LLNL-TR-699859

LAWRENCE LIVERMORE NATIONAL LABORATORY

Study of the integrity of pressurized LEH window assemblies at cryogenic temperatures for NIF targets

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August 5, 2016

This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

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The National Ignition Facility (NIF) is a directorate of LLNL, a DOE Lab, and is home to the world's largest laser. This laser shoots its 192 beams at a target about the size of a pencil eraser. Within the target are two main chambers and depending on the type of shot, those chambers need to be pressurized to a certain point at a very low temperature (18 Kelvin). The component used for keeping the hohlraum at its designated pressure is a Laser Entrance Hole (LEH) window, made from a thin (0.5um) polyimide film and an aluminum washer attached with a miniscule amount of polymeric adhesive. One issue that has been known to happen is the chambers will leak, at very low rates (5.0E-7 mBar-liter/s and under). At higher pressures significantly larger leak rates have been observed.

Due to the increase of leaky targets that NIF has seen since October 2015, a series of experiments to test the ability of a new type of window (B type) to withstand pressures up to 1100 torr at 9K have been conducted. These windows had double the typical amount of glue holding the 0.5 micron film to the washer. Whilst they had some interesting features on the surface they have performed markedly better than the previous "A" type windows. Each experiment consisted of loading the sample in a cryostat, performing a room temperature pressure test to 75 torr to ensure that it was leak tight, lowering the temperature to 9K and then bringing the sample to 1100 torr (approx.) while monitoring the leak rate. Afterwards a second test to 75 torr was performed at room temperature. Overall the "B" type windows performed very well staying leak tight or only showing signs of virtual leaks. A virtual leak is often an air pocket or bubble that forms while the sample is being cooled or originally made and when the sample gets cold enough the pocket bursts/collapses and lets out a steady stream of helium or another substance that the helium leak detector is able to detect. These leaks are considered virtual because they decrease over time. We had two hypotheses on how these virtual leaks were happening. First perhaps they weren't so much virtual leaks as permeation: i.e. the sample wasn't getting cold enough. At any temperature higher than 150 K these windows have shown permeation. Second, there were a number of blisters, bubbles and other interesting textural anomalies on the surface of the washer, perhaps these were bursting or releasing air while the pressure was being increased. It was determined that the reason behind these peculiar virtual leaks was the loosening of the cold head to the sample after several uses and the corresponding misalignment leading to permeation.

The LEH window is the last piece of the construction of a NIF target. There are two windows on each, one on each end. If an issue should arise with a LEH window then there is not much to be done about conserving the rest of the components of the target, which is a expensive loss each time it happens. The windows are made from an aluminum washer, about ~6mm in diameter, which is bead blasted to roughen up the surface and then coated with a thin layer of glue. On top of the glue a 0.5 micron polyimide film is stretched and then coated with a thin layer of aluminum. This creates a window of ~4mm diameter free-standing film, which can then be attached to a target. LLNL does not do this construction on site it is contracted out to a company called Luxel.

In 2013-14 there was a sharp increase in the number of LEH windows that were leaking at relatively low pressures for reasons unknown. The pattern of the leaks was such that a leak rate would be steady for approximately 300-400 torr and then a jump would occur (See Figure 1). Eventually it was determined that the cause of the problem was the angle by which the UV glue was being cured at. The UV cure glue was being used to attach the LEH window to the thermal mechanical package (TMP) can. Once the angle was corrected the percent yield of sealed LEH windows returned to 100%. However late in 2015, leaks behaving in the same nature began to show up again, and it was unclear why.

There are three proposed mechanisms by which the LEH windows are leaking. The first is that there is a small pinhole somewhere in the freestanding film. This is the most unlikely because before any film is shipped from Luxel, it must pass a 50-75 torr room temperature pressure test. The second is a tear in the film at the edge of the washer. (See figure 2) This type of damage suggests that the film is under additional stress at this edge portion and/or the edge of the washer itself is what is doing the damage. Lastly, it has been hypothesized that there are small channels under the window that do not get completely filled by the glue and if they connect to the edge of the freestanding portion of the film then the pressure can escape through them. These channels were the mechanism being most directly tested over the course of my experiments.

