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Exposure of NIF relevant polymeric samples to Deuterium-Tritium gas at elevated temperature and pressure

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ABSTRACT

The purpose of the experiments described in this paper was to expose samples of polymeric materials to a mixture of deuterium-tritium (DT) gas at elevated temperature and pressure to investigate the effects (i.e. damage) on the materials. The materials and exposure parameters were chosen with to be relevant to proposed uses of similar materials in inertial fusion ignition experiments at the National Ignition Facility.

Two types of samples were exposed and tested. The first type consisted of 10 4-lead ribbon cables of fine manganin wire insulated with polyimide. Wires of this type are proposed for use in thermal shimming of hohlraums and the goal of this experiment was to measure the change in electrical resistance of the insulation due to tritium exposure. The second type of sample consisted of 20 planar polymer samples that may be used as ignition capsule materials.

The exposure was at 34.5 GPa (5010 psia) and 70° C for 48 hours. The change in electrical resistance of the wire insulation will be presented. The results for capsule materials will be presented in a separate paper in this issue.

INTRODUCTION

The purpose of these experiments was to expose samples of polymeric materials to a mixture of deuterium-tritium (DT) gas at elevated temperature and pressure to investigate the effects (i.e. damage) on the materials. The materials and exposure parameters were chosen to be relevant to proposed uses in inertial fusion ignition experiments at the National Ignition Facility. Proposed targets for ignition experiments are 2 mm diameter spherical shells made of either a beryllium alloy or a polymer.¹ These targets will be mounted within a hohlraum which will be instrumented with thermometers and heaters for developing the correct thermal profile to form spherically symmetric layers of cryogenic solid deuterium-tritium (DT) within them.² The symmetrization process, beta-layering, is driven by the heat generation of the tritium beta decay,³ but may also be enhanced by the introduction of energy via infrared laser light. One of the potential methods for filling the polymer target shells with the correct amount of DT is to diffusion fill the target by immersion of the entire hohlraum in a pressure vessel filled with DT gas at high pressure and elevated temperature, followed by a cooldown to cryogenic temperature so that the excess DT may be pumped out of the pressure vessel without causing the target shell to burst. In this proposed fill process both the polymer shell and the polymeric insulation of the wires to the hohlraum heaters and thermometers would be exposed to the high pressure DT. In the experiment described in this paper test samples of possible target materials and wire insulation were subjected to a DT exposure at elevated temperature and pressure similar to that of the proposed diffusion fill method in an effort to determine if the materials are damaged by tritium.

DESCRIPTION OF EXPOSURE SYSTEM

In order to expose the samples a new exposure capability was developed at the LANL tritium facility. A commercially available pressure vessel was obtained and mounted within a heater jacket and attached to the existing facility high pressure tritium gas handling system, Figure 1. The stainless steel vessel is sealed with a gold plated stainless steel sealing ring to effect a high pressure tritium compatible leak tight seal. The sample space within the vessel allows for moderately large samples with a cylindrical volume of 50 mm dia and 225 mm length. The operational temperature and pressure limits of the vessel are 70° C and 35 GPa (5100 psia) with engineered over temperature and over pressure protection devices set at 95° C and 46 GPa (6700 psia). The vessel is directly connected to a pressure gauge and the rupture disk that sets the over pressure protection limit, and these three components are isolated from the facility high pressure manifold with two manual valves in series. While a nominal 50% deuterium 50% tritium isotopic mixture was desired for the test reported in this paper, the facility has the capability to mix any isotopic ratio of hydrogen.

DESCRIPTION OF SAMPLES

Two types of samples were prepared. The first type consisted of 10 4-lead ribbon cables of #36 manganin wire insulated with single build polyimide. These wires were commercially available, produced by MWS Wire Industries.⁴ The 10 ribbons were wound under tension around an aluminum spool of triangular cross section. The vertices were rounded with a radius of 1 mm, chosen to be similar to the tightest bends anticipated for hohlraum wiring. The 40 leads were prepared at one end with a copper pin to facilitate attaching a meter. The second type of sample consisted of 20 planar polymer samples representing materials that may be used in capsule fabrication. A separate paper in this issue⁵ will detail the results of this study.

