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Qualification of Target Chamber Vacuum Systems Cleanliness using Sol-Gel Coatings

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SPECIFICATION

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Qualification of Target Chamber Vacuum System Cleanliness using Sol-gel Coatings NIF5010684_OA02012.doc Version 4.1 UCRL-TR-218169	**Technical Reviewer:** Charles Petty, Chuck Thorsness, Pete Biltoft, Bill Gourdin	**Date:** October 21, 2002
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	Approver: Irving F. Stowers	**Date:** October 21, 2002

1. SCOPE

1.1. This document defines the procedure necessary to qualify the airborne molecular cleanliness (AMC) of vacuum systems (enclosures or large components) that are placed within the National Ignition Facility (NIF) *target chamber* or are attached to it and communicate with it during vacuum operation.

1.2. This test is specific to the NIF *target chamber* because the allowable time dependent rate of rise in the pore filling of a sol-gel coated SAW sensor is based on some nominal change-out time for the disposable debris shields. These debris shields will be sol-gel coated and thus they represent a means of "pumping" AMCs from the target chamber.

The debris shield pumping rate sets the allowable change in pore filling with time specified in the test procedure.

1.3. This document describes a two-part procedure that provides both a static measurement of sol-gel pore filling at the end of a 48-hour test period and a dynamic record of pore-filling measured throughout the test period. Successful qualification of a vacuum system requires that both the static and dynamic measurements meet the criteria set forth in Section 7 of this document.

2. REFERENCE DOCUMENTS

The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue used shall be the one in effect on the date of request for quotation. Any conflict between this specification and the referenced document shall be brought to the attention of LLNL in writing for resolution before the seller takes any action.

2.1. NIF0081065 - *Practical Implementation of Surface Acoustic Wave Sensors for Monitoring of Airborne Molecular Contaminants: Electronic Components*, C. B. Thorsness, J. F. Kimmons, and P. E. Miller, April 15, 2002.

2.2. NIF0087129 – *Ellipsometer Measurements of Sol-gel Coated Silicon Wafers*.

2.3. NIF0087128 – *Cleaning Procedure for Sol-gel Coated Silicon Wafers*.

3. DEFINITIONS

3.1. ***Cleanroom Glove:***

- Allegiance[1] latex gloves {powder free}; **or**
- Allegiance nitrile [acrylonitrile] gloves {powder free}; **or**
- Safeskin[2] Hypoclean Critical Latex gloves HC335; **or**
- Safeskin Hypoclean Critical Nitrile gloves 61012.

3.2. ***Sol-gel Coated SAW Detector****:* a Surface Acoustic Wave (SAW) detector consisting of an electronics package (LEA99-165131[3]), a KF-40 flange mounted detector head, and a nominal 1ω (≈ 1,053-nm) anti-reflective (AR) ammonia-hardened sol-gel coated sensor chip.

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[1] Allegiance Healthcare Corporation, 1430 Waukegan Road, McGaw Park, IL 60085, 847-689-8410, 800-964-5227, http://www.allegiancehealth.com

[2] Safeskin Corporation, 12671 High Bluff Drive, San Diego, California, 92130, 800-462-9989, http://www.safeskin.com

[3] LEA99-165131 Schematic, Oscillator Board, SAW Amplifier.

3.3. ***Sol-gel Coated Silicon Wafer:*** a 100-mm diameter electronic grade polished silicon wafer[4] in a fluoroware carrier[5]. The wafer shall be coated on one side with a nominal 1ω (≈ 1053 nm) **OR** 2ω (≈ 526 nm) anti-reflective (AR) ammonia-hardened sol-gel coating. Wafers may be used after applying a fresh 1ω or 2ω sol-gel coating or reused after cleaning by following the wafer cleaning procedure described in NIF0087128.

3.4. Vacuum Pump:

3.4.1. ***Roughing Pump:*** Dry, oil-free roughing pump with the minimum ultimate pressure ≤ $1x10^{-2}$ Torr such as the DS Series of Dry Scroll Roughing Pump from Varian[6].

3.4.2. ***High Vacuum Pump:*** Dry, oil-free high-vacuum pump capable of producing an ultimate pressure ≤ $1x10^{-9}$ Torr. Examples of acceptable pump types include magnetically levitated turbomolecular pumps, such as the STP series from Seiko Seiki[7] or cryogenic pumps such as the Cryo-Torr series supplied by CTI Inc[8].

3.5. ***Visibly Clean:*** A surface free of oil, grease, cutting fluids, dust, soil, rust, and other foreign compounds.