All leaks are not the same, they follow the same general pattern as is illustrated in Figure 1, but there are different ways that these leaks have been observed. The windows are tested 3 times each, once at room temperature to 75 torr, then at cryogenic temperatures (9-18K) to failure or 1100 torr, and then once more at room temperature to 75 torr. There are four different outcomes that have been observed when performing these three tests. The first is there is a leak at the first room temperature, the leak remains the same or similar at cold temperatures, and is still bad when brought to room temperature again. In short the first type is a failure that has fully compromised the film. The second type of leak is good at both room temperature leaks but doesn't hold up when at very cold temperatures. The third and most common type of leak is good at the first room temperature, fails at cryogenic temperatures and then fails again when at room temperature the second time. The fourth type of leak is the least common, the film starts bad, gets worse when at cold temperatures and stays worse when brought back up to room temperature (Figure 3).

Because leaking was becoming such a prevalent occurrence with the LEH windows, a new type of window was made. The first type of window ("A" Type) was constructed as described earlier with a single layer of epoxy coating the washer. The new type of window ("B" Type) had roughly double the amount of glue between the washer and film. Additionally, there are some significant textural differences between the "A" Type and "B" Type windows. Most notably the “B” Type windows have many nodules ad air bubbles where the “A” Types do not (see Figures 4 and 5 for comparison).

Initially the project was meant to consist of determining the mechanism that the “A” Type windows were leaking from, so nine samples were made using a variety of batches of old(er) LEH windows. Additionally, when the new “B” Type windows were received from Luxel, six more samples were made using those windows. Each sample, whether “B” or “A” was prepared for the experiments using the same procedure. The objective when preparing the samples is to ensure it is leak tight between window and base and not to disturb the film-washer bond. Stycast 2850 (type of glue) was used to adhere the aluminum washer to a manufactured 8-VCR washer, which is thicker so that it can be reused. The 2850 behaves the same as aluminum when cooled (has similar CTE) and forms a leak tight seal every time. Then Stycast 1266 (different type of glue) is used for its lower viscosity (after the curing of the 2850) to wick under the washer while not getting on the edge of the LEH window and compromising the experiment.

Each sample was tested to 75 torr at room temperature, then brought down to 9 Kelvin. At 9K the sample was then pressurized to 1100 torr, or until a significant leak occurred. For the first three samples it was the procedure to stop the test at first sign of leak, but later that was amended to going to 1100 torr unless the leak was upwards of 1.0E-3 mBar-liter/s. In the first round of testing we did a combination of both “A” and “B” type windows. Five “A” and six “B” Type windows were tested to maximum pressure and the leak rate at that point was measured. The mixed results we got from the “A” Type was somewhat expected given that those were the washers that were causing the leaks on the actual targets, however we saw some very surprising trickling leaks coming from the “B” Type windows. Leaks that Luxel had not seen, when they ran their tests (the same procedures were followed at LLNL and Luxel), and leaks that weren’t following the typical pattern (Figure 1). The pattern these new leaks were following was as the pressure would increase, so would the leak rate, to a point and then it would start to come down, if it was left at that pressure. These trickling leaks were therefore established as virtual leaks.

A virtual leak is a fixed small volume, usually a bubble or air pocket, that was formed either during the cooling of the sample or its original manufacturing. When pressure is applied to this bubble it will compress forcing out a stream of air, helium, or another substance that the helium leak detector recognizes as a leak. Observation of these types of leaks has shown that the leak rates go up to a certain point and then begin to decrease if the pressure is held constant. The leaks can dissipate in a matter of minutes or in several hours (See Figure 7).

