Preparation of GaN / Sapphire for TEM using Tripod Polishing

1.0: Purpose

To develop and help refine a TEM specimen preparation protocol for the successful production of XTEM specimens of a GaN thick film on Sapphire. The specimen preparation process should be relatively fast, produce high quality specimens with large thin areas and be repeatable. Preparation using a Model 590 Tripod Polisher® will be done with a final ion thinning step to fine polish the specimen for TEM examination. Upon successful completion of specimens TEM micrographs will be presented to show the quality of the specimen produced.

2.0: Experiments

Wafer pieces of Sapphire substrate containing a thick film of GaN grown on the top surface were obtained for TEM specimen preparation. The specimens obtained were grown by MOCVD into a thick film of GaN. Cross sections were to be made using the Model 590 Tripod Polisher® with a series of progressively finer diamond abrasive films.

2.1: Initial Specimen Preparation

Specimens were first cut into small 3 mm^2 pieces for mounting onto the SEM stub. Cutting was completed using a Model 850 Wire Saw using a 14 micron boron carbide abrasive slurry. The Model 850 Wire Saw is a good choice for cutting due to the low mechanical damage induced during cutting and reduced risk of film delamination. Following specimen cutting the specimens were mounted onto an SEM stub using a low metling point wax such as MWH 135. The wax is an acetone soluble glycol pthalate wax which becomes fluid at around 130°C. Multiple specimens were mounted onto the stub to increase specimen throughput. The area of interest for TEM was non-specific so the multiple specimen mounting presented no problems in locating the specific site of interest. Below is an illustration of the specimen mounting arrangement.

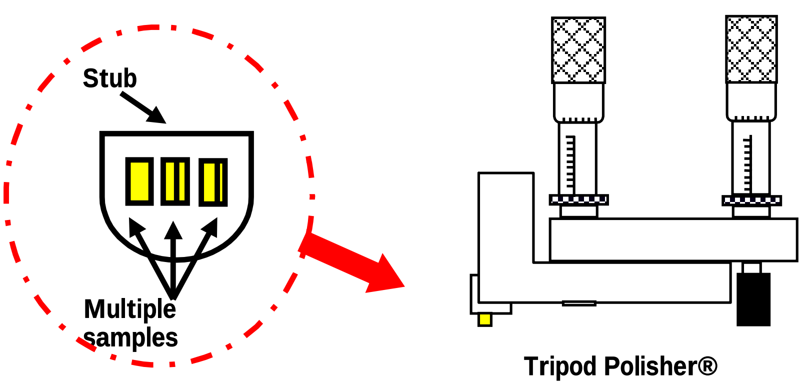

Figure 1: Schematic illustration of the specimen mounting configuration for specimen preparation using the Model 590 Tripod Polisher ®. Multiple specimens are mounted onto the SEM stub using a low melting point wax. The stub is then placed into the Tripod for polishing.

2.2: Polishing the Specimen

After the speicmens were mounted and placed into the Tripod Polisher® they were polished using a Model 910 Lapping and Polishing machine. The Model 910 provides the high stability, high torque and low speeds required for preparing a TEM specimen using mechanical thinning methods such as these. Polishing requires two stages: first side and second side polishing. Both stages of polishing are completed using a hard glass lapping plate with plain backed diamond lapping films. Diamond films of 30, 15, 6, 3, 1, and 0.5 µm are used for rough a fine polishing of the specimen to the desired area. In this case only the surface finish was monitored to ensure that a high quality polish was obtained. Final polishing was completed using a Multitex polishing cloth combined with 0.05 µm colloidal silica solution to obtain a smooth, damage free surface.

