REPORT DOCUMENTATION PAGE

Form Approved
OMB NO. 0704-0188

1. AGENCY USE ONLY (Leave Blank)	2. REPORT DATE January 28, 2002	3. REPORT TYPE AND DATES COVERED Final Progress Report 7/1/01-12/31/01

4. TITLE AND SUBTITLE
Determination of SiO2 Mechanical Behavior Via High Temperature Microtensile Testing

5. FUNDING NUMBERS
G DAAD19-01-1-0662

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8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

U. S. Army Research Office
P.O. Box 12211
Research Triangle Park, NC 27709-2211

10. SPONSORING / MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES
The views, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation.

12 a. DISTRIBUTION / AVAILABILITY STATEMENT

Approved for public release; distribution unlimited.

12 b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

The goal of this STIR project was to develop and demonstrate the capability to carry out the first direct determination of the mechanical properties of SiO_2 thin films at typical oxidation temperatures and stresses. There is little doubt that time-dependent inelastic deformation plays a significant role in SiO_2 films. There are various ways of obtaining mechanical properties data, but the most direct and the least ambiguous in interpretation is testing under uniaxial loading. Unfortunately testing specimens of bulk vitreous SiO_2 is not sufficient because this material is believed to have mechanical properties that differ from those of thermally grown SiO_2. Our approach was to conduct tensile tests on microscopic specimens of thermally grown SiO_2 from which the silicon substrate has been chemically removed. The four stages of the project were to fabricate thin SiO_2 specimens, to test the specimens at room temperature, to demonstrate a high temperature furnace compatible with the tensile tester, and to acquire a demonstration measurement of the high temperature mechanical behavior of SiO_2. The first three stages of the project were successfully completed. The fourth stage, demonstration of high temperature measurement, was attempted but could not be completed within the time frame of the project.

14. SUBJECT TERMS
SiO2, thin film, high temperature, tensile test

15. NUMBER OF PAGES
10

16. PRICE CODE

17. SECURITY CLASSIFICATION OR REPORT	18. SECURITY CLASSIFICATION ON THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	**UNCLASSIFIED**	**UNCLASSIFIED**	**UL**

NSN 7540-01-280-5500

Standard Form 298 (Rev.2-89)
Prescribed by ANSI Std. 239-18
298-102

List of Illustrations

Statement of Problem

Silicon oxidation is the most important single process in semiconductor technology and hence, one could argue, the most important industrial process in the world. It is widely used to isolate adjacent active devices in *VLSI* and *ULSI* structures by forming so-called field oxides between devices. More importantly, it is used to form the gate regions in transistor devices, the so-called gate oxides. Because the oxidation of an atom of silicon to form a molecule of SiO_2 involves a substantial and highly nonuniform expansion in volume, very large mechanical stresses, on the order of 500 MPa, are induced in oxide regions. These stresses are widely thought to play a very important, and often deleterious, role in various aspects of the oxidation process.

The goal of this STIR project was to develop and demonstrate the capability to carry out the first direct determination of the mechanical properties of SiO_2 thin films at typical oxidation temperatures and stresses. From the known properties of bulk vitreous silica, there is little doubt that time-dependent inelastic deformation plays a significant role in SiO_2 films. There are various ways of obtaining mechanical properties data, but the most direct and the least subject to ambiguities in interpretation is testing under uniaxial stress. Unfortunately testing standard tensile specimens of bulk vitreous SiO_2 is an unattractive prospect, because this material is believed to have mechanical properties that differ from those of thermally grown SiO_2.

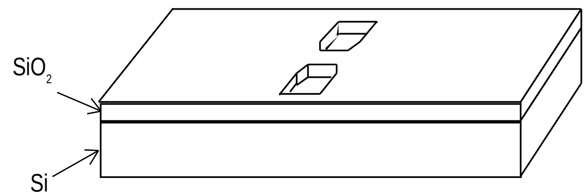
Our approach was to conduct tensile tests on microscopic specimens of thermally grown SiO_2 film from which the silicon substrate has been chemically removed. The four stages of the project were to fabricate thin SiO_2 specimens, to test the specimens at room temperature in a custom-built microtensile system, to demonstrate a high temperature furnace compatible with the tensile tester, and to acquire a demonstration measurement of the high temperature mechanical behavior of SiO_2. The first three stages of the project were successfully completed. The fourth stage, demonstration of high temperature measurement, in ongoing.