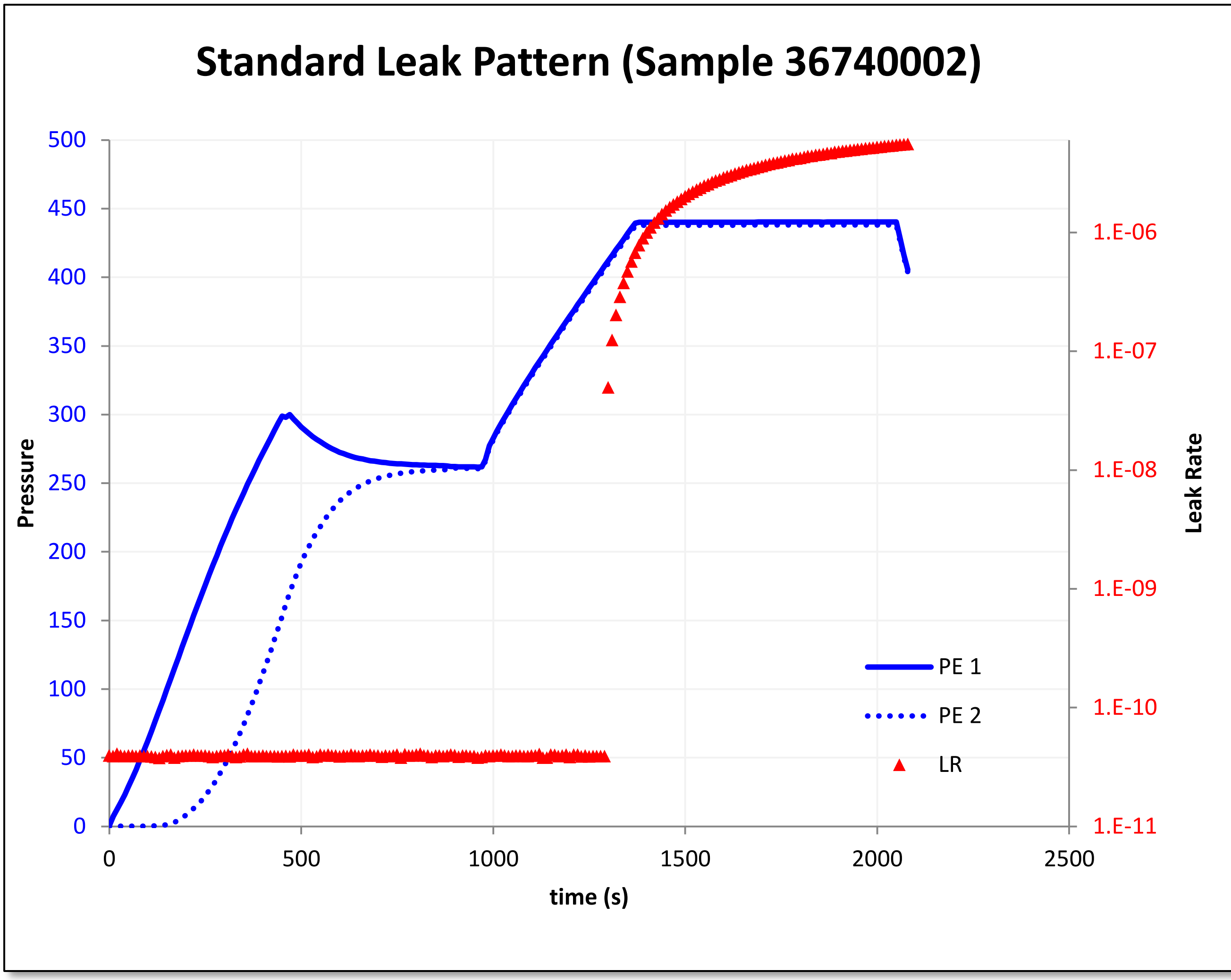
There was discussion for several days over what the cause of these virtual leaks could be, eventually we came to two possible hypotheses. The first hypothesis was that the nodules that dotted the surface were pockets of air that were releasing a stream of air, helium, or some other substance that the helium leak detector was picking up as a leak. The idea was that as the pockets ran out of air to let out the leak rate would decrease, causing the pattern we were seeing. There are several reasons this turned out to be discounted. The first is that the number of nodules on the surface of the washer did not correspond with the extent of the leak rate. Several samples with no nodules had high leaks, and some samples with a multitude of different features had no leaks at all. Secondly, some of the “A” Type windows showed signs of virtual leaks and there are no nodules on the surface of those washers. Finally, the leak rates changed with pressure which should not have had any direct effect on the leak rates if they were in fact caused by the nodules.

