Dynamic Mechanical Analysis of Cryogenic Target Materials

Charles Wan

Penfield High School

Advisor: Kenneth L. Marshall

University of Rochester

Laboratory for Laser Energetics

Summer High School Research Program 2012

Abstract:

Adhesive strength is of critical importance for cryogenic laser-fusion targets when bonding the plasma-polymerized polystyrene copolymer plastic target shell to the support stalk (a narrow-diameter fiber of silicon carbide or Zylon PBO). Norland 68 and Dymax 921 gel, two rapid-curing, low-to-medium viscosity UV curable adhesives, are currently used for target fabrication depending on which fiber material is employed. Neither of these adhesives is rated for operation at cryogenic temperatures, and the interfacial bonding strength of these adhesives to the target shell has not been thoroughly characterized, other than through in-use testing. To compare the interfacial strengths of these two adhesives, a sample preparation method involving a polystyrene loop glued to a polystyrene strip was developed and tested with a Perkin Elmer DMA8000 dynamic mechanical analyzer. This instrument is capable of determining mechanical properties over a wide range of temperatures and dynamic loads. Although results showed some scatter in adhesive-strength data points for both Norland 68 and Dymax 921 gel at cryogenic temperatures, test runs revealed that a decrease in temperature resulted in an increase in interfacial bond strength between the adhesives and the polystyrene. Further research in sample preparation could lead to more consistent results.

Introduction:

The Laboratory for Laser Energetics at the University of Rochester conducts nuclear fusion through the direct-drive approach to inertial confinement fusion using the 60-beam OMEGA laser. In a direct-drive shot, lasers irradiate a cryogenic spherical target containing deuterium and tritium (isotopes of hydrogen). If the target reaches a density of 200-300 g/cm^3 and a temperature of approximately 100 million degrees Celsius, a state of ignition can be achieved, in which the fusion reactions of the hydrogen are self-sustaining.[1] Nuclear fusion is a source of clean, renewable energy, and could be a solution to sustainability issues.

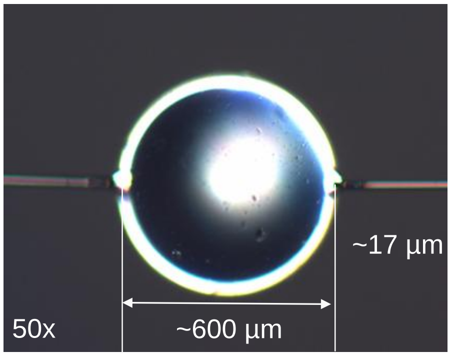
Cryogenic targets are glued to support stalks at room temperature with either Norland 68 or Dymax 921 gel, depending on which fiber material is employed. In the Type 1 geometry, a single stalk of 17 μm silicon carbide fiber is attached to the target with Norland 68, while in Types 4,5, and 6, a tripod with a 14 μm PBO fiber is attached to the target with Dymax 921 gel.[2] Targets are glued to the stalks at room temperature, bonding the adhesive to the plasma-polymerized polystyrene copolymer plastic shell of the target. The entire setup is cooled at a slow rate until ~20 K is reached. The cooling process can last tens of hours, even days.[3] Targets are cooled to cryogenic temperatures to deposit a uniform DT ice layer inside the target. The main purpose of freezing targets is for efficiency; a frozen solid has a much higher density than a warm gas, which makes the initial compression require less energy.[1] Prior to the shot, while targets are sitting stationary, being cooled, or being transferred, several failure modes can occur. In extremely rare cases, the bulk adhesive will fracture. Sometimes, fibers will detach from the target and the adhesive. However, the most prevalent failure mode by far is that the target will separate from the adhesive while the adhesive remains intact. This indicates that the interface of the adhesive bonded to the polystyrene experienced failure. Since little prior research has been conducted in this area of interfacial strength, objectives included characterizing this strength and making comparisons between the adhesive strengths of Norland 68 and Dymax 921 bonded to polystyrene.

The Perkin Elmer DMA 8000 dynamic mechanical analyzer is an instrument capable of determining mechanical properties over a wide range of temperatures and dynamic loads. The DMA 8000 was used to measure the interfacial strengths of Norland 68 and Dymax 921 gel to polystyrene. The DMA measured the amount of force required to break samples that were created specifically to fit the DMA's loading geometry. The DMA has been used to measure properties such as Young's Modulus, tensile strength, compressive strength, damping, and strain to failure in high performance organic fibers and polymers such as Zylon PBO, Kevlar, and M5 PIPD.[4,5,6,7]

Experimental:

Since the DMA 8000 requires a specific loading geometry, a sample preparation method had to be developed. The initial attempt to create samples involved taking two 17 μm silicon carbide fibers and gluing them with Norland 68 to opposite ends of a 600 μm solid polystyrene microsphere (Figure 1(a)). Once the fibers were attached, the adhesive was cured and the ends of the fibers were glued across a hole-punched paper so that samples could be loaded and tested with the DMA (Figure 1(b)). This sample preparation method was designed to closely mimic actual target fabrication.

a.)



b.)

