Advanced Photon Source

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Pre-stressing and Atmospheric Pressure Testing of Large-Area Polymer Film X-Ray Windows

Section where used:

This procedure will be used by APS staff and resident APS beamline users in laboratory work areas located in buildings 432A, 400, and 401.

Changes made in this revision:

- Author changed from B. Stillwell to N. Bechtold

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Pre-stressing and Pressure Testing of Large-Area Polymer Film X-Ray Windows

1 INTRODUCTION

1.1 Purpose

Establish a process to control hazards for building, vacuum pressure testing, and *in-situ* studying of large area experimental polymer film x-ray windows.

1.2 Scope

This procedure addresses the potential hazards that may arise during the pre-stressing, vacuum pressure testing, and *in-situ* studying of polymer film x-ray windows with areas less than or equal to 144 in^2.

1.3 Applicability

This procedure applies to work done in laboratory work areas in buildings 400 and 401 by AES staff and APS resident users.

1.4 References

Pressure Systems Safety, LMS-PROC-313

Vacuum Systems Consensus Guideline for Department of Energy Accelerator Laboratories, Brookhaven National Laboratory, BNL-81715-2008-IR, https://icmsdocs.aps.anl.gov/docs/groups/anl/documents/guideline/aps_1273737.pdf

1.5 Type of Procedure

A general description of work activities and hazard controls is given. Because the intent of this work is to investigate a variety of experimental window designs differing in size, thickness, geometry, material, and assembly, a step-by-step procedure is not practicable. In lieu of a step-by-step procedure, a safety checklist that ensures controls are in place to address potential hazards will be completed prior to commencing experimental work.

1.6 Hazard Controls

Controls are implemented to limit the magnitude of sudden forces and releases of energy that may result from a polymer film window rupture, which is expected to occur routinely during testing. In addition, controls are implemented to prevent the allowed forces and releases of energy from presenting a personnel hazard. Hazard controls are described in detail in section 4.

Hazards were previously documented under Experiment Safety Assessment Form (ESAF) – Experiment ID 87081. This document is now expired but a copy is available in ICMS, APS_1442869.

2 BACKGROUND

The use of large-area x-ray detectors is common at the APS and many of the techniques that utilize these detectors require discrimination of small signal levels. To maximize the signal received at the detector, it is generally advantageous that the x-ray flight path to the detector be in vacuum. Most commercially-available detectors cannot be used in a vacuum environment, so large x-ray windows are typically used to terminate the vacuum volume immediately upstream of the detector. Often, these windows are made of polymer films such as Kapton or Mylar, which couple high strength and low x-ray absorption. For minimum absorption, such windows are generally made of the thinnest films possible. For some applications, the window may also be pre-stressed so that its flatness is better maintained under differential pressure. Clearly, an x-ray window optimized for minimum absorption is one that is stretched as much as possible whereby the tensile stresses during operation approach the failure strength of the material. However, in an uncontrolled environment, a sudden failure of a large vacuum window could present a personnel and equipment safety hazard.

3 PREPARATION OR PREREQUISITE ACTIONS

3.1 Argonne Work Planning and Control Requirements

Prior to start of work, all workers must ensure that ANL Work Planning and Control (WP&C) requirements have been met. At the time of this writing implementation of Argonne WP&C is described by APS procedure "Work Planning and Control at the APS." The WP&C process may indicate that additional training, procedures, and/or hazard controls are necessary.

3.2 Experimental Material Inspection and Tracking

Prior to pre-stressing and vacuum pressure testing, polymer films and experimental window assemblies shall be inspected for mechanical flaws that may lead to failure. These flaws may include but are not necessarily limited to: incomplete adhesion of the polymer film to the flange; sharp edges on the flange that may contact and compromise the polymer film window; adhesive on the window area of the polymer film; and holes, tears, creases, discoloration, or other mechanical inconsistencies in the polymer film. In addition, the history of the material will be assessed to ensure that it was made by a reputable manufacturer, that it has not been exposed to environmental conditions (moisture, solvents, sunlight) that could compromise material properties, and that it has not significantly aged. A material tracking document, included as Appendix B of this procedure, will be created to collect this information for each batch of material received.

Materials will be stored in labeled boxes that associate the material with the relevant tracking document. During testing, anomalous observations will be recording on the material tracking document. Prior to testing, the material tracking document will be checked to verify the history and expected quality of the material. Acceptance of a material for test will be at the discretion of the personnel conducting the test.

4 PROCEDURE

4.1 General Description of Work

Experimental window assemblies will consist of a sheet of thin polymer film attached by an adhesive or clamping device to a common commercial aluminum or stainless steel vacuum flange. The polymer film will cover a custom-machined aperture in the flange. Prior to attaching the film to the flange, the film may be stretched with a pre-tensioning device. During the stretching process, the tension of the film may be assessed by observing the resonant modes of the window. These modes may be excited by tapping the center of the window with a round mallet or by exposing the window to sound at a controlled frequency.