The second hypothesis stated that the virtual leaks we were seeing weren’t leaks at all, but permeation because the films were not getting cold enough. When the polyimide films are pressurized they must stretch to accommodate the growing pressure. To accommodate the film stretching the aluminum coating must do the same, but aluminum doesn’t stretch the way polyimide does so it cracks (See Figure 8). When the aluminum cracks helium molecules can get through the film, but only at warm temperatures. Permeation does not occur at low temperatures. When the cold test is run at 9K we expect the sample to be somewhere between that value and approximately 10K off the reading because the temperature gage is on the cold head not within the sample chamber. That being said, as the pressure was increasing on the window the aluminum was cracking and because the temperature was not as low as the system was telling us it was permeation was occurring. The reason this was hypothesized in the first place was because it was noted when loading one of the samples that the cold head was a little loose and slightly off kilter. Every time we had loaded or removed a sample the cold head had become looser and depending on which way the wrenches were moving the sample chamber the cold head’s points of contact could come out of alignment. Sometimes the alignment was in place and the sample would get cold enough, but sometimes it was far enough off that we believe the sample was almost 100 K off the value the temperature gage was reading. This explains the irregularity in which samples we saw with the virtual leaks. After reattaching the cold head to the sample and ensuring the sample was cold enough all of the “B” Type windows had no leaks. To ensure the temperature was accurate we performed the room temperature test to 75 torr on an already tested sample that showed a leak rate, recording the permeation value or starting leak rate, cooling the sample with the 75 torr and watching the leak rate decline with the cooling of the sample. Figure 9 illustrates the confirmation of this hypothesis, and Figure 10 contains the updated table containing the results of all nine of the “A” Type windows and six “B” Type.

In addition to the pressure testing the three of the “A” Type windows were further analyzed under SEM or through multiple retests to determine which type of leak they were exhibiting and what mechanism they were leaking by.

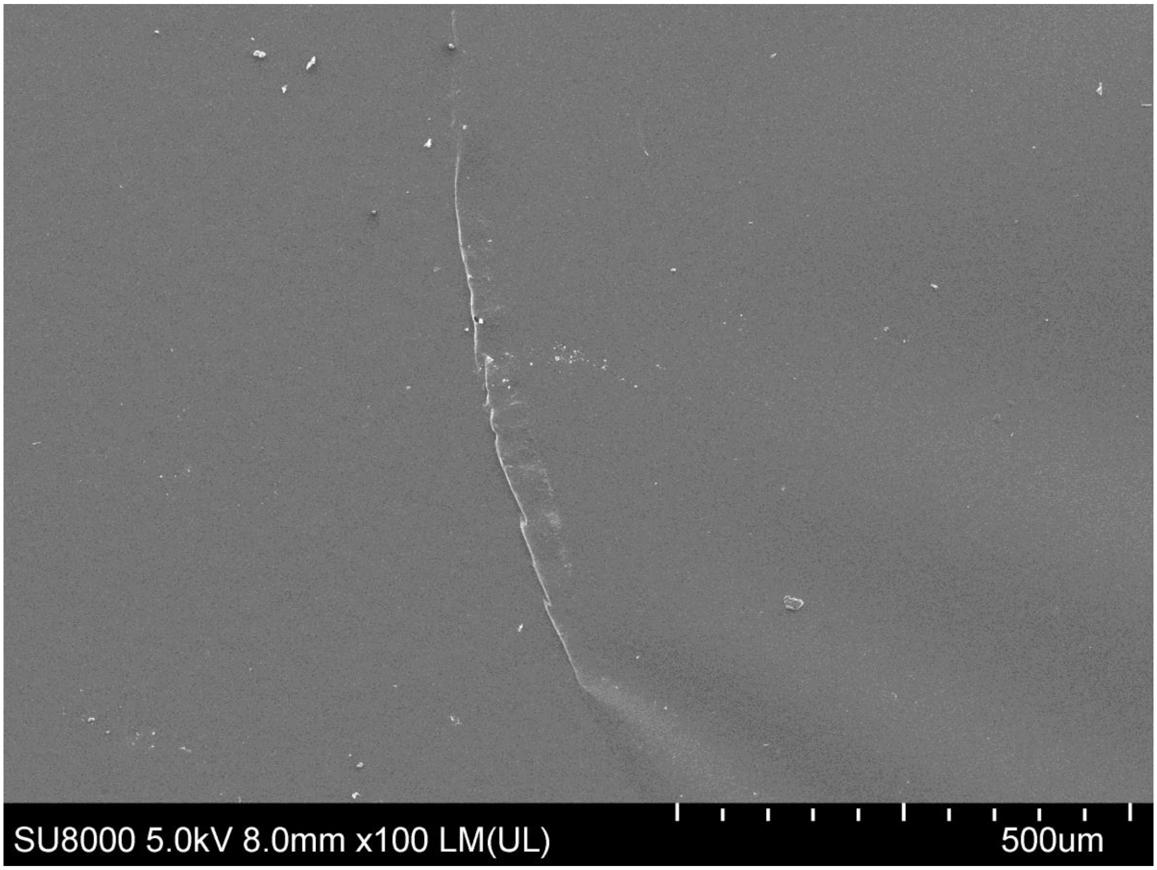
In conclusion “B” Type (2x glue) windows work better than the standard “A” Type windows, so Luxel has switched to production of B type windows for the time being. Overall, there are three possible mechanisms by which the leaks may be occurring: pinhole, tear along edge of freestanding and washer, or channels under the film. The double layer of glue could help/solve two of the three mechanisms; it seems that it has at the very least closed the channels. Going forward the nodules that were visible on the B type washers should be observed until it can be confirmed that they are benign.

