Cryogenic Permeability of Polyimide Shells

James Wang
McQuaid Jesuit High School
Rochester, NY

2001 Summer Research Program for High School Juniors
Advisor: Dr. David Harding

LABORATORY FOR LASER ENERGETICS
University of Rochester
250 East River Road
Rochester, NY 14623-1299

Polyimide shells are used as targets in Inertial Confinement Fusion (ICF) experiments. These shells are filled with DT gas, then cooled to low temperatures for cryogenic laser shots. During target preparation, shells require careful pressure and temperature control due to their sensitivity. The pressure is governed partly by permeation through the shell wall, which can only be determined with the knowledge of the shell permeability over a wide temperature range. A new method was developed to test the permeability of shells at temperatures ranging from 123K to 23 K. This method produced results at room temperature comparable to those using current setups. Arrhenius' relationship was observed from room temperature to 120K and the activation energy for permeation was determined for shells prepared under different processing conditions. The results will allow the permeability of shells to be predicted for various temperatures.

I. INTRODUCTION

Nuclear fusion is a promising source of energy for the future. One of the current approaches toward nuclear fusion is Inertial Confinement Fusion, which uses lasers to compress a spherical shell no larger than 1 mm filled with DT gas. The shells used in the OMEGA Laser System at the University of Rochester are hollow spheres made of Polyimide with walls of 3-5 μm thick and weight of 0.2-0.4 mg (see Figure 1). The design of an OMEGA target calls for a solid 100 μm thick layer of solid DT uniformly adhered to the inner surface of the shell. To prepare these cryogenic targets, the shells must first be filled to 1000 atm at 300K, then cooled slowly to 19K. The cooling process induces a pressure gradient across the shell wall due to non-uniform temperature distribution in the pressurization chamber[1]. To avoid too high of a pressure gradient, which will cause the shell to buckle or burst, the cooling rate must be slow. The exact rate of cooling can only be determined by knowing permeability (Kp) of the shell wall from cryogenic to room temperature. In this study, a new setup was developed to test the permeability of polyimide shells at a broad temperature range (see Figure 2). This setup can also be used to determine the properties of the polyimide shell prepared under the specific processing conditions at room temperature, which are well characterized, and at cryogenic temperatures, which are unknown.

II. EXPERIMENTAL

The schematic drawing of the apparatus for measuring the shell Kp at low temperatures is shown in figure 2. The setup consists of the supply line, pressure transducer (MKS Instruments 722A, 1000 torr), temperature controller (Cole Parmer DigiSense), sample chamber (11/3 inch conflats with 3/8 inch pore), cooling mechanism, and leak detector (Edwards Spectron 600D). The sample chamber had a system of three sealed conflats. The permeation surface was glued to the middle conflat. Upstream of the sample chamber was the supply line containing Helium and the pressure transducer. A copper ring surrounding the chamber with two fingers immersed in nitrogen (77K) cooled the sample chamber. A 400-watt cartridge heater and a K-type thermal couple were attached to the copper housing to allow for temperature control. The temperatures measured include 130K, 200K, 251K, and 295K. The leak detector measured the Helium leak rate in the unit of standard cc/s, which was then converted to mol/s using the ideal gas law.

Equation 1:

$$n / t = (Kp \times A \times \delta P) / l$$

n = mols of gas
t = time (s)
Kp = permeability constant
A = surface area of membrane
δP = pressure differential
l = thickness of membrane

The shell-plate was prepared by drilling 6 shell-sized holes in a miniature brass plate (Area: 1.5 cm^2, Thickness: 0.25 cm), as shown in figure 3. Six shells were glued to the plate using Epoxy, which is impermeable to gases, in a manner that covered the holes. Holes were then poked through the shells on the hemisphere below the brass plate. This allowed gas to enter the shell through the hole and permeate through only one layer of the shell membrane. This shell-plate was attached to the central conflat using Epoxy. The diameter of the shells used and the thickness of shell walls were measured using previously reported methods[1].