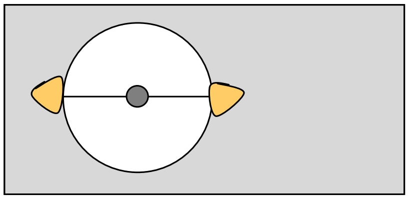
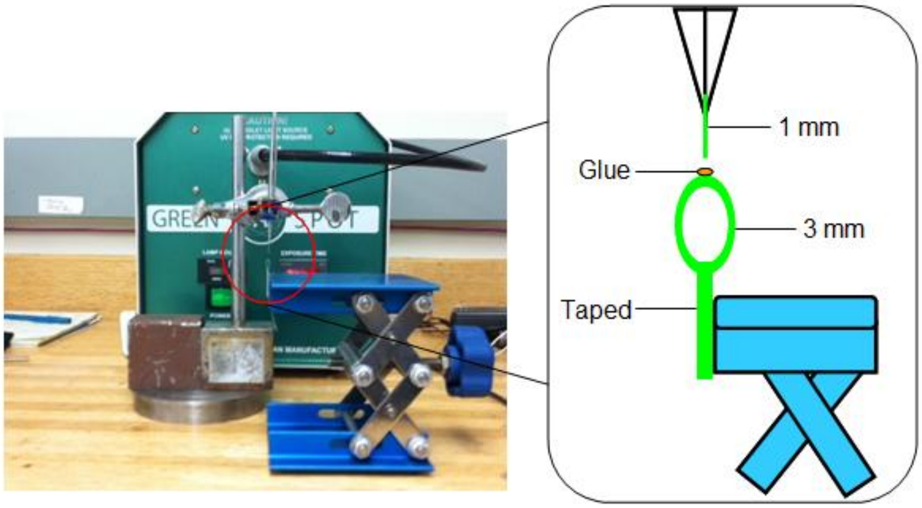


Figure 1: Original sample preparation method. a.) The attachment of the silicon carbide fibers with Norland 68 adhesive to the polystyrene ball is viewed under a Leica microscope. b.) The attached fibers to the polystyrene were glued onto a hole-punched sheet of paper, which was then used to test the strength of the polystyrene to the adhesive in the DMA 8000.

However, some major setbacks prevented this method from being successful. On average, sample preparation time was more than two hours. The glue spot connecting the polystyrene to the silicon carbide experienced natural variation that was uncontrollable. Additionally, samples being loaded into the DMA often had fibers detach from the sphere. Samples were too delicate and fragile, and small amounts of stress or force caused failure.

As a result, a new sample preparation method was developed that was both time efficient and compatible with the DMA. This method, shown in Figure 2, involved gluing a polystyrene strip to a polystyrene loop with 0.5 μL of adhesive. A polystyrene loop was formed by taping the ends of a 3 mm thick, 53 mm long polystyrene strip together. Once the loop was formed, it was attached to the side of a lab jack. A single polystyrene strip 1 mm wide was held by a chemistry clamp with tweezers. 0.5 μL of adhesive was put on the loop, and the lab jack elevated the polystyrene loop with the adhesive into the polystyrene strip. Once this was done, the adhesive was cured for twenty seconds with a Green Spot UV light source with a wavelength of 300-480 nm. The entire procedure took less than ten minutes, allowing for an efficiency previously unachievable with the old sample preparation method.

a.)



b.)

Figure 2: Pictures of sample preparation setup, materials, and completed sample. a.) The chemistry clamp holds the polystyrene strip, while the polystyrene loop is attached to the blue lab jack. The entire setup is placed in front of the Green Spot light source for immediate curing. b.) This is a completed sample.

Finished samples were loaded into the DMA 8000 (Figure 3). Both room temperature and cryogenic tests were conducted for Norland 68 and Dymax 921 gel samples. For cryogenic tests, samples were cooled at a rate of 5 K/min until 98 K was reached in an effort to mimic the extremely slow cooling rate of actual targets. (Samples were not tested at 20 K because the DMA 8000 is unable to conduct tests at temperatures lower than 98 K.) The lower cooling rate ensured that the adhesive would break in a manner that tested the interfacial strength between the polystyrene and the adhesive, as opposed to the bulk adhesive strength itself. Once samples had been cooled, an increasing load rate of 0.5 N/min was applied on the samples until breakage occurred. The mean force to breakage was measured with the DMA 8000's software on Microsoft Excel.