3.6. ***Visibly Clean Verification:*** A surface verified as ***Visibly Clean*** when viewed without optical aid by a person of normal visual acuity, natural or corrected, from a distance between 2-5 feet, while surfaces are illuminated by glare-free light of at least 100 foot-candles intensity.

4. TEST SETUP

4.1. The vacuum system or vacuum component to be qualified shall be tested under environmental conditions similar to those in which the system or component will be utilized in the NIF. The vacuum system or vacuum component shall be evacuated utilizing its *inherent vacuum pumping* system or if this is not possible, enclosed within a larger pre-qualified vacuum system (previously shown to pass this qualification test when not containing the vacuum component under test). The larger pre-qualified vacuum system will need to have at least 2 ports for 1) the ***Sol-gel Coated SAW Detector*** head and 2) a pressure gage.

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[4] Typical supplier: Tygh Silicon Inc, 135 Lindbergh Avenue, Livermore CA 94550, 925-371-1223, 925-371-1666 fax, tyghsil@pacbell.net; typical price $6.00ea.

[5] Typical supplier: Entegris, 952-448-8181, 952-556-8022 fax, #H22-40-0615: 100 mm natural polypropylene tray, $1.18ea; #H22-401-0615: 100 mm natural polypropylene cover, $1.14ea; #H22-402-0615: 100 mm natural polypropylene spring, $0.50ea; #C30-0215: wafer tongs, $50.00ea.

[6] Varian Vacuum Technologies, 121 Hartwell Avenue, Lexington, MA 02421; 781-861-7200, 781-860-5437 fax.

[7] Seiko Seiki Representative Office, 1130 Ringwood Court, San Jose, CA 95131; 408-922-5932

[8] CTI-Cryogenics, Nine Hampshire Street, Mansfield, MA 02048, 508-337-5000.

4.2. The *inherit vacuum system* or *enclosing vacuum system* shall be capable of pumping the volume from atmospheric pressure to ≤ $1x10^{-4}$ Torr in a period of ≤ 12 hrs.

4.3. Vacuum testing shall be conducted at nominal room temperature, 20°C ±5°C.

4.4. The vacuum system or vacuum component shall be ***Visibly Clean*** before beginning the qualification test or at its design cleanliness level, (e.g., Level 100-A/3 or Level 300-A).

5. QUALIFYING PROCEDURE FOR STATIC MEASUREMENT OF PORE FILLING

5.1. Overview: Static measurements of pore filling are made using a pair of ***Sol-gel Coated Silicon Wafers***. The porosity, as measured by ellipsometer, of a pair of ***Solgel Coated Silicon Wafers*** are compared before and after exposure to the vacuum environment, under test, for a period of no less than 48 hours.

5.2. Measure the pore-filling fraction of two (2) ***Sol-gel Coated Silicon Wafers*** to be used in the qualification test using an ellipsometer. All ellipsometer measurements are to be made under a nitrogen purge and in accordance with the detailed instructions found in NIF0087129. Handle the ***Sol-gel Coated Silicon Wafers*** with wafer tongs[9].

5.3. Perform the qualification test by inserting the two (2) ***Sol-gel Coated Silicon Wafers*** into the vacuum system or vacuum component while wearing ***Cleanroom Gloves*** and handling the ***Sol-gel Coated Silicon Wafers*** with wafer tongs. If the vacuum system is large compared to the size of the silicon wafers, place the wafers at opposite ends of the system to ensure that there is only a small difference between the depositions of organic components onto the wafers at these extreme locations.

5.4. Begin the test cycle by starting the ***Roughing Pump*** and then the ***High Vacuum Pump.*** When the chamber reaches a pressure of ≤ $1x10^{-4}$ Torr, the 48-hour test cycle will commence. During pumping, the vacuum enclosure should reach a pressure of ≤ $1x10^{-4}$ Torr within 12 hrs of the start of the test. This pressure should be measured upstream of the vacuum limiting orifice. <u>If the vacuum system does not achieve a pressure ≤ $1x10^{-4}$ Torr within 12 hours, the vacuum enclosure is deemed to not meet NIF requirements.</u>

5.5. After 48 hrs, slowly vent the enclosure to atmosphere using dry, contaminant free gas such as the boil-off from a liquid nitrogen dewar.