Assembled windows will be mounted to a vacuum chamber that is to be subsequently evacuated. The deflection of the window under vacuum will be assessed using a digital micrometer rigidly mounted on the air side of the window, which contacts the center of the window via a spring-loaded plunger. Optical techniques that do not require direct contact with the window may also be used.

It is to be expected that the polymer thin film used in these activities may rupture during the pre-stressing or vacuum pressure activities.

4.2 Controls

4.2.1 Control of Hazards that Could Result from the Force Imparted by a Sudden Differential Air Pressure of 15 psi

Rupture of a window enclosing an evacuated volume will result in a sudden differential air pressure of approximately 15 psi in the vicinity of the window. Objects in the vicinity of this differential air pressure will experience a force directed into the vacuum chamber that may be as great as 15 psi times the largest cross-sectional area of the object parallel to the plane of the window.

To prevent personal injury from body parts or other objects being drawn into the vacuum chamber by such a force, an exclusion zone will be established in the vicinity of a window under pressure within which personnel and loose items are disallowed. Any item inside the exclusion zone must be positively secured so as to remain fixed under the forces expected from a sudden differential air pressure of 15 psi. The exclusion zone will be defined by a sphere with a radius equal to the largest linear dimension defining the window aperture and centered on the center of the window aperture. A Huygens construction, shown in Figure 1, demonstrates that, at that distance, a pressure wave emanating from a complete and instantaneous window failure will be distributed over an approximately-ellipsoidal area, shadowed by the vacuum chamber, approximately six times the area of the window. The exclusion zone will be enforced by the placement of a shield, made of a thin polycarbonate sheet, around the experimental assembly.

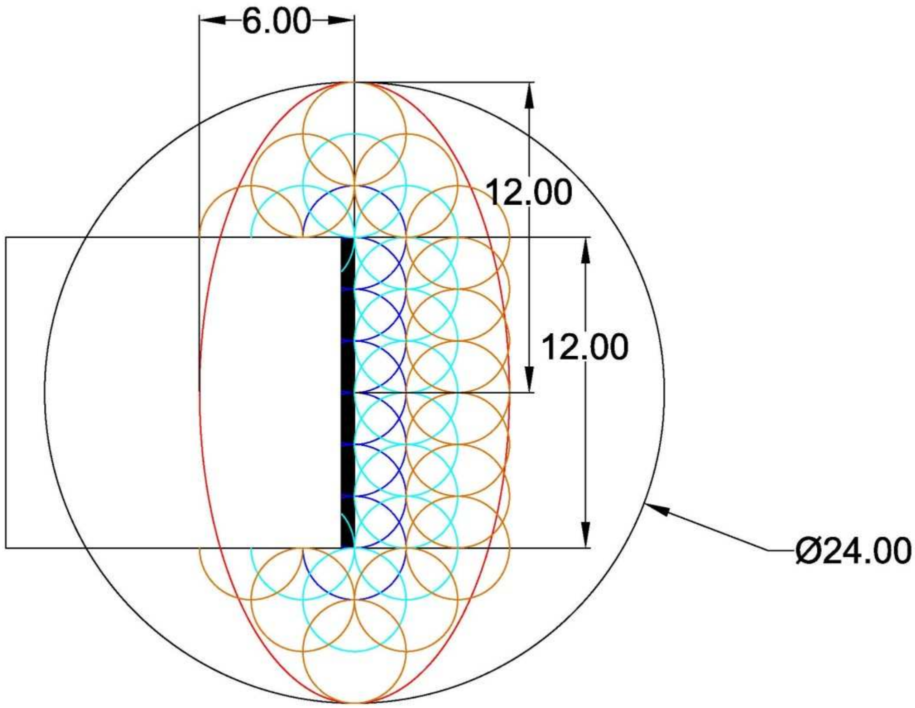

Figure 1: Huygens construction showing propagation of pressure disturbance resulting from a 12 inch-diameter window rupture.

In addition, the amount of energy that may be imparted to an object by such a rupture should be considered. This energy is proportional to the enclosed vacuum volume and determines the length of time over which a differential air pressure due to a window rupture will exist. To reasonably limit this energy, the evacuated volume shall be no greater than 500 in^3. To achieve this value, solid blocks of plastic will be placed inside the vacuum chamber. The energy associated with a vacuum volume of 500 in^3 is approximately 847 J.

A rupture of a window under vacuum pressure will also result in some movement of the vacuum chamber. Because of the large inertia of the vacuum chamber relative to air, this movement is expected to be small. However, the vacuum chamber will be rigidly mounted to the work table to minimize the extent and effect of this motion. Experimental window assemblies will also be vacuum pressure tested with the window oriented horizontally so that any movement of the chamber is directed vertically.

4.2.2 Control of the Ruptured Window Material Shrapnel Hazard

A rupture of a thin film polymer window may result in the ejection of pieces of that window. Because these pieces may present an eye hazard, all personnel in the room where pre-tensioning or vacuum pressure testing are taking place are required to wear safety glasses.