Figure 1



In Figure 1 the leak rate remains constant up until about 400 Torr, when a huge jump in leak rate occurs rendering (in this case) the target unusable

Figure 2



On the left side of Figure 2 is the washer and the right is the free standing portion of the film, the tear is right alongside the edge where the free standing portion begins and the washer ends.

Figure 3

LEH Leaks	Initial RT	10K	Final RT	# Seen
Type 1				**4**
Type 2				**18**
Type 3				**4**
Type 4		**Gets worse**		**2**

Figure 3 summarizes the four different types of leaks, orange is a failing leak rate, red is failing worse, and green is passing.

Figure 4

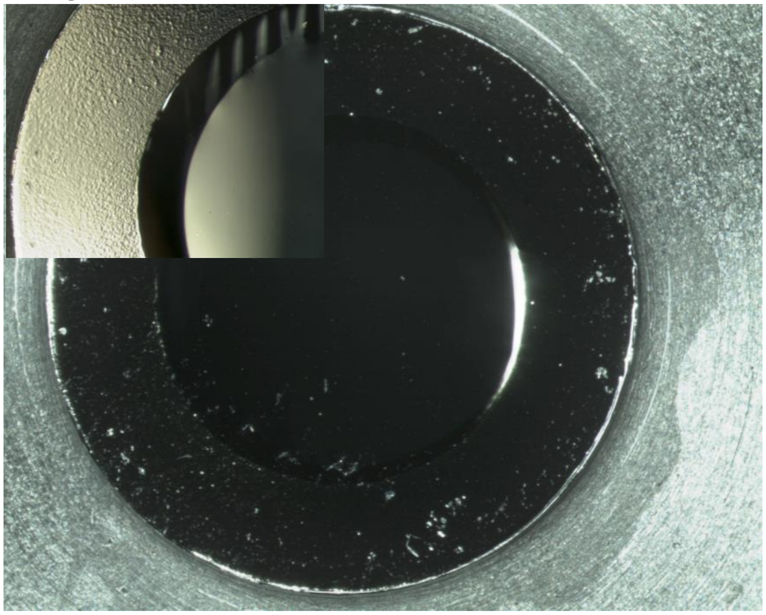


Figure 4 shows an overhead image of the “A” Type window and the inset is higher magnification image of the texture on the surface of the washer