This new setup was calibrated against existing setups, including a flat-film apparatus[1] and a Time Constant setup[2] for shells. The shells tested were either cured in N_2 or air[1]. The upstream pressure was approximately 1.4 atm, and the type and grade of gas used was UPC Helium.

III. RESULTS & DISCUSSION

The accuracy of the measurements taken on the new shell-plate setup at room temperature is confirmed by comparing its results with those using other testing setups (as shown in Table 1). There are, however, minor differences between the data, which may be attributed to the following reasons:

(1) Variations in the permeability due to the fundamental microstructure differences between film and shells. Processing conditions allow shells to be more uniformly thick than films due to the difference in surface area.

(2) The pressures used for each apparatus are different. In the Time Constant setup, the shells are filled with 7.5 atm, while in the other two setups, only 1.4 atm is applied to the shells or film. The Kp of polymeric materials usually have a dependence on pressure, so the higher the pressure, the higher the Kp.

At low temperatures, the results obtained using the new setup also agree with previous measurements using flat films[1] (as shown in Figure 4). The temperature dependence of Kp of both air and N_2 cured samples followed Arrhenius' relationship (Equation 2) over the experimental temperature range (Table 2). The thermal activation energy for permeation (Ep) obtained from the slope of the linear relationships in Figure 4, which were 18.1 kJ/mol and 18.6 kJ/mol for N_2 and air cured samples respectively, and the following equation.

Equation 2:

$$Kp = Kp_o \times e^{-Ep/RT}$$

It was also observed that the N_2 and air cured samples maintained the same permeability relationships at low temperatures.

IV. CONCLUSION

Knowledge of permeability at a broad temperature range is important to Inertial Confinement Fusion experiments because permeation is a critical process for preparing cryogenic targets. With the new shell-plate setup, permeability of shells can now be measured at a broad temperature range. The activation energy for permeation has been determined for polyimide shells in this study, which will allow for the prediction of the shell permeability at low temperatures. It was also observed that shells prepared under various curing conditions maintained their permeability differences at cryogenic temperatures.

V. ACKNOWLEDGMENTS

My progress this summer would not be possible without the help of the entire Target Fabrication departments at the University of Rochester Laboratory for Laser Energetics. I would especially like to thank Dr. David Harding for incorporating me into his department. Sal Scarantino patiently helped me with my testing apparatus. Feng-Yu Tsai directed and supervised my entire project. He is the largest contributor to my progress, and I thank him for a great time this summer.

I would like personally to thank Dr. R. S. Craxton and all of University of Rochester Laboratory for Laser Energetics for allowing me to explore the world of scientific research. During my research time at the laboratory, I have learned more than any high school course could ever teach me. I hope that more students will be exposed to this wonderful program and will embrace the scientific frontier as I have this summer. Thanks again.