PRE-EXPOSURE MEASUREMENTS OF THE WIRE SAMPLES

The resistance between each wire and the aluminum spool was measured using an AVO BMM80 megohmmeter that was calibrated by the LANL calibration lab. The resistance data is given in Table 1 and is only summarized here. Thirty one of the 40 leads were measured initially (February 14, 2005). The meter has the capability of measuring insulation resistance at 50, 100, 250, 500, and 1000 volts and the maximum resolvable resistances are 10, 20, 50, 100, and 200 G Ω , respectively. The leads were tested at successively higher potentials starting with 250 V. The wires are manufacturer rated to 1200 VDC. Twenty two of the 31 (71%) wires initially tested had resistance readings of >200 G Ω , i.e. the insulation was fully functioning. Of the remaining 9 wires tested, most of them had greater than 50 G Ω at 500 V or lower and had a significantly lower resistance at 1000 V. Most of these 9 exhibited arcing between the other bare end of the wire and the spool at 1000 V, so it is not clear whether or not the insulation was fully functioning or these low measurements were due to current travel through arcing.

EXPOSURE PROCEDURE AND PARAMETERS

The samples were exposed to DT gas at the same time. The planar samples were passed into the glovebox at LANL tritium facility in their sample holders, Figure 2. They were transferred from the sample holders to the exposure hardware with a vacuum tweezers. The hardware was screwed together and inserted into the exposure vessel, Figure 1. The vessel was sealed, attached to the facility high pressure manifold, and evacuated. On March 1, 2005 the vessel was heated to 70° C over an hour's time and held at that temperature for a couple hours before pressurization with DT was begun. The vessel initially leaked at low pressure and so it was evacuated and allowed to cool down. The main vessel seal was tightened and the procedure was repeated the next day. The vessel was heated in an hour, allowed to sit at temperature for

one hour and then pressurized over 40 minutes to 34.5 GPa (5010 psia) with DT (pressurization complete at 11:10 a.m. March 2, 2005).

The samples were exposed to the 34.5 GPa (5010 psia) DT at 70° C until 11:47 a.m. March 4, 2005. At that point the vessel heating was turned off and cooling began. At 12:30 p.m. the gas pressure was let down, the vessel fully evacuated, and the cooling continued (cooling is an exponential decay back to 20° C with a time constant of approximately 5 hours). On March 7, 2005 the vessel was opened and the samples removed into the glovebox. The planar samples were repackaged into their sample holders while in the glovebox. The sample holders were removed from the glovebox to be decontaminated and transported back to LANL Polymers and Coatings Group (holders A&B on 3/9/2005, C&D on 3/21/2005, and E on 3/24/2005) for the post exposure measurements. The wire samples were left in the glovebox for post measurements.

The DT gas used was analyzed both before and after the exposure. The gas was 1% H, 49% D, 45% T, and 5% ³He (molecular percentages rounded to the nearest whole percentage are reported here). The isotopic analysis of the gas was performed in the LANL tritium facility using a high resolution magnetic sector mass spectrometer (VG 3001). There was no significant change between the initial and final analyses, though there was a slight increase in both H and oxygen. After the samples were removed from the exposure vessel the glovebox activity level increased from about 20 to about 100 mCi/m³ as the samples and exposure vessel offgassed.

POST-EXPOSURE MEASUREMENTS SAMPLES

After the exposure to DT the resistance between each wire and the aluminum spool was measured using an AVO BMM80 megohmmeter that was calibrated by the LANL calibration lab and introduced into the tritium glovebox. The first post exposure measurements were made on

March 10 and 11, 2005, and some of the wires were measured several more times over the next month to observe the long term effects on the insulation.

