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ANALYSIS OF HAZARDS IN A FLAMMABLE GAS EXPERIMENT AND DEVELOPMENT OF A TESTING REGIME FOR A POLYPROPYLENE VACUUM WINDOW

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Abstract

Far Infrared Spectroscopy (Far-IR) is a bend magnet infrared beamline at the Canadian Light Source. The beamline utilizes a gas cell loaded with experimental gas which light is bounced through and a spectrometer to measure the absorption of the gas. For an experiment at Far-IR utilizing methane and nitrogen at 100 K temperatures, issues with icing and inconsistent absorption gradients were noted at the Polymethylpentene Rigid Plastic (TPX™) window separating the cell filled with the flammable gas mixture from the vacuum of the spectrometer. The possibility of replacing the existing windows with new 50-micron thick polypropylene window was investigated. Material properties were not available for polypropylene at the operating temperature of the experiment. Due to the hazardous nature of the gas being held back a hazard analysis was carried out to identify potential risks and mitigations for the change. Additionally, with material properties unavailable, a testing regime was established to ensure the polypropylene could survive in the experimental environment. The experiment was successfully completed. using the modified window assemblies.

INTRODUCTION

The Far-IR beamline uses several different Fourier Transform Infrared Spectroscopy (FTIR) methods during operation. When a FTIR method is to be utilized with a gas the beamline is equipped with gas cells of a known light path length. Some experiments require the use of flammable or potentially hazardous gasses. For safe handling of these gasses the beamline is equipped with a hazardous gas exhaust system; an explosion proof vane pump is used for post-experiment gas removal into the dedicated exhaust system. Far-IR's 2 m gas cell has a volume of 300 L and includes a cold nitrogen gas cooling system used to maintain cell contents at cryogenic temperatures when required. The 2 m cell is separated from a Bruker© spectrometer by a pair of windows; several window materials can be used. One such material is Polymethylpentene Rigid Plastic (TPX™). The windows separate the rough vacuum of the spectrometer from the cell's experimental conditions while simultaneously allowing synchrotron light to pass through. The 2 m cell and Bruker© spectrometer can be seen in Fig. 1.

In one instance, complications with the TPX windows at cryogenic temperatures interfered with the collection of quality data. A proposed modification of the windows to address these complications introduced uncertainty as to whether the modified windows could survive the experimental operating conditions.

Figure 1: Far-IR 2m gas cell and Bruker© spectrometer.

BACKGROUND

Two primary issues arose with the 6.6 mm thick TPX windows during an experiment. This experiment used the 2 m cell with a 1 atm methane mixture at 100 K. Ice would form in-vacuum on the spectrometer side interfering with transmission through the windows. TPX has a temperature-dependant absorption spectrum and as the methane moved within the cell, the temperature of the windows would fluctuate. This fluctuation produced inconsistent experimental conditions and unrepeatable results. Therefore, alternate window solutions were investigated. A requirement for a new window material was transparency in the visible range as a laser is used to align optics prior to an experiment. The FAR-IR beamline staff proposed 50 µm polypropylene windows as they had been observed to have better absorption spectra in previous published work [1].

OBJECTIVES

1. Design a new window assembly to eliminate icing and minimize absorption problems.
2. Complete a hazard analysis of risks introduced by window modification.
3. Test if proposed polypropylene windows can safely withstand experimental conditions.

FINAL DESIGN AND CONSIDERATIONS

The new window assemblies featured numerous modifications from the original TPX window to address the issues observed. To address ice formation, a two-window design with internal vacuum break was adopted. This design is pictured in Fig. 2. The added vacuum gap minimized icing by reducing heat transfer from the methane, maintaining the spectrometer side window at a higher and more stable temperature. Creating a smaller vacuum space reduced the

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amount of water available to freeze on the cell side window. A vacuum seal-off valve was added to the body of the assembly to allow this new vacuum space to be pumped down. A 3D printed cap was added to the valve to prevent movement in the event of window failure.

