Model IV3

Ion Milling Parameters for Various Materials Systems

Applications Laboratory
Report 35

Sample
Preparation

1.0: Purpose

A wide range of materials systems exist in industry today and present special problems to cross sectional TEM specimen preparation. Many materials currently employed in thin film and semiconductor research consist of widely varying atomic number materials with varying hardness. From a specimen preparation standpoint these materials systems create difficult thinning phenomena such as preferential thinning leading to unequal thickness in the TEM specimen.

To overcome these problems low angle ion milling with relatively high powered ion guns has been successfully employed, allowing a wide range of different materials to be ion milled to electron transparency with great success. This report will outline various ion milling parameters such as ion beam angle of incidence, gun current, time, thickness, etc. to handle certain materials systems. All procedures listed below assume a specimen thickness of < 50 microns.

2.0: Experiment and Procedures

Following are basic guidelines for the preparation of a variety of materials using the Model IV3 Ion Mill. The processed used for these samples is a good starting point for preparation of these materials systems.

Silicon Based Materials

The preparation of Si based cross sections can be done a number of ways. In this case the specimen is mounted to the single sided specimen holder.

First Side Milling

Gun Voltage: 10 kV
Gun Current: 3.5 mA
Beam Angle: 3-4 degrees

Using two guns, the specimen is rotated for approximately 1 hour. Following the two gun thinning milling is then switched to one ion gun thinning with the specimen oscillating. The beam should be perpendicular to the interface of interest. The movement at the interface should be around 0.1-0.15 mm.

Second Side Milling

Gun Voltage: 10 kV
Gun Current: 3.5 mA
Beam Angle: 3-4 degrees

Using two guns, the specimen is rotated for approximately 1 hour until the Si is red in color. Following two gun thinning milling is switched to one ion gun with the specimen oscillating. The beam should be perpendicular to the interface of interest. The thinning will be carried out for about 0.5-1 hour or until perforation at the interface is seen.

Sapphire Based Materials

Sapphire preparation is similar to that of Si materials except a slightly larger ion beam incidence angle is used coupled with a slight increase in gun current.

First Side Milling

Gun Voltage: 10 kV
Gun Current: 4 mA
Beam Angle: 5-6 degrees

Using two guns, the specimen is rotated for approximately 2-2.5 hours. Following this process the milling is switched to one ion gun milling with the specimen oscillated perpendicular to the ion beam. This stage of thinning is done for about 1-1.5 hours, or until the interface region movement is seen to be about 0.1-0.15 mm.

Second Side Milling

Gun Voltage: 10 kV
Gun Current: 3.5 mA
Beam Angle: 3-4 degrees

Using two guns, the specimen is rotated for approximately 2 hours. Following this process the milling is switched to one ion gun with the specimen oscillated perpendicular to the ion beam. This stage of thinning is carried out for about 1.5-2.5 hours or perforation.

Approximately 0.5 hours prior to perforation you can see the interference fringes in the sapphire. These are usually visible when using the reflected light mode of the microscope as opposed to transmitted light. The fringes are the best indication for thinness as the optical terminator will not work for transparent materials.

Glass Materials

Glass materials mill in a similar manner as Si based materials. The process is basically the same for glass as Si using the following gun parameters.

Gun Voltage: 10 kV
Gun Current: 3.5 mA
Beam Angle: 3-4 degrees

If the color of the ion beam begins to change to an orange color during the thinning of glass, this is an indication that the glass contains Na. In this case use a 7 kV ion beam with 2 mA of gun current for about 4 hours. Once you have reached perforation mount the sample in the two sided specimen holder and clean the surfaces of the specimen using a 2 kV ion beam, 2 mA of gun current at 8-10 degrees for 2 minutes. Although a good sample is not guaranteed, this method sometimes works for cleaning off the mobile Na portion of the specimen.

3.0: Conclusion

Although automatic optical termination is not really an option for most of these materials, it is possible to monitor the thickness of the specimen using a fairly high powered microscope. Simply focus on the top and bottom of the specimen and measure the difference in height on a calibrated microscope. This will enable determination of specimen thickness during the course of ion milling. The only drawback is the necessity to remove the specimen holder each time a measurement is done. Another method is to glue a piece of Si to the surface of the specimen at the beginning of preparation. At the final stages of thinning with the ion mill the Si will exhibit characteristic color changes during the thinning process which are a function of specimen thickness. This can be used as a guide as well and can be monitored in situ.