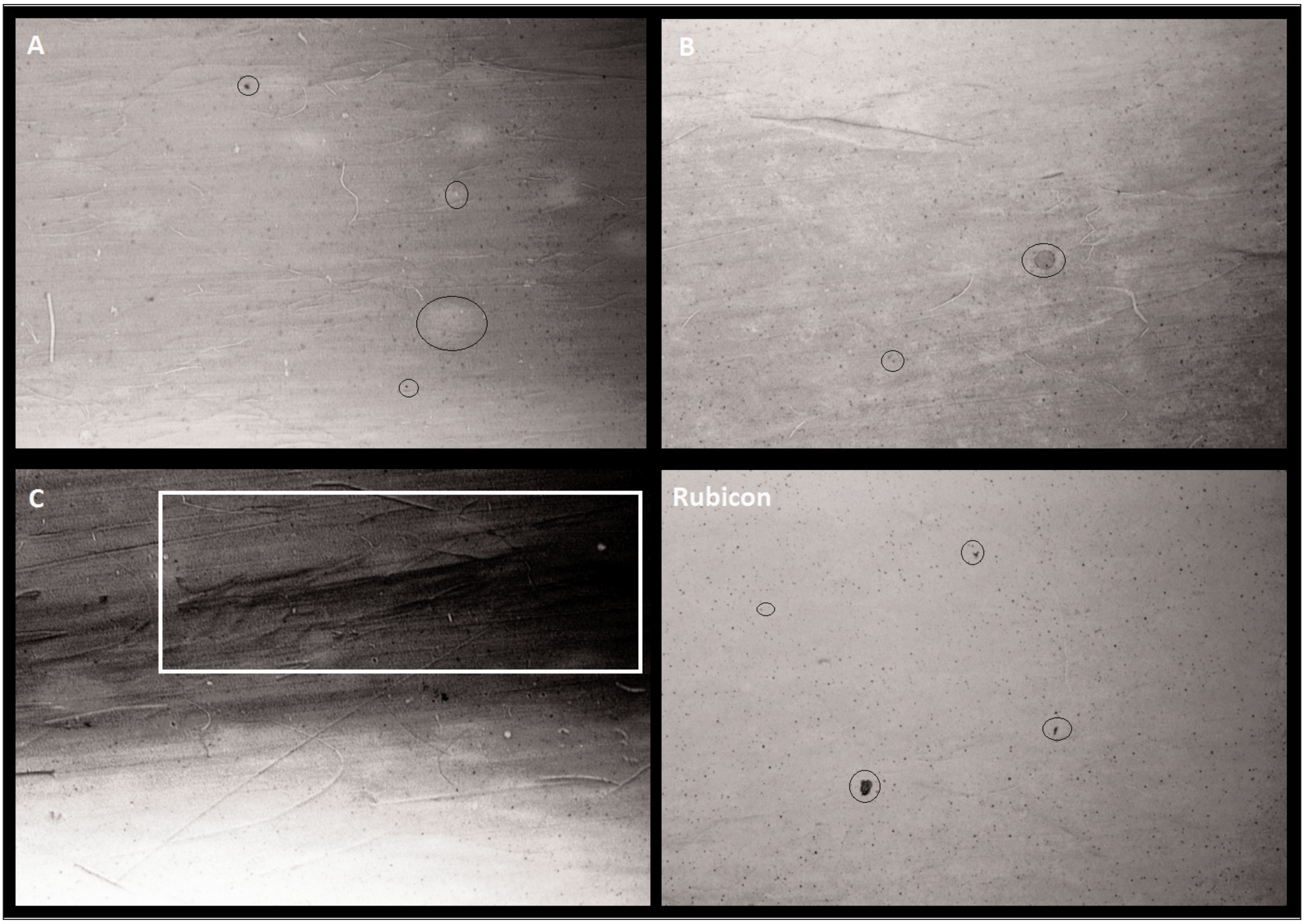

Figure 2: X-ray topography images of c-plane sapphire. Light and dark spots (such as those circled) are artifacts from imaging and developing and are unrelated to crystal structure. Boxed-in region is an example of a tangle, or large band of defects.

they are quite sparse, and the defect density is clearly much lower than that of any of the competitor material (as indicated by the lack of obvious streaks). It is noteworthy that the defect density of each material shows a strong correlation with the FWHM values from Table 1. While this is not surprising, this correlation helps to validate each individual measurement. Lastly, it should be noted that, while only one Rubicon sapphire sample was presented for this study, several Rubicon samples, each randomly selected from stock, were studied with similar results.

Single-crystal sapphire has been available for industrial applications for some time. While no other material is as useful as sapphire for GaN-based LEDs (due to superior lattice and optical properties), available sapphire has historically been associated with high defect densities and poor crystalline quality compared with some other widely available semiconductor materials. As the quality of the semiconductor substrates has a direct impact on the quality of epitaxial films deposited on the substrate, improvements in crystallinity are paramount to improvements in epitaxial devices, such as LEDs. This relationship is particularly important for more sensitive applications such as high-brightness LEDs, where a push toward greater efficiency and higher output power are key for competitive advantage and essential for continued cost reduction and overall industry growth.

By exercising more control over the production of our sapphire material through a vertically integrated approach, Rubicon Technology has demonstrated vast improvements in the overall quality of sapphire crystals that make them much more suited to advanced applications, including high-efficiency LEDs. As demonstrated through various techniques of x-ray characterization, Rubicon has confirmed the ability to produce sapphire crystal of a significantly higher quality than has previously been possible by any large-scale producer of sapphire crystals. ■

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