Summary of Results

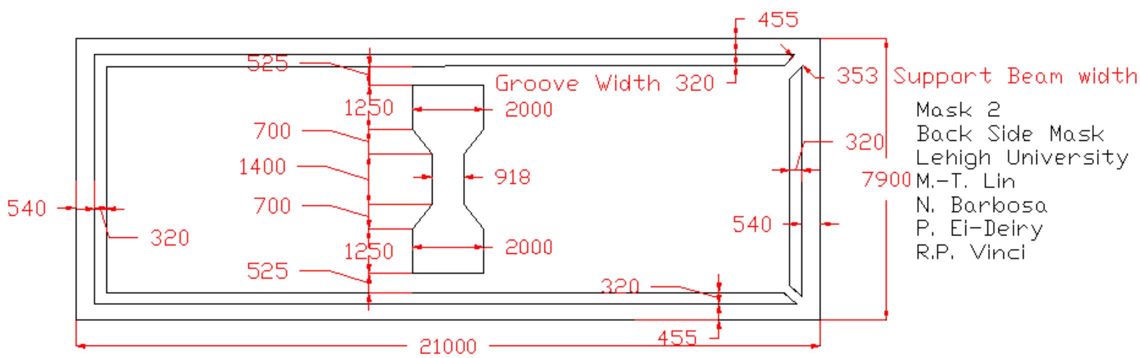
<u>Stage 1: Sample Fabrication</u>

The sample design is shown in Figures 1a and 1b. Eighteen individual specimens are fabricated on each 3-inch wafer. Three different process methods were attempted. The first and second processes, based on KOH and ion plasma etching, had reproducibility problems due to the low selectivity between SiO_2 and silicon. In particular, under-etching of the silicon left poor conditions at the ends of the beams and residual silicon on the backside. Examples of the specimens formed by these processes are shown in Figures 2 and 3.

Ultimately, the dry-etch methods were replaced by a TMAH-based wet-etch technique, necessitating a new backside mask design. This approach proved to be efficient and cost effective, producing high quality beams. The beams that resulted are represented in Figures 4 and 5. Typical yield on a single wafer was approximately six beams out of eighteen due to failures during final rinse.



a.



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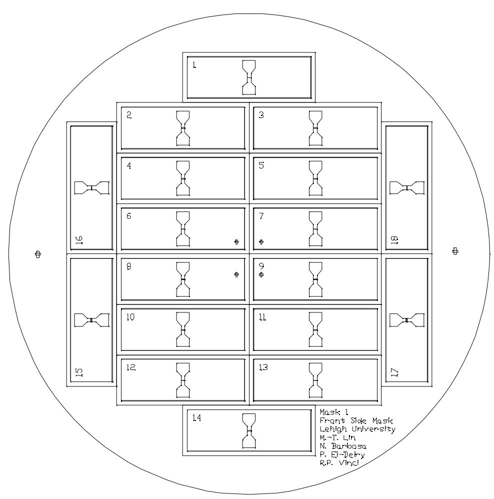


c.

Figure 1. (a) Individual die schematic diagram (not to scale), (b) Backside mask dimensions, (c) Die layout on 3 inch wafer.