A polycarbonate shield, described in section 4.2.1, will be placed so as to intercept any shrapnel on a straight-line trajectory towards personnel during pressure testing. A graded approach will be used to determine if this shielding is also needed during pre-tensioning. Experience with Kapton suggests that it is highly unlikely to fragment during rupture. However, pre-tensioning shall initially be done with a polycarbonate shield in place. Upon discovery of the actual implications of window failure during pre-tensioning operations, personnel may opt to pretension windows without the polycarbonate barrier in place.

A similar graded approach will be used to determine if face shields are necessary during the pre-tensioning and pressure testing operations. Upon discovery of the actual implications of window failure during pre-tensioning and vacuum pressure testing, personnel may opt to conduct these activities without a face shield.

4.2.3 Control of the Acoustic Hazard

A rupture of a thin film polymer window will generate a loud noise. Because this noise could be damaging to human hearing, all personnel in the room where pre-tensioning or vacuum pressure testing is taking place are required to wear hearing protection, namely ear plugs or ear muffs.

Additionally, the noise resulting from a window rupture may startle personnel in adjoining areas. These areas will be conspicuously posted so as to make such personnel aware of the possibility of sudden, loud noises. If it is determined that the noise generated by a rupturing window is unacceptably loud in adjoining areas, the experiment will be relocated to a more appropriate location.

4.2.4 Control of the Experimental Area to Authorized Personnel

During pre-tensioning and initial vacuum pressure testing of the polymer film window, only personnel who are authorized by appropriate line management and are knowledgeable of this procedure will be permitted in the experimental area.

During vacuum pressure testing, the experiment may be left unattended once the window has demonstrated integrity under vacuum pressure. The enclosure will be conspicuously posted, "DANGER: EXPERIMENTAL VACUUM SYSTEM UNDER TEST. DO NOT MOVE OR OTHERWISE DISTURB THIS SET-UP. EAR PROTECTION MUST BE WORN WHILE WORKING IN THIS AREA."

4.2.5 Pre-Experiment Checklist

Prior to each pre-tensioning operation or vacuum pressure test, a checklist will be completed to ensure that all of the controls described in this document are in place. That checklist is included as Appendix A in this document.

5 DOCUMENTS/RECORDS CREATED BY THIS PROCEDURE

The documents/records listed below will be created in the execution of this procedure and must be retained as indicated.

Description of Document/Record (include ID number, if applicable)	Custodian	Storage Location and Medium	Retention Requirement
Completed Checklist for Pre-Stressing and Pressure Testing of Large-Area Polymer Film X-Ray Windows (Appendix A)	S. Weigand	432-A004 Paper	Until experiment completion.
Completed Experimental Polymer Window Material Tracking Sheet (Appendix B)	S. Weigand	432-A004 Paper	Until experiment completion.

6 TRAINING REQUIRED

ESH 195 – Personal Protective Equipment
ESH 174 – Noise and Hearing Conservation Training

7 FEEDBACK AND IMPROVEMENT

If you are using this procedure and have comments or suggested improvements for it, please go to the APS Policies and Procedures Comment Form* to submit your input to a Procedure Administrator. If you are reviewing this procedure in workflow, your input must be entered in the comment box when you approve or reject the procedure.

Instructions for execution-time modifications to a policy/procedure can be found in the following document: Field Modification of APS Policy/Procedure (APS_1408152).

* https://www.aps.anl.gov/Document-Central/APS-Policies-and-Procedures-Comment-Form

APPENDIX A

Checklist for Pre-stressing and Pressure Testing of Large-Area Polymer Film X-Ray Windows

Name ____

Date __________________

Pre-stressing

____ All personnel in the area are authorized and trained as per this procedure.

____ Material tracking document has been checked to verify material quality prior to testing.

____ Window and pre-tensioning apparatus has been inspected for defects and other material anomalies that may compromise safe and predictable operation.

____ Shrapnel shielding is in place (if applicable).

____ Personnel protective equipment is worn by all personnel in area: safety glasses and (if applicable) face shields.

Vacuum Pressure Testing

____ All personnel in the area are authorized and trained as per this procedure.

____ Material tracking document has been checked to verify material quality prior to testing.

____ Window assembly and vacuum chamber apparatus has been inspected for defects and other material anomalies that may compromise safe and predictable operation.

____ Shrapnel shielding is in place.

____ Noise hazard notifications are posted inside and outside the experiment area.

____ Pressure hazard notification is posted inside and at the entrances of the experimental area.

____ Personnel protective equipment is worn by all personnel in area: safety glasses, hearing protection, and (if applicable) face shields.

APPENDIX B

Experimental Polymer Window Material Tracking Sheet

Manufacturer:

Vendor:

Date Received:

Material Certifications (if included in shipment):

Commercial name and/or chemical composition of material:

Physical dimensions of material:

Storage / Handling Requirements:

Environmental Exposures (moisture, sunlight, radiation, chemical, temperature, etc):

Observations of anomalous material qualities (cuts, creases, voids, scratches, discoloration, etc):

Other comments: