Shock Testing of Silicon Nitride and Beryllium Thin Film Membranes


ard Vanfleet*, Steven Liddiard**, Sterling Cornaby**, Robert Davis*

*Brigham Young University, Provo, UT, USA, robert_davis@physics.byu.edu
**Moxtek Inc., Orem, UT, USA, scornaby@moxtek.com

ABSTRACT

Silicon nitride and beryllium thin film membranes are often used in X-ray windows for elemental analysis systems. A determination of the shock that these membranes can sustain without breaking is valuable for these applications. In this study thin film membranes of both materials were subjected to shocks produced by a pendulum shock test apparatus. The silicon nitride windows were found to catastrophically fail at an acceleration of 8,145.62 g or less while the beryllium windows were subjected to accelerations as high as 30,516.41 g without failing.

Keywords: membrane, shock, silicon nitride, beryllium, x-ray windows

1 INTRODUCTION

Free standing thin film membranes are an ever increasing part of technology. As devices containing thin membranes become more common in products available to the general public, there is a greater demand for more reliable, robust and durable thin film materials. One application where thin film membranes are used is in X-ray windows. X-ray windows require thin films consisting of low Z elements which are able to transmit soft X-rays, and withstand the stress required to maintain a vacuum. X-ray windows are used in X-ray analysis systems in electron microscopes. They are also used in hand held devices for many applications including mining and scrap metal analysis as well as heavy metal detection in consumer product screening.

Shock test studies have been performed on electronics [1] and membranes related to biological systems [2], but literature showing the robustness of free standing membranes is not readily found. In this study the durability of free standing thin film membranes subjected to shocks created using a bar contact pendulum shock apparatus was investigated.

Two common materials used for X-ray windows are low stress silicon nitride and beryllium. The latter being the more commonly used material in the industry. In this study both of these membranes were subjected to a series of shocks and their ability to withstand those shocks was investigated. In order to have similar X-ray transmission properties, silicon nitride membranes must be substantially thinner than beryllium. In this study we compare the robustness of 100 nm and 200 nm thick silicon nitride membranes as well as 8 μm thick beryllium membranes. Because the thin silicon nitride membrane is only a few hundred nm thick, they were fabricated on a silicon support structure. Theoretical X-ray transmission curves for all three windows are provided in figure 1.

![Figure 1: Theoretical X-ray transmission curves for 8 μm free standing beryllium membranes and 100 nm and 200 nm silicon nitride membranes with silicon support structure.](image)

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![Figure 2: Silicon nitride membrane (gold color) on silicon support structure (purple area).](image)

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2 MEMBRANE FABRICATION

2.1 Support Structure Silicon Nitride Windows

Silicon nitride windows on a silicon support structures were fabricated using three inch diameter double polished <110> silicon wafers. Low stress silicon nitride was deposited in a Canary LPCVD reactor at a pressure 345 mTorr and a temperature of 825°C. The flow rates for NH₃ and SiCl₂H₂ were 10 sccm and 60 sccm respectively. 100 nm thin films were deposited with a deposition time of 16 min and 200 nm thin films were deposited with a deposition time of 30 min.

The silicon support structure was patterned using optical lithography. Using optical lithography, photoresist (HPR 504) was spin cast on the nitride coated wafers and soft baked for 30 min at 150°C. The wafers were then exposed to UV light using a Perkin Elmer aligner. The exposed wafers were developed in diluted KOH (0.3% in H₂O) for 1 min, and hard baked for 30 min at 150°C.

The patterned photoresist was used as an etch mask as the exposed silicon nitride was etched away using an Anelva RIE DEM-451 etcher. The reactive gases in the etching process were O₂ and CF₄ with flow rates of 3.1 sccm and 25 sccm respectively. The pressure was set to 100 mTorr and the power was set at 100 W. The exposed silicon nitride was completely etched away, leaving behind silicon. The photoresist was the removed by soaking the wafers in Nanostrip for four hours. The wafers were then placed in a KOH solution (33% in H₂O at 56.6°C) for 17 hours to anisotropically etch the silicon. The silicon nitride acts as both the etch mask and the etch stop, leaving behind only a silicon nitride membrane. The wafers were then rinsed in water and allowed to air dry. An image of the silicon nitride thin film membrane and its support structure can be found in figure 2.

2.2 Beryllium Windows

Eight micron thick beryllium windows were obtained from Moxtek Inc.

Both the beryllium and silicon nitride windows were epoxied on to titanium mounts in order to simulate device conditions and provide an interface for the shock test apparatus.

3 SHOCK TEST APPARATUS

In order to subject the thin film membranes to controlled repeatable shocks, a pendulum shock test apparatus was constructed as shown in figure 3. A pendulum arm is suspended at a controlled angle, which can be varied from 10 to 90 degrees using a stainless steel pin. By pulling the pin the pendulum arm is released and swings down freely until it strikes a stainless steel crossbar, which results in a shock to the thin film. The magnitude of the shock can be increased or decreased by increasing or decreasing the angle from which the pendulum arm is released.

An accelerometer and the thin film membranes were mounted to the end of pendulum arm just below the contact point between the pendulum arm and the cross bar as shown in figures 4 and 5. A line to a vacuum pump was connected to one side of the thin film membrane and evacuated to simulate practical device conditions and provide a membrane failure detection mechanism. The titanium mount was tightened against a rubber oring by screwing on the mount cover.

Figure 3: Shock test apparatus design. The pendulum arm can be released from varying angles and strike a stainless steel crossbar causing a shock on the thin film membranes.

Figure 4: Front view of accelerometer and window holder. The accelerometer can be attached in the small hole to the left of the window. The membrane and mount are attached to the holder by screwing on the mount cover.
4 EXPERIMENT

The accelerometer and the mounted thin film membrane were attached to the shock apparatus and the pressure was pulled down to 30 mTorr or less. The pendulum arm was then released and allowed to strike the stainless steel cross bar. The acceleration of the pendulum arm was recorded using the accelerometer before, during, and after the shock. After each release, the maximum reported shock was recorded, and the vacuum pressure and the thin film membrane were examined to verify that the film withstood the shock. If the pressure remained at 30 mTorr or less and there were no visual signs of film destruction, the process was repeated at an increased angle.

If the thin film membrane did not show signs of destruction or substantial hermetic failure after the maximum angle release was performed, the pendulum arm was raised to an angle greater than 90 degrees and released manually. If the membrane still showed no signs of destruction when released from an angle near 180 degrees, the pendulum was released from an angle greater than 90 degrees and additional acceleration was manually applied to the pendulum arm to increase the shock.

5 RESULTS

All of the thin film membranes had no noticeable change in pressure until the membrane was visually destroyed. Figure 6 provides a graph of the maximum shock that each membrane type was subjected to for every release. The shock values include those that resulted in catastrophic failure and those where no destruction was reported.

It was found that the 100 nm silicon nitride membranes were able to withstand larger shocks than the 200 nm membranes made of the same material and structure. The catastrophic shock acceleration range measured for 100 nm thick silicon nitride membranes with this geometry of silicon support structure was found to be 5062.44 to 8145.62 g. The catastrophic acceleration shock range measured for 200 nm film membranes was 3301.34 to 4912.6 g.

Beryllium membranes were found to be able to withstand shocks much greater than silicon nitride geometry. There were no beryllium membranes which met catastrophic failure due to shocks. Beryllium membranes were able to withstand accelerations of up to 30,516.41 g without failing.

6 CONCLUSION

Beryllium membranes were found to be more durable than silicon nitride membranes on this support structure geometry. The shock required demonstrates catastrophic failure of beryllium was not found in this study. Given the superiority of beryllium to withstand shocks compared to silicon nitride, it appears to be the preferred material for application that may require higher durability such as hand held devices. Both silicon nitride and beryllium appear to withstand the normal shocks experienced in microscopy applications.

![Figure 5: Cross section accelerometer of window holder. The connected tube can be connected to a roughing pump to pull vacuum. The membranes provide a hermetic seal.](image)

![Figure 6: Maximum acceleration shock per lease. The data above include both the shocks which lead to catastrophic failure shocks were not detectable destruction was found.](image)

REFERENCES