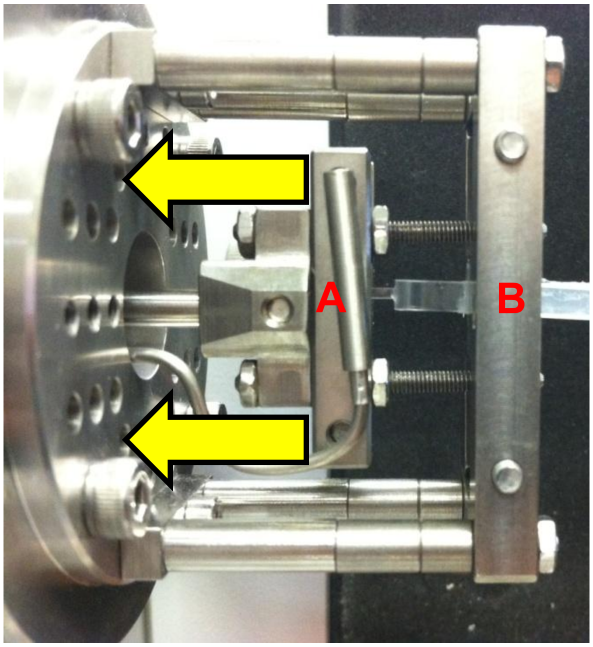


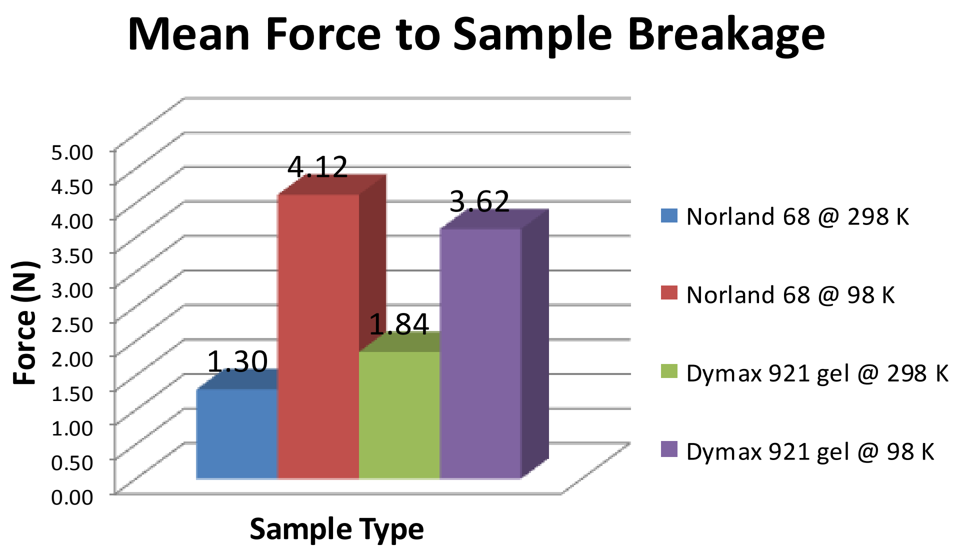
Figure 3: Sample loaded into DMA for testing. Clamp "A" applies the increasing load to the strip, while Clamp "B" holds the loop end of the sample. The force required to break the sample is then measured.

Results and Conclusion:

The results are summarized in Figure 4. The interfacial strength between the polystyrene and the adhesives increased as temperature decreased. For both adhesives, the mean force required to break samples at cryogenic temperatures was higher than the mean force required to break them at room temperature. Although Norland 68 samples required slightly more force at cryogenic temperatures to break than Dymax 921 gel samples, an insufficient number of data points prevented a statistically significant comparison between the two adhesives. Standard deviations also exist within results that make a direct comparison between the interfacial strength of the Norland 68 and Dymax 921 gel with polystyrene not possible due to a relatively wide variation in data.

Of additional importance was that the Norland 68 and Dymax 921 gel samples broke in a “golf-tee” formation (Figure 5), in which the bulk adhesive was not fractured, but the adhesive cleanly broke from the polystyrene on the loop. This is of critical importance because it indicates that the mean force to breakage was measuring the interfacial strength between the polystyrene and the adhesive, as opposed to the bulk adhesive strength.

a.)



b.)