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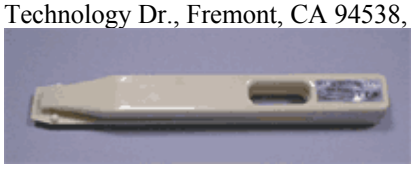
[9] Typical supplier: Model E100-100 made of PPS (Polyphenylene Sulfide) and distributed by NetMotion, 4160 Technology Dr., Fremont, CA 94538, 1-800-790-7837, www.netmotion.com



5.6. Immediately remove the ***Sol-gel Coated Silicon Wafers*** while wearing ***Cleanroom Gloves*** and handling the ***Sol-gel Coated Silicon Wafers*** with wafer tongs. Store each the ***Sol-gel Coated Silicon Wafers*** in their fluoroware carriers.

5.7. Measure the void volume of the two (2) ***Sol-gel Coated Silicon Wafers*** using an ellipsometer under nitrogen purge as described in NIF0087129. Average the two values and calculate the pore-filling fraction.

6. QUALYFING PROCEDURE FOR DYNAMIC MEASUREMENT OF PORE FILLING

6.1. Overview: dynamic measurements of pore filling are made by recording the resonant frequency of the ***Sol-gel Coated SAW Detector*** as a function of time. Prior calibration of the SAW package establishes the relationship between the resonant frequency of the sensor and the fractional pore filling of the sol-gel coating.

6.2. Calibrate the ***Sol-gel Coated SAW Detector*** using procedures described in NIF0081065.

6.3. Install the ***Sol-gel Coated SAW Detector*** head, using an aluminum seal or a pre-baked Viton seal onto a KF-40 vacuum port onto the vacuum enclosure. This vacuum port shall be located upstream of the limiting orifice between the vacuum enclosure and the vacuum pump. Further, any tubing associated with this vacuum port shall be of a large diameter (> 1 inch) and of a minimum length (< 3 inch).

6.4. Begin the vacuum test cycle by starting the ***Roughing Pump*** and then the ***High Vacuum Pump.*** When the chamber reaches a pressure of $\leq 1x10^{-4}$ Torr, record the time of day to acknowledge that the 48-hour test cycle has begun. This pressure should be measured upstream of the vacuum limiting orifice. <u>If the vacuum system does not achieve a pressure $\leq 1x10^{-4}$ Torr within 12 hours, the vacuum enclosure is deemed to not meet NIF requirements.</u>

6.5. Continuously record the signal from the ***Sol-gel Coated SAW Detector***.

6.6. After 48 hrs, record the time of day and slowly dry-gas vent the enclosure to atmosphere.

6.7. Archive the electronic record of the ***Sol-gel Coated SAW Detector*** and create an electronic file containing a graph of the pore-filling fraction versus time.

6.8. NOTE: A 20% pore filling is equivalent to a 0.1-0.15% transmission loss at 3-omega (351 nm).

7. QUALIFICATION REQUIREMENTS

7.1. **If** the average pore filling of the ***Sol-gel Coated Silicon Wafers*** is found to be ≤ 20%, **AND**

7.2. **If** the pore filling indicated by the ***Sol-gel Coated SAW Detector*** is ≤ 20% **AND**

7.3. **If** the *rate of change* of the pore filling as measured by the ***Sol-gel Coated SAW Detector*** is ≤ +0.5% / 12 hours as measured during the 12 hours immediately *prior* to venting the vessel, **AND**

7.4. **If** the vacuum system achieved a pressure of ≤ $1x10^{-4}$ Torr during the first 12 hours of the test cycle, **THEN**

7.5. The target chamber vacuum component is ***Vacuum Qualified*** for use on NIF.

8. TEST DOCUMENTATION

8.1. Records of qualified vacuum systems or vacuum components shall be submitted to the:

- Applicable LRU/Component Group Leader with the following information for incorporation into his/her permanent records concerning the LRU/Component and to the
- NIF Non-Optical Materials Group Leader (currently Bill Gourdin) with the following information for incorporation into the NIF0073131 Materials Testing Database:

8.1.1. A brief technical description of the vacuum system or vacuum component.

8.1.2. Average pore filling of ***Sol-gel Coated Silicon Wafers*** at the completion of the test.

8.1.3. Rate of change of pore filling as measured during the last 12 hours of the 48-hour test cycle as measured by the ***Sol-gel Coated SAW Detector***.

8.1.4. Electronic file containing a graph of the pore filling as a function of time as measured by the SAW sensor.

Revision History							
Rev	**Description**	**Author**	**Date**	**Reviewed By**	**Date**	**Approved By**	**Date**
OA	Initial Release	Irv Stowers John Ertel Phil Miller	10/21/02	Charles Petty, Chuck Thorsness, Peter Biltoft, Bill Gourdin, George Hampton, Jim Pryatel	10/21/02	Irving F. Stowers	10/21/02