The resistance data is given in Table 1 and is only summarized here. The resistance of the insulation of 39 leads was measured. Eight leads were measured on March 10, 2005 and 31 were measured on March 11, 2005 (6 and 7 days after the exposure ended). Of the leads measured on the first day the maximum insulation resistance was 15.0 G Ω at 100 V (the same lead was measured to have 9.9 G Ω at 250 V). The others ranged from 6.2 to 12.0 G Ω . Of the leads measured on the second day the maximum resistance was 3.66 G Ω at 250 V (the higher scales were not used as the resistance measured fell within the 250 V scale and it was desired to avoid creating arcing within the glovebox) and the minimum resistance was 760 Ω . Most of the measurements fell within the range of 1.5 to 2.5 G Ω . This showed a dramatic drop in insulation resistance compared to the measurements made prior to the exposure.

Three of the leads measured on March 10, 2005 were re-measured on March 11, 2005 (about 18 hours later) and in all three cases the latter measurement was significantly (a factor of 3 or more) lower than the former measurement. This suggests that the “damage” to the insulation from tritium absorbed/exchanged into it was ongoing. However the selected leads that were measured several times further over the next month did not show significant change from the values measured on March 10 as shown in Figure 3.

LESSONS LEARNED

In performing this exposure and making the post exposure measurements several observations were made that could be used to improve future operations in this or other facilities. One of the issues that needed to be dealt with was the contamination of the planar sample holders. These brass sample holders (Figure 2) were left in the tritium glovebox while the

samples were in the exposure vessel. During that time they absorbed enough surface contamination, so that an extensive effort needed to be expended to decontaminate them. For future operations their time of residence in the glovebox should be strictly limited to the time necessary to unload the samples before the exposure and to reload them after the exposure. A second set should be available (in this case time and cost would have allowed this redundancy, though that might not be feasible for complex or expensive holders) so that the samples could be unloaded into a fresh set to minimize the contamination of the set sent out of the tritium facility for analysis. These samples holders were fabricated with knurled surfaces to help facilitate holding with glovebox gloves. Knurled surfaces however increase the surface area of the piece and are more difficult to decontaminate than smooth surfaces. Decontamination would have been better facilitated by a design that was smooth (maybe even polished) with appropriate handling hardware, e.g. a spanner wrench designed to replace the function of the knurling.

The cut ends of some of the wire leads appeared to have been a source of arcing that dominated the resistance measurements rather than the actual insulation characteristics. In any future tests of wires the ends which are not attached to the meter should be insulated or left free to be pulled a sufficient distance from the body of the spool.

SUMMARY

We have developed a new capability to expose large samples to Deuterium-Tritium mixtures at elevated pressures and temperatures. Polymer samples key to ignition hohlraum performance were exposed to DT at elevated pressure and temperature. The change of resistance of polyimide wire insulation was measured and found to be significant.

ACKNOWLEDGEMENTS

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REFERENCES

1. S. W. HAAN, et al., "Update on NIF Indirect Drive Ignition Target Fabrication Specifications," *Fusion Sci. and Technol.*, **45**, 69 (2004).
2. J. J. SANCHEZ AND W. H. GIEDT, "Thin Films for Reducing Tamping Gas Convection Heat Transfer Effects in a National Ignition Facility Hohlraum," *Fusion Sci. and Technol.*, **44**, 811 (2003).
3. J. K. HOFFER and L. R. FOREMAN, "Uniform solid deuterium-tritium fuel layers resulting from radioactively induced sublimation," *J. Vac. Sci. Technol.*, **A 7**, 1161 (1988).
4. Part # 4/36 manganin HML Butyral Bond; www.mwswire.com
5. J. R. SCHOONOVER, W. P. STECKLE, Jr., N. ELLIOT, P. S. EBEBY, A. NOBILE, "Fourier Transform Infrared Spectroscopic Analysis of Plastic Capsule Materials Exposed to Deuterium-Tritium (DT) Gas," to be published in these proceedings.