VI. REFERENCES

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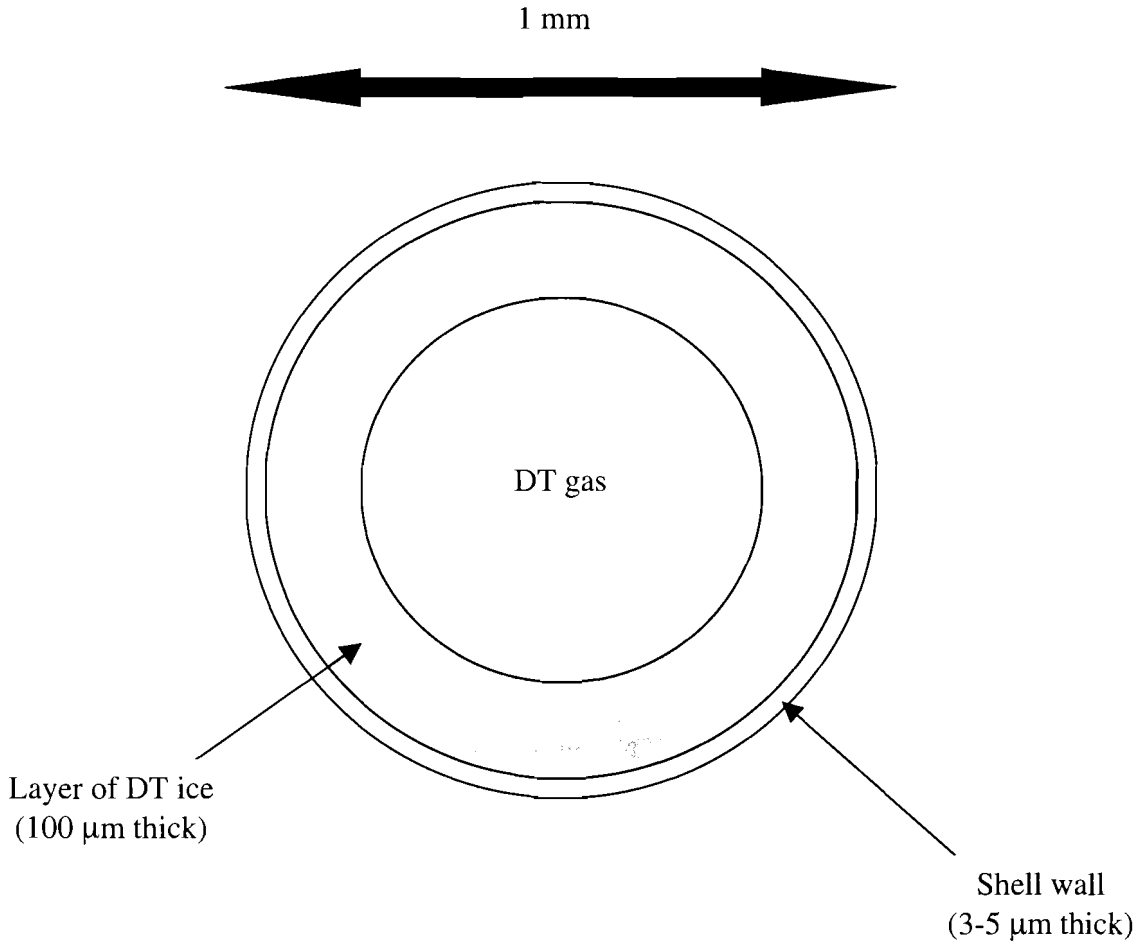

FIGURE 1: A schematic of the design of OMEGA targets.