Figure 5

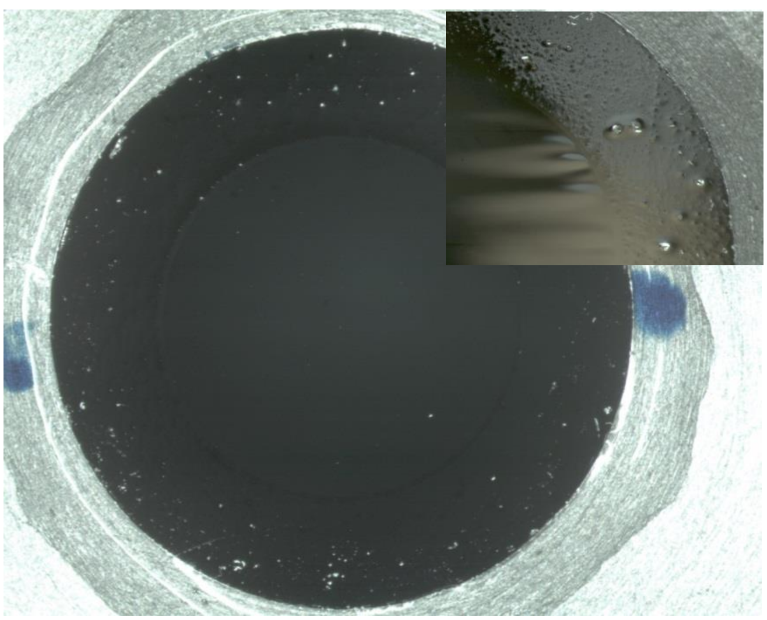


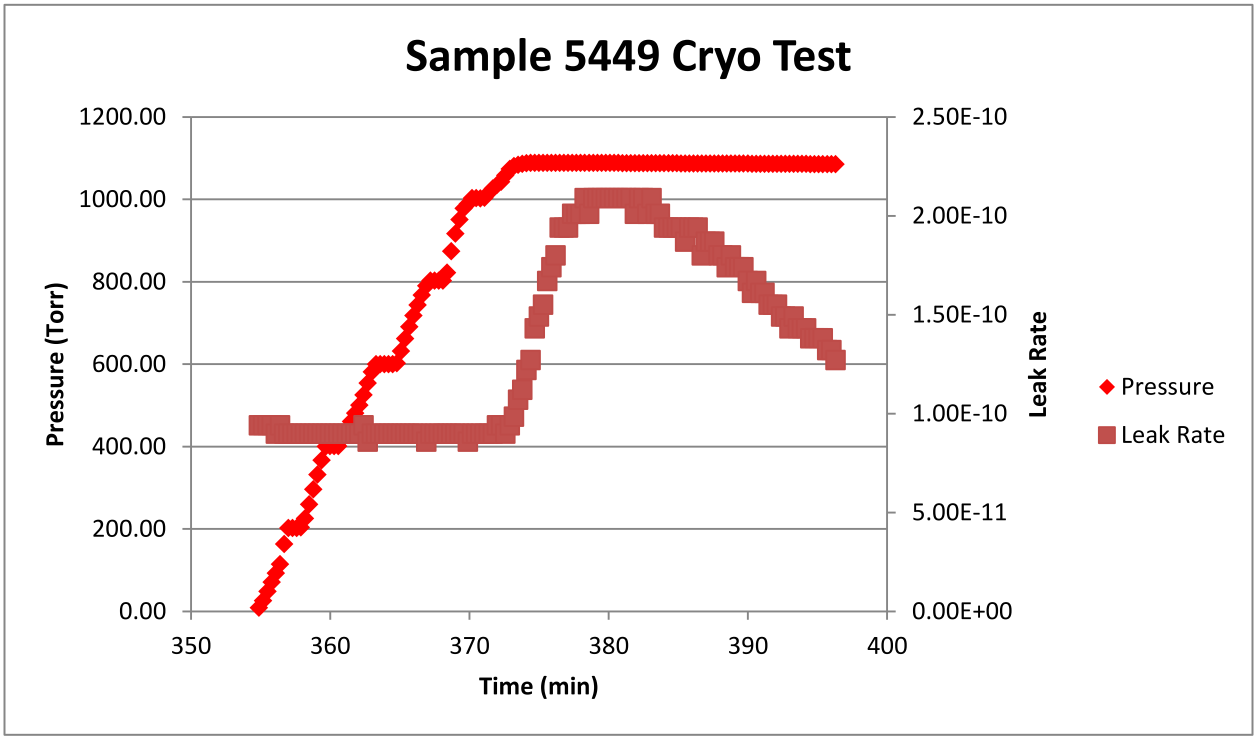
Figure 5 show the “B” type washer and the inset shows the textural features seen on the top of the washer.