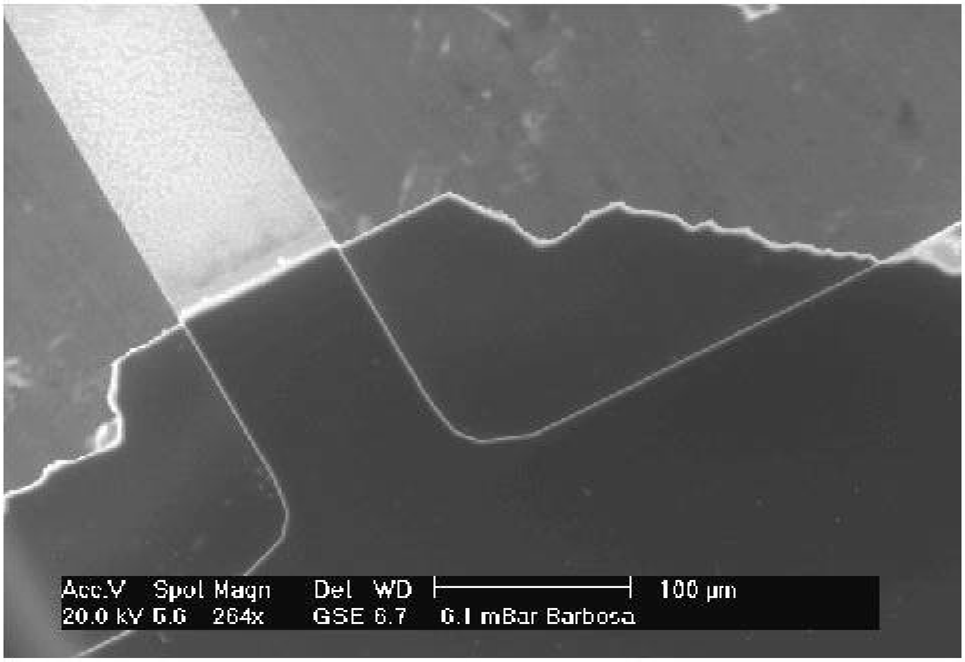


Figure 2. Close-up view of one end of a free-standing oxide beam fabricated by KOH and plasma etching. Under-etching of the substrate is visible. Image produced by Environmental Scanning Electron Microscopy (ESEM).

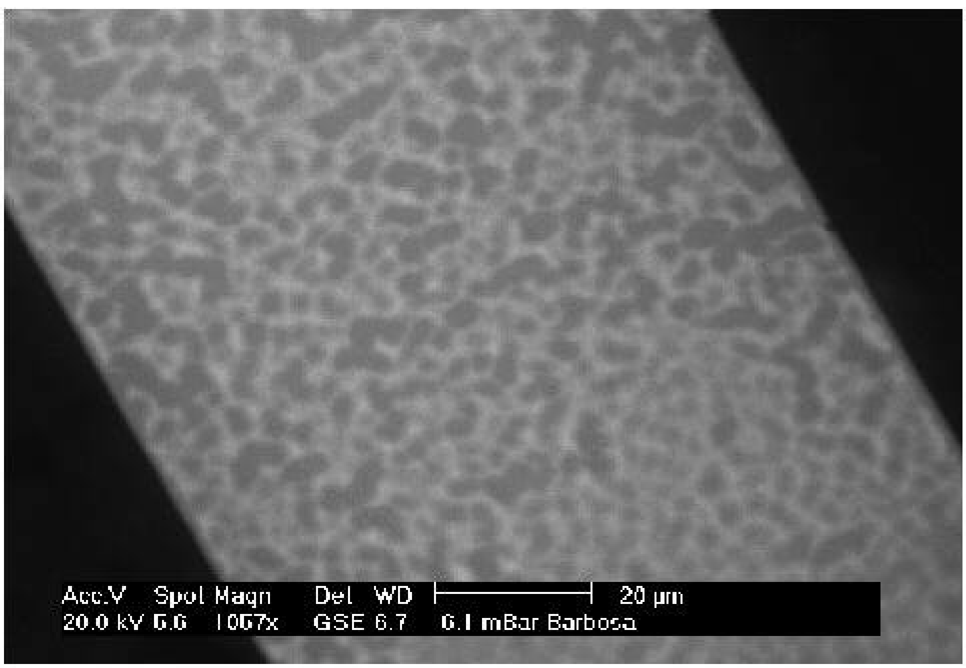


Figure 3. Close-up view of the underside of a free-standing oxide beam fabricated by KOH and plasma etching. Incomplete silicon removal is visible. Image produced by ESEM.