TABLE 1

Wire Number	Resistance before	Resistance after
1R	>200 GΩ	10 GΩ@100V, 8.0 GΩ@250V 3/10/05
1G	>200 GΩ	10GΩ@250V, 6.6 GΩ@500V 3/10/05; 2.6 GΩ@250V 3/11/05
1N	>200 GΩ	9.6 GΩ@250V 3/10/05
1B	>200 GΩ	9.0 GΩ@100V, 8.1 GΩ@250V 3/10/05
2R	>200 GΩ	2.5 GΩ@250V
2G	>100 GΩ on 500V scale, arcing on 1000V	2.9 GΩ@250V
2N	>200 GΩ	2.6 GΩ@250V
2B	Wire was broken off	Wire was broken off
3R	>200 GΩ	2.4 GΩ@250V
3G	>200 GΩ	2.3 GΩ@250V
3N	>200 GΩ	1.7 GΩ@250V
3B	>200 GΩ	1.8 GΩ@250V
4R	>200 GΩ	1.7 GΩ@250V
4G	>200 GΩ	2.2 GΩ@250V
4N	>200 GΩ	2.3 GΩ@250V
4B	~10.8 GΩ on 500V scale	0 GΩ@50V, >9.99 MΩ ohmmeter setting
5R	>200 GΩ	1.9 GΩ@250V
5G	>200 GΩ	1.9 GΩ@250V
5N	>200 GΩ	1.9 GΩ@250V
5B	>200 GΩ	760 Ω ohmmeter setting
6R	>200 GΩ	2.2 GΩ@250V
6G	>200 GΩ	2.3 GΩ@250V
6N	>200 GΩ	1.8 GΩ@250V
6B	>200 GΩ	3.0 GΩ@250V
7R	>50 GΩ on 250V scale	2.1 GΩ@250V
7G	>100 GΩ on 500V scale, ~138MΩ arcing on 1000V scale	2.7 GΩ@250V
7N	1.6 GΩ on 250V scale	2.4 GΩ@250V
7B	>50 GΩ on 250V scale, ~32MΩ on 1000V scale	3.2 GΩ@250V
8R	>200 GΩ	3.6 GΩ@250V
8G	>50 GΩ on 250V scale, 22MΩ, arcing on 1000V scale	1.9 GΩ@250V
8N	>50 GΩ on 250V scale, 2.3MΩ, arcing on 1000V scale	3.0 GΩ@250V
8B	<11 GΩ on 100V scale, 225MΩ on 250V scale	2.1 GΩ@250V
9R		6.2 GΩ@250V 3/10/05
9G		15 GΩ@100V, 9.9 GΩ@250V 3/10/05
9N		6.0 GΩ@250V 3/10/05; 1.5 GΩ@250V 3/11/05
9B		12 GΩ@250V 3/10/05; 1.7 GΩ@250V 3/11/05
10R		2.3 GΩ@250V
10G		2.2 GΩ@250V
10N		3.5 GΩ@250V
10B		2.8 GΩ@250V

The post measurements for which a date is NOT noted were performed on March 11, 2005.

FIGURE CAPTIONS

Figure 1. Photograph of material exposure vessel.

Figure 2. Photo of sample holders for planar polymer samples.

Figure 3. Plot of wire insulation resistance change over several weeks after DT exposure.