Temp. (K)	Adhesive	Trials	Total Force (N)
298	NOA 68	10	1.30±0.52
	Dymax	8	1.84±1.16
98	NOA 68	9	4.12±0.86
	Dymax	7	3.62±1.15

Figure 4: Summary of results. a.) A graph of mean force to breakage for Norland 68 and Dymax 921 gel at both room and cryogenic temperatures. b.) A table that summarizes results with number of trials and standard deviation for each condition.

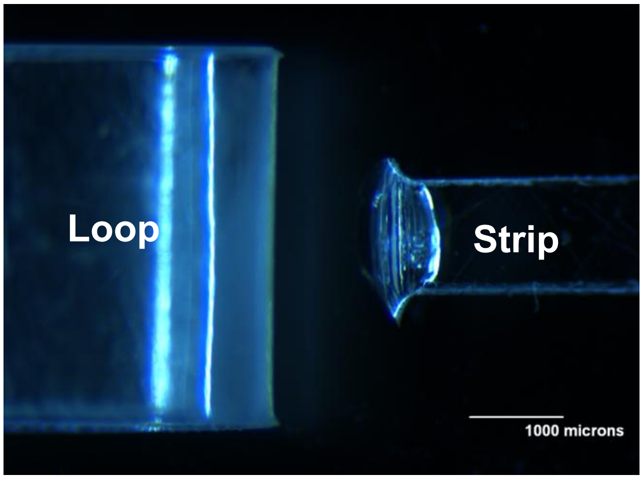


Figure 5: Microscope view of sample after load was applied. The "golf-tee" formation is evident, as the adhesive on the strip made a clean break from the loop.

Future Work:

Since a relatively large standard deviation exists, additional research is needed to maximize consistency in data. Some aspects of the sample preparation method can be altered to achieve more precision. For example, standardizing the glue spot dimensions could stabilize results. Although a volume of 0.5 μL is already the standard, the three-dimensional shape of the glue spot tends to experience variation, which could impact the consistency of results. Additionally, an alternate curing system which would take the entire sample preparation setup and place it onto a turntable with the UV light shining on the sample from a fixed source could lead to more precise data points (Figure 6).

Once consistent data is achieved, future work could focus more on characterizing mechanical properties such as the average strength, Young's Modulus, damping, and viscoelastic nature of the adhesive to polystyrene interface.

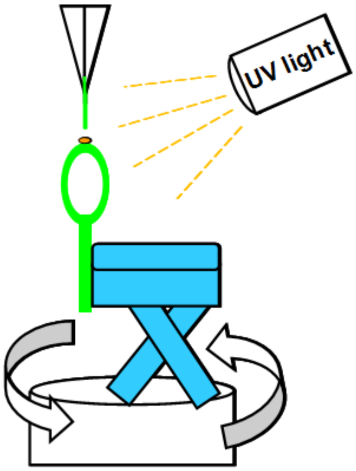


Figure 6: Possible rotating curing system. The entire sample preparation setup would be placed on a rotating stand, as illustrated. A rotating curing system would ensure that the adhesive would be cured in a standardized, consistent method.

Acknowledgements:

I would like to thank the Laboratory for Laser Energetics and Dr. R. Stephen Craxton for giving me the opportunity to conduct research and for reviewing my project report. I would also like to thank my advisor, Mr. Kenneth L. Marshall, for giving me instruction and guidance throughout this experience. Likewise, I am grateful for the additional support from Dr. Stephen Jacobs. Finally, I would like to acknowledge Mathias Ferber, Mackenzie Hall, and Debbie Saulnier for their assistance with the completion of my project.

References:

1.) L.H. Gresh, R. L. McCrory, J.M. Soures, "*An Introduction to Inertial Confinement Fusion,"* Laboratory for Laser Energetics, Rochester, March 2009.

2.) Brian Rice, Ken Marshall, Stephen Jacobs, "*Project for Target Vibration Reduction at Time of Shot,*" Presentation, March 1st, 2012.

3.) Dave Turner, private communication.

4.) Mackenzie Hall, "DMA Measurements and comparisons of M5 PIPD and Zylon PBO fibers," Memorandum, May 25th, 2012.

5.) "Assessment of compressive properties of high performance organic fibers," A. Andres Leal, Joseph M. Deitzal, John W. Gillespie Jr., *Computing Science and Technology 67* (2007) 2786-2794.

6.) "Interfacial behavior of high performance organic fibers," A. Andres Leal, Joseph M. Deitzal, Steven H. McKnight, John W. Gillespie Jr., *Polymer 50* (2009) 1228-1235.

7.) "Compressive Strength Analysis for High Performance Fibers with Different Modulus in Tension and Compression," A. Andres Leal, Joseph M. Deitzal, and John W. Gillespie Jr., *Journal of Composite Materials* (2009) 43: 661.