Figure 6

		At 10K			
Serial #	**LEH Window Type**	**Max press (torr)**	**Leak Rate (mBarL/s)**	**Virtual leak seen?**	**Pass**
28870971	A	872	2.00E-03	No	No
28870972	A	602	2.80E-08	Possibly	Yes
28870973	A	601.4	1.90E-09	Possibly	Yes
33740329	A	1090.1	8.00E-10	No	Yes
33740330	A	1091.3	1.32E-03	No	No
5438	B	1086.2	3.60E-10	No	Yes
5445	B	1091	1.80E-07	Yes	Yes
5447	B	1094.4	6.40E-07	Yes	Yes
5449	B	1086.6	1.10E-10	No	Yes
5451	B	1087	6.40E-10	No	Yes
5452	B	1091.3	5.30E-06	Yes	Yes

Figure 6 is a summary table of the results from the first round of experiments. The leak rates observed with the “B” Type films are significantly smaller than those of the two failing “A” Type.

Figure 7



Sample 5449 stayed constant to a point, then the leak rate increased and as it was left at 1100 torr, the leak rate decreased almost all the way back down to its original baseline leak rate.

Figure 8

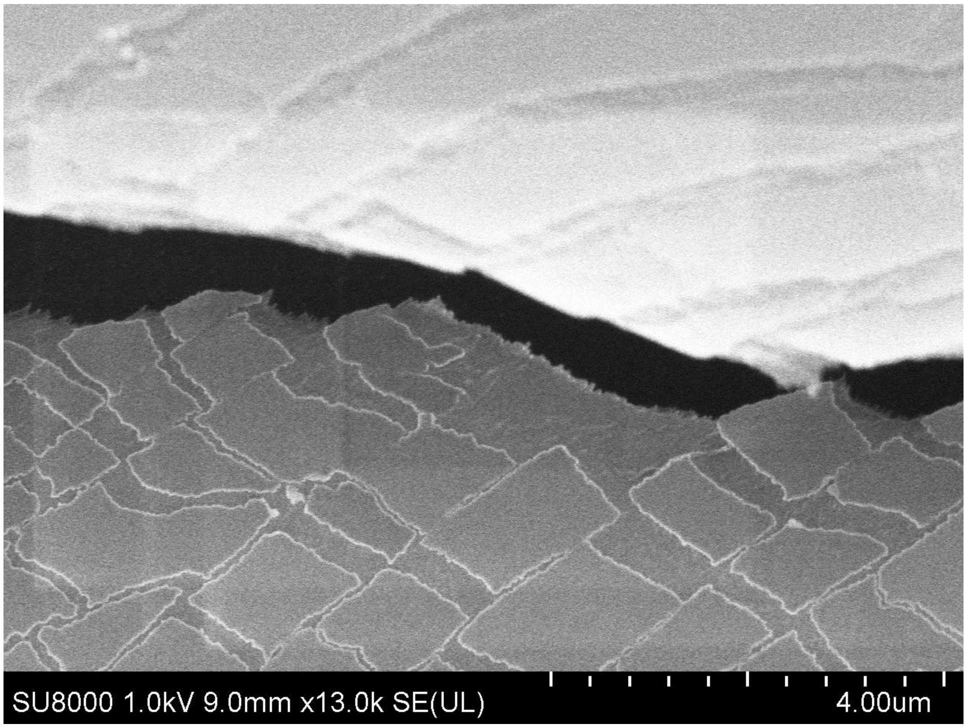
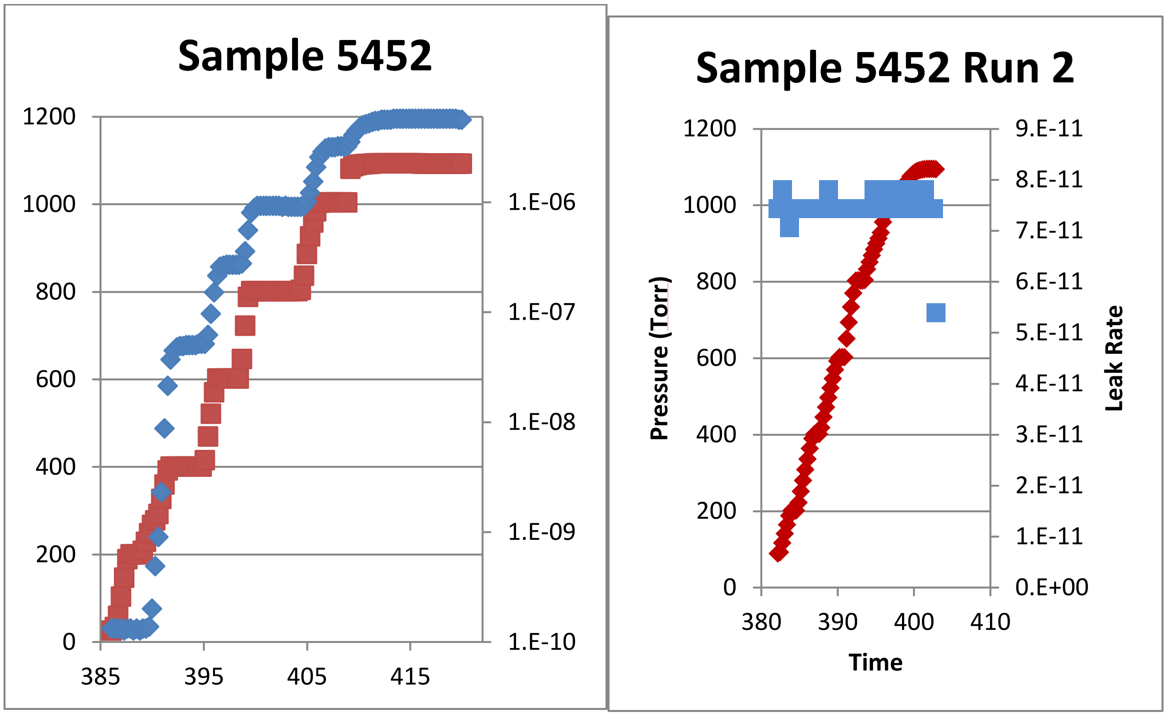