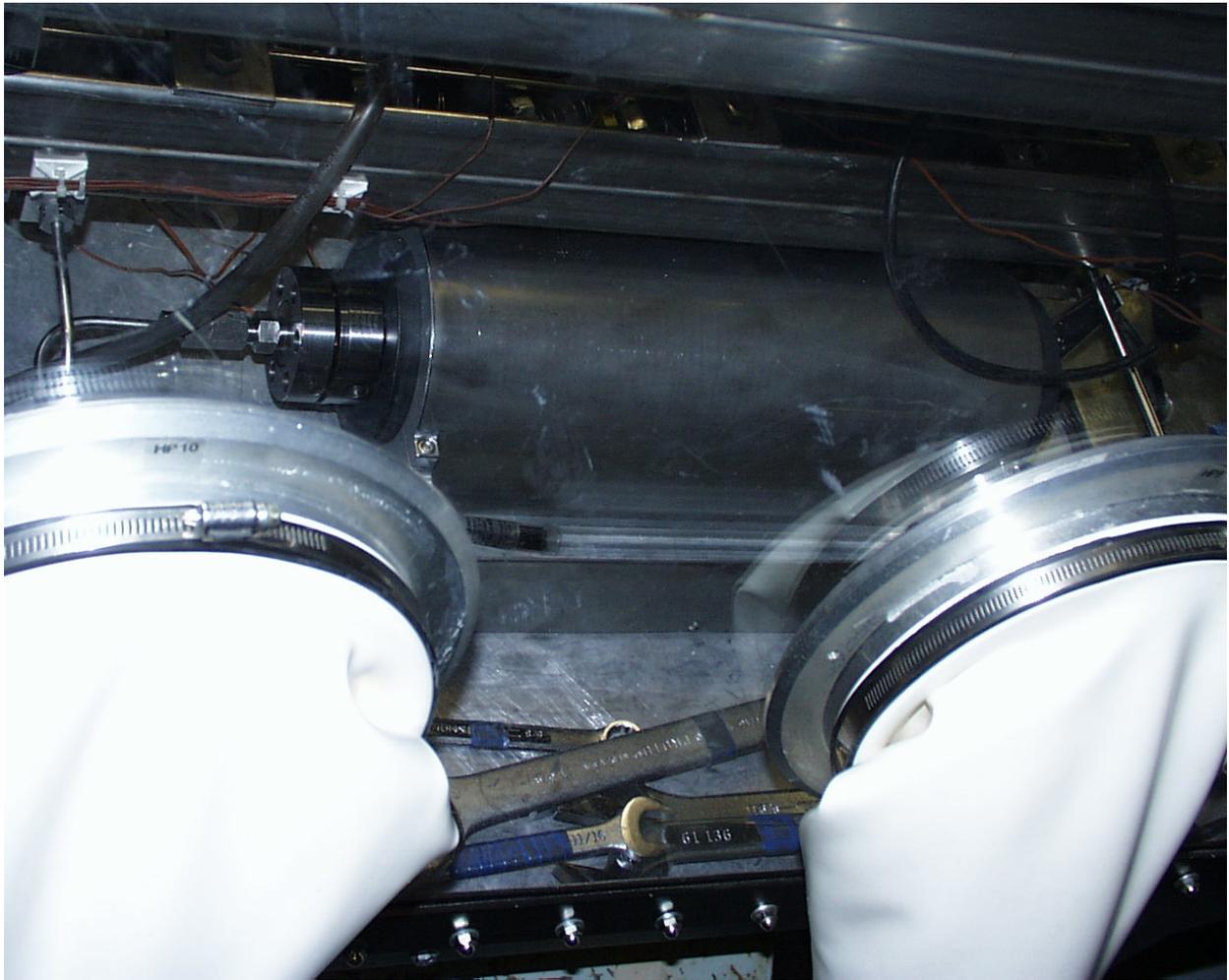


Figure 1



Figure 2

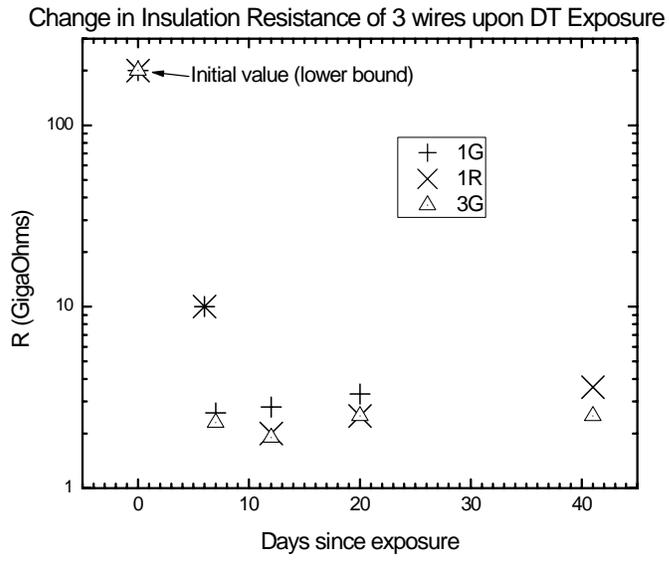


Figure 4