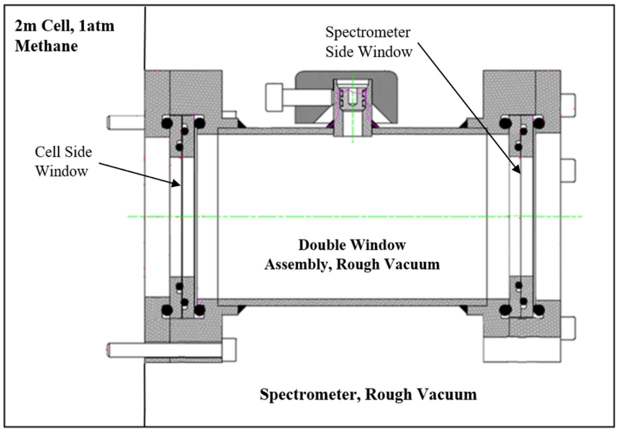

Figure 2: Double window assembly final design.

The variable absorption problem was addressed by switching the window material to polypropylene. Polypropylene has significantly lower absorption compared to TPX; any variations in the absorption due to temperature were negligible. The reduced thickness of the window had the added benefit of increasing the amount of light that reached the experimental sample. The new windows were clamped between two retaining plates. The retaining plates were designed to have the same thickness as the original TPX windows allowing for interchangeability during testing. O-rings around and within the retaining plates were used to form a vacuum seal in the double window assembly, and firmly secure components in place.

HAZARD ANALYSIS

The experiment utilized different mixtures of methane gas that would create a flammable mixture if mixed with air. Therefore, during the design process a hazard analysis was carried out to identify potential risks and their significance that would be introduced by the proposed modifications to the windows. The scope was limited to failure modes introduced through failure of the polypropylene windows.

Potential Failure Scenarios

Three scenarios were identified for gas release in the event of a window failure. The first scenario is window failure while the spectrometer scroll pumps were off. Experimental gas would be confined to the spectrometer and 2 m cell. The second scenario occurs if the spectrometer scroll pumps are left running, possibly due to human error. Experimental gas would be pumped from the spectrometer into the Far-IR hutch. The third scenario occurs if the spectrometer scroll pumps are left running and routed to vent into the beamlines hazardous gas system. Experimental gas would be pumped unregulated from the spectrometer into the hazardous gas system. This was a proposed modification to regular procedures to possibly eliminate the risk of scenario two. Typically, the spectrometer scroll pumps are not exhausted to the hazardous gas system.

Severity Analysis

The severity of the hazard for each potential scenario was analysed. For the analysis, a worst-case scenario of 100% methane in the 2 m cell was assumed. Severity was determined by investigating whether the scenario could possibly produce a flammable mixture and the potential damage ignition would cause.

In the first scenario the gas is contained to the 2 m cell and the spectrometer, eliminating the risk of the methane mixing with air. The mixture will remain 100% methane and be above the upper flammable limit [2]. Therefore, there is minimal hazard in the first scenario.

In the second scenario the methane would be released directly into the experimental hutch. If dispersed equally with the volume of air in the hutch, the 300 L of methane could form a mixture below the lower flammable limit [2]. However, as the methane mixes with the hutch air it could form a gas cloud capable of igniting. The result would be partial volume deflagration in the hutch. The potential damage from ignition was calculated using the methodology presented in *SFPE Handbook of Fire Protection Engineering 5th edition*. In this calculation the peak pressure of a fully stoichiometric mixture ignition is scaled by the mass of methane for the scenario in question over the mass of methane in a fully stoichiometric mixture. The peak pressure of 7.1 bar g for a methane ignition was taken from table 69.1 in *SFPE Handbook of Fire Protection Engineering* [3]. The mass of methane released from the 300 L 2 m cell was 0.5385 kg, significantly less than the 6.89 kg needed to fully mix with the 116310 L of the hutch.

$$P = (7.1 bar\ g)\frac{0.5385kg}{6.89kg} = 55.38kPa$$

The result was a scaled peak pressure of 55.38 kPa. As per table 69.2 in the *SFPE Handbook of Fire Protection Engineering,* this corresponds to a “Near complete destruction of houses” [3]. This represents a significant hazard to human life and equipment for the given scenario.