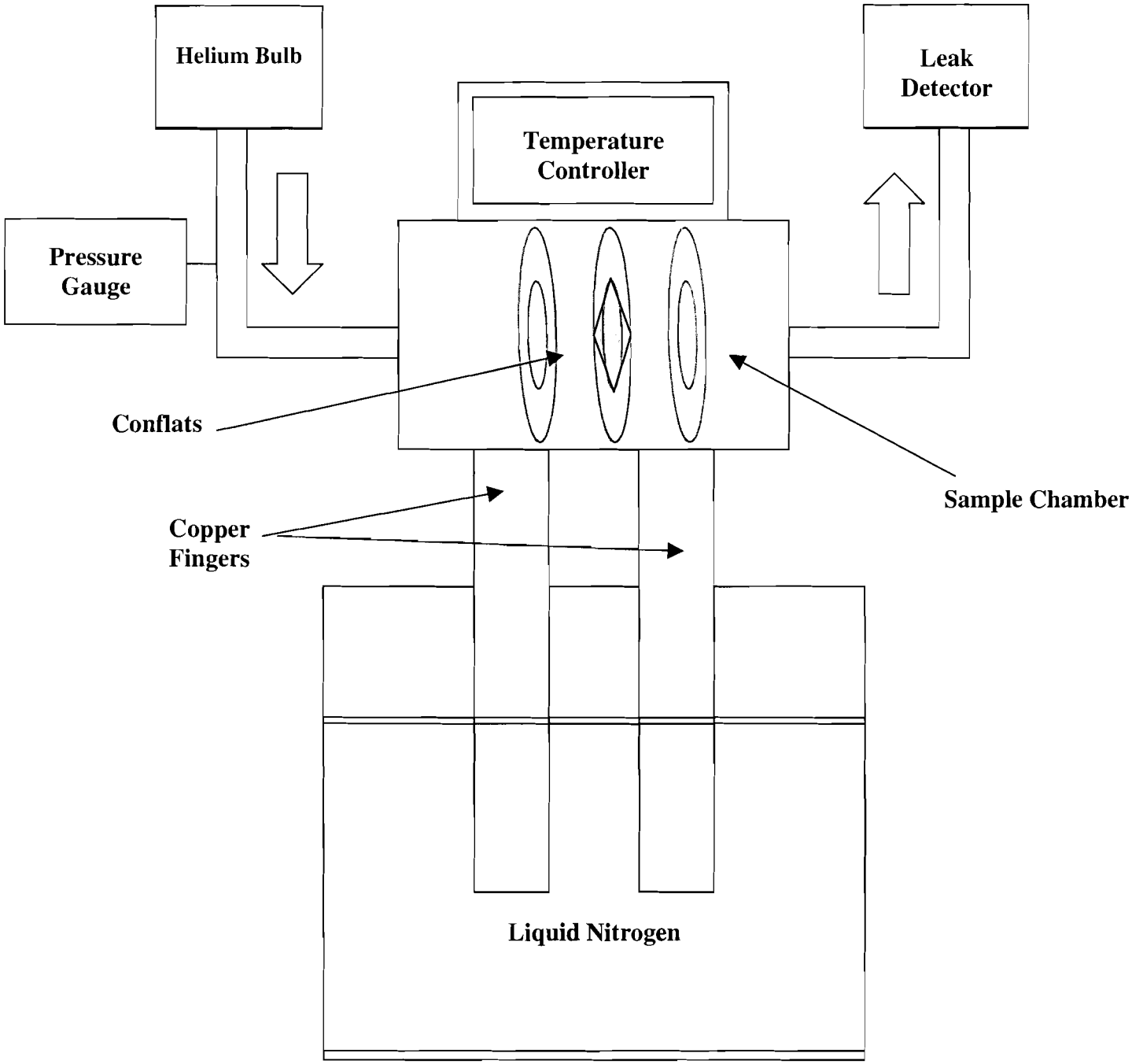

FIGURE 2: A schematic drawing of the experimental setup. Upstream of the sample chamber are the Helium reservoir and pressure gauge, and downstream is the leak detector. The three conflats create an airtight chamber where the film can be tested. Copper fingers extending from the sample chamber are dipped in liquid nitrogen. The temperature of the system is regulated by the temperature controller.