Photo Credit Cindy Larson

This is an SEM of sample 5 which had a leak rate of 1.32E-3. The pieces that look like scales are the cracked pieces of Aluminum coating on top of a torn portion of the polyimide film.

Figure 9



On the left is the first run of sample 5452, in which the red represents pressure and the blue represents leak rate. You can see the leak rate rises with the pressure. On the right after ensuring the temperature was cold enough, you can see there are no virtual leaks and the leak remains constant for the duration of the run.

Figure 10

		At 10K			
Serial #	Type	Max press torr	Leak Rate mbarL/s	Virtual leak seen?	Pass
28870971	A	872	2.00E-03	No	no
28870972	A	602	2.80E-08	Possibly	Yes
28870973	A	601.4	1.90E-09	Possibly	Yes
33740329	A	1090.1	8.00E-10	No	Yes
33740330	A	1091.3	1.32E-03	No	No
33740331	A	1085.7	1.60E-04	No	No
29970606	A	1000.4	1.60E-06	Very small one	No
29970607	A	1087.4	5.00E-11	No	Yes
29970608	A	1083.9	1.10E-10	No	Yes
5438	B	1086.2	3.60E-10	No	Yes
5445	B	1090.7	6.20E-11	No	Yes
5447	B	1091.3	6.00E-11	No	Yes
5449	B	1086.6	1.10E-10	No	Yes
5451	B	1087	6.40E-10	No	Yes
5452	B	1092.6	5.30E-11	No	Yes

This table summarizes the outcome of all of our samples up to 1100 torr, with their most successful run (absence of virtual leaks for the “B” Type windows) The “B” Type windows were more successful across the board, compared to the varied results from the “A” Type.

Acknowledgement

This work was performed under the auspices of the US Department of energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

Work cited:

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