For the third scenario, where methane is vented into the hazardous gas system via the spectrometer scroll pumps, the pumping rate of methane relative to the system flow rate must be determined. The hazardous gas system has a capacity of 23.6 L/s. Hazardous exhaust systems are typically designed to maintain gas levels below 25% of the lower flammable limit [4]. To achieve this, methane would have to be vented at a rate lower than 0.295 L/s. The two spectrometer scroll pumps each vent at a rate of 8.3 L/s for a total rate of 16.6 L/s [5]. This is significantly higher than the allowable rate; therefore, an explosive mixture like the second scenario could form in the system.

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Mitigations

To minimize the risks related to a window failure, several mitigations for the identified hazards were developed. To prevent the occurrence of the second and third failure scenarios, the spectrometer scroll pumps must be verified to be off prior to and during the experiment. To mitigate the risk of a flammable mixture forming in the hazardous gas system the methane mixture must be diluted with nitrogen prior to removal from the cell and spectrometer. Finally, any pumping to remove methane shall be done by the 2 m cell's explosion proof vane pump.

MATERIAL TESTING REGIME

Due to the significance of the worst-case scenario in event of a failure, testing must be carried out on the polypropylene prior to use. Relevant material properties could not be found for polypropylene at the operating temperature of 100 K and to ensure the material would survive as a vacuum window, testing was required.

Failure Modes

Four failure modes for the new windows were highlighted as requiring testing. The first failure mode investigated is fatigue. The new windows would undergo two load cycles for each use. One cycle occurs as the window assemblies' internal vacuum space is pumped down, followed by unloading as the cell and spectrometer are pumped down. The second cycle occurs as the cell is loaded with the experimental gas. If the experiment is performed many times, it is reasonable to assume fatigue failure is a possibility. The second mode of failure is creep. This is a risk as the thin windows would be held under vacuum a load for the extent of the experiment. Each experimental measurement lasted several hours; however, the window would be held under load while the cell and methane were cooled in preparation. The total time the window would be held under vacuum is estimated at 24 hours. If the cell side window were to fail, the spectrometer side window would be subject to the third mode of failure; a sudden shock caused by the methane mixture rushing into the assembly's vacuum space. This would cause a sudden increase in pressure on the spectrometer side window. The final failure mode is a weakening of the window due to low experimental temperature.

Tests Developed

To address each mode of failure a test was developed to be carried out on the windows prior to use. To test fatigue, the assembly would be pumped down and then let up 15 times. Fifteen cycles were selected as it was significantly higher than the two cycles that a given window would experience and could still be completed in the time frame available before the experiment. To test creep, the assembly would be pumped down and held under vacuum for at least one week at room temperature. This value was selected as it was significantly longer than the 24 hours of the experiment while still being completable in the available time frame. To address the failure due to a shock, a test flange was created to hold vacuum behind one of the windows in the assembly. Failure would be manually induced on the opposite window. This test was carried out on both the TPX and polypropylene windows, as the TPX would be needed for the cold temperature testing. The potential failure at low temperature was addressed by completing an in-situ experiment using solely nitrogen in the 2 m cell and TPX as the spectrometer side window. TPX was used preventively as in the event of failure of the polypropylene window, TPX would be capable of surviving the experimental conditions.

Test Regime Construction

After identification of the required tests, a testing regime was developed. The fatigue and creep tests were carried out first as they represent the lowest stress situation; fatigue was tested first as it simulates a standard use case. Rupture tests were then carried out on the polypropylene and TPX windows, due to the requirement that they be capable of withstanding a shock during cold temperature in-situ testing. The windows were checked between each test for visual damage and pumped down to ensure they still held vacuum. Cold temperature was tested last as it exposed the window to nearly identical experimental conditions.

CONCLUSION

The hazard analysis identified that the worst-case scenario for failure represented a significant hazard. Mitigations were developed allowing for the experiment to proceed with the polypropylene windows. Material testing was needed due to the unknown properties of polypropylene and hazard significance. Material testing demonstrated that polypropylene windows could survive the operational conditions of the experiment. The new windows were used for the experiment after successfully passing testing.

Future Work/Limitations

The extent of the tests was limited to maintain a reasonable timeframe for completion. For example, the fatigue testing was limited to 15 cycles. This was deemed sufficient relative to the two cycles seen in the experiment. This necessitated the polypropylene windows be changed after each use. For reuse of the windows in the assembly a higher number of tests cycles would be needed. As a result of these limitations, the polypropylene window assembly was only approved for use in the experiment investigated.

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