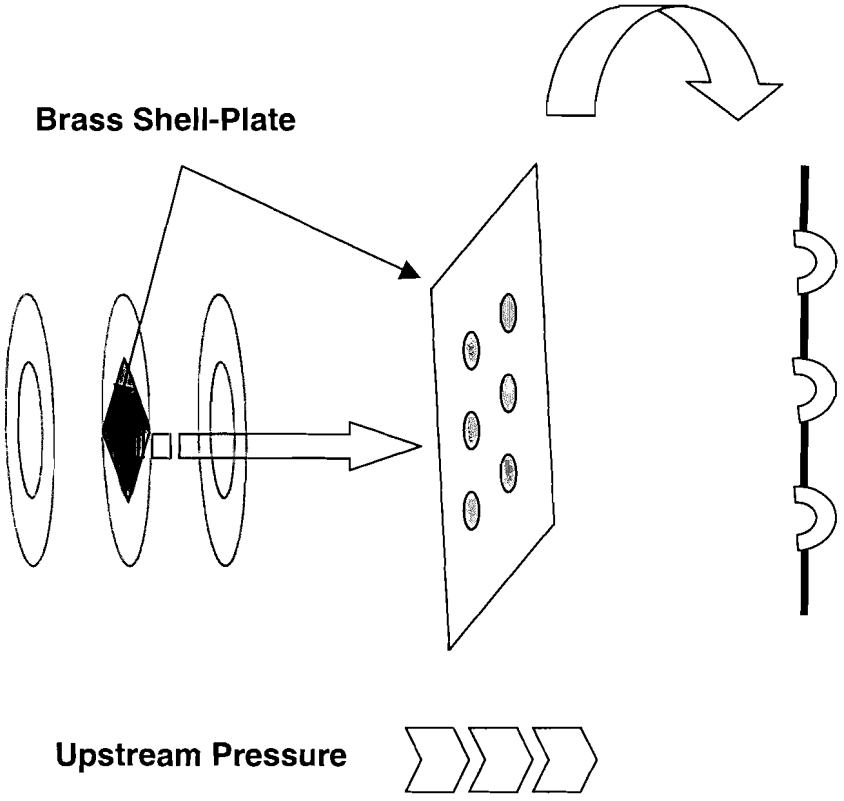

FIGURE 3: The design of the sample chamber. The shell holder was a brass plate with 6 holes drilled through it. Shells were glued to the plate using Epoxy glue and punctured on one side. The shell holder was attached to a conflat flange using Epoxy glue.

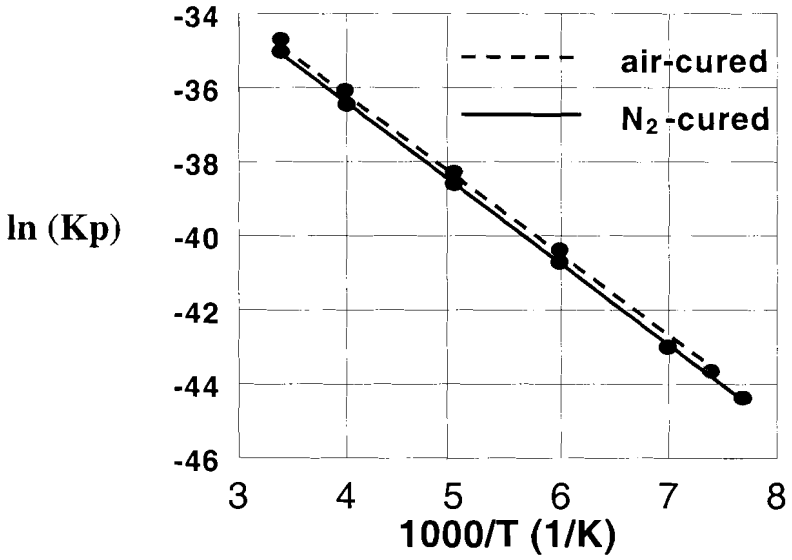

FIGURE 4: Temperature dependence of Helium Kp of air & N_2 cured samples.

$\frac{mol \bullet m}{sec \bullet Pa \bullet m^2}$	Time Constant	Flat-Film	Shell-plates
K_p	5.9E-16	4.6E-16	4.8E-16

TABLE 1: Comparison of the Helium permeability of polyimide measured using different setups. The permeability results of polyimide film tested in the flat-film setup were obtained from previous work.

$\frac{mol \bullet m}{sec \bullet Pa \bullet m^2}$		Temperature (K)			
		295	250	200	167
Curing Medium	Air	8.6E-16	2.4E-16	2.4E-17	3.0E-18
	N_2	4.8E-16	1.2E-16	1.4E-17	1.6E-18

TABLE 2: Helium permeability of air & N_2 at cryogenic temperatures.