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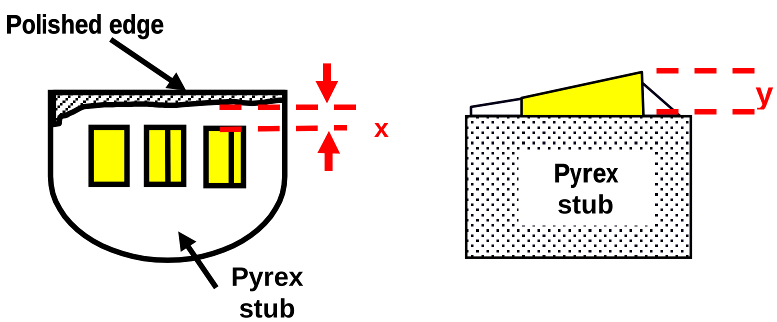
Following the first side polish, the specimens are mounted onto a Pyrex stub using the same mounting wax and then polished at a slight angle, known as the “wedge angle”. The magnitude of this angle dictates the amount of thin area which will be available from the front edge to the back edge of the specimen. Selecting a proper angle depends both upon how much thin area is desired and the difficulty of the specimen. In this case a wedge angle of approximately 0.5° was used. Using the same progression of diamond films as the first side, the second side of the specimen was prepared to a thickness of less than 10 µm.

2.3: Calculating Specimen Thickness

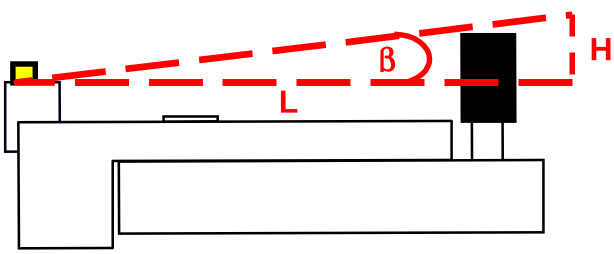
To determine the final thickness of the specimen a relatively easy technique can be employed. During the final stages of polishing (usually starting around 3 µm) the Pyrex stub begins to polish. By measuring the distance the specimen is from this polished edge and by calculating the wedge angle, the specimen thickness can be determined in situ during polishing. This helps to determine when to switch abrasive film grits and to help determine the final thickness prior to ion thinning. Below are two schematics and equations that help illustrate this effective technique.



A

Specimen Thickness (Y) = Tan (β) × (X)

β = Wedge Angle
X = distance from polished edge of stub
Y = specimen thickness



B

$$\textbf{Wedge Angle } (\beta) = \tan(\beta) = \frac{H}{L}$$

Figure 2: Schematic illustrations of the techniques used for measuring specimen thickness. The edge of the Pyrex stub is polished in the final stages of preparation. Measuring the distance from the polished edge and using a simple geometric equation the specimen thickness can be determined.

3.0: Results

Using the Tripod Polisher® for preparation followed by a brief ion thinning step, good TEM specimens can be obtained using this technique. Below is a TEM image of a cross section prepared by this method, illustrating a GaN film on a Sapphire substrate. Both the Sapphire and GaN film are electron transparent over a wide range. The image was taken at low magnification. The total specimen preparation time was approximately 3 hours, with ion thinning comprising over half of that time. Total preparation time of the Tripod Polisher® was about 1.5 hours.

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Figure 3: XTEM image of a GaN film on Sapphire prepared using the Tripod Polisher® followed by ion thinning at low angle. As can be seen the image shows electron transparency through a wide range and exhibits thin areas both in the GaN film and Sapphire substrate.

4.0: Conclusion

As can be seen the Tripod Polisher® can be a valuable tool in preparing TEM specimens of all types, including hard materials such as Sapphire and GaN. The ability to monitor the specimen thickness directly, to adapt the tool to prepare virtually any material type, and the high specimen throughput make the Model 590 Tripod Polisher® a unique tool for TEM specimen preparation technique. This same technique can also be applied to similar materials systems such as SrTiO3, SiC, and other hard ceramic type materials.

5.0: References

J. Benedict, R. Anderson, S. Klepeis, M. Chaker, A Procedure for Cross Sectioning Specific Semiconductor Devices for both SEM and TEM Analysis, ed. R. Anderson, Mat. Res. Soc. Proc. vol 199, Pittsburgh, PA USA. Pp. 189, 1990.

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