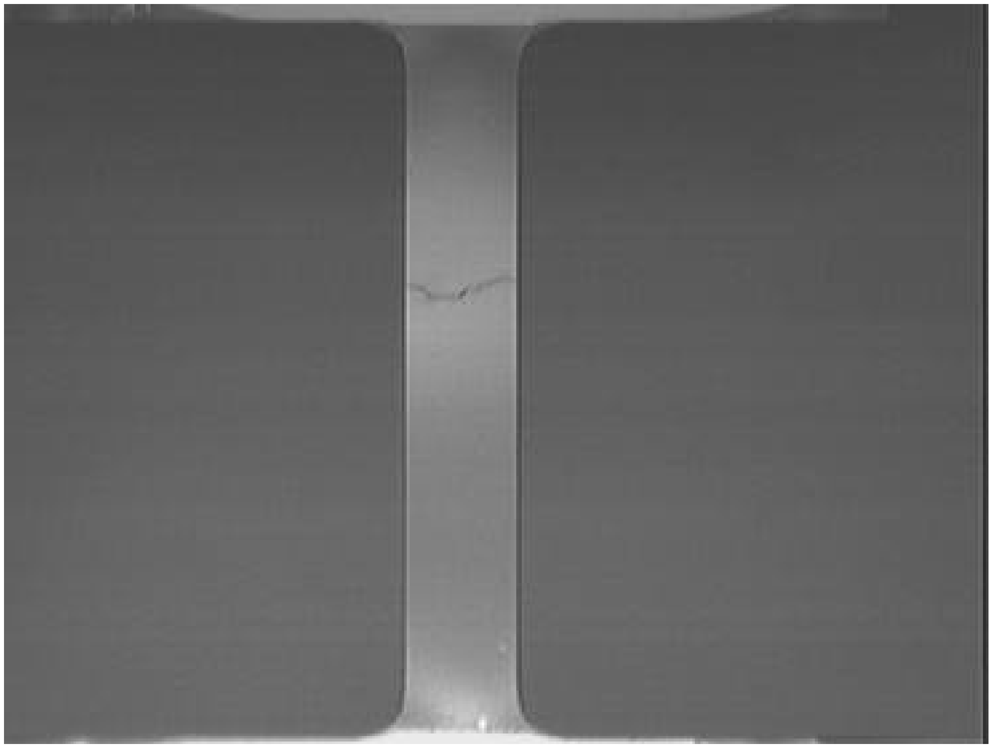


Figure 4. Top view of free-standing oxide beam fabricated by TMAH etching (crack produced during processing). Image produced by Light Optical Microscopy (LOM).

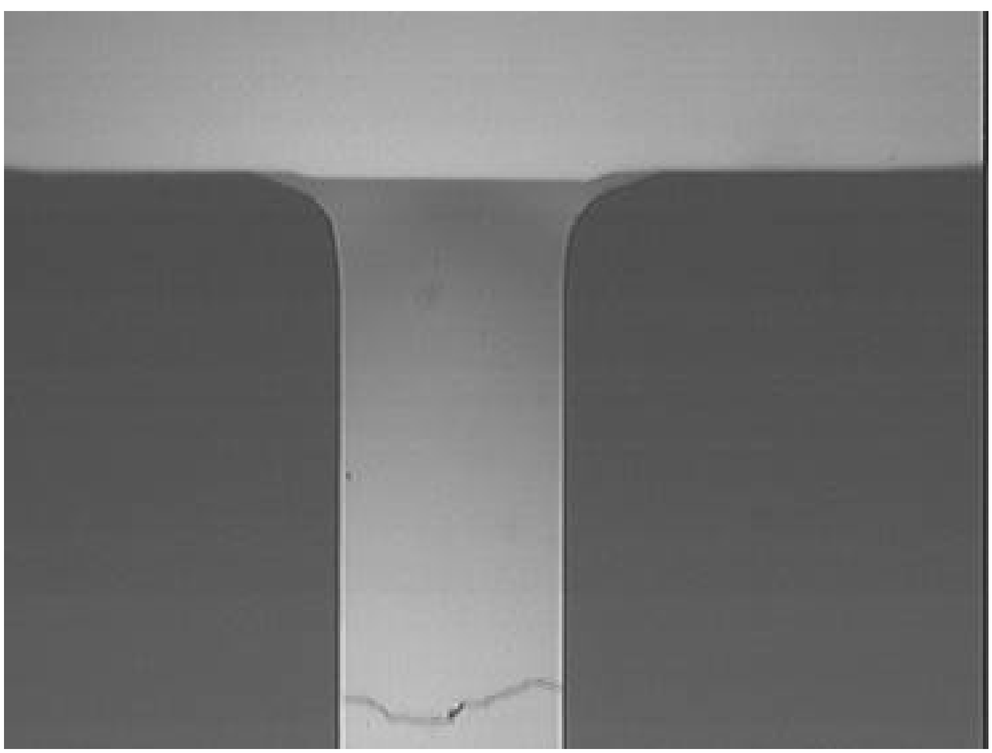


Figure 5. Close-up view of one end of a free-standing oxide beam fabricated by TMAH etching (crack produced during processing). Image produced by LOM.

Stage 2: Room Temperature Testing

Room temperature testing was carried out in a custom-built microtensile system. The results from one such test are shown in Figure 6. The stress-strain curve shows elastic behavior followed by brittle fracture at approximately 500 MPa. Excellent linearity was observed in the elastic region, demonstrating the quality of the specimen and the effectiveness of the test instrument for this purpose. The slope of the elastic portion of the curve yielded an average elastic modulus of approximately 64 GPa. This compares well with the expected range of 70-71 GPa for bulk SiO_2. The discrepancy between the expected and measured values may be due to compliance in the load train or may be related to imperfections in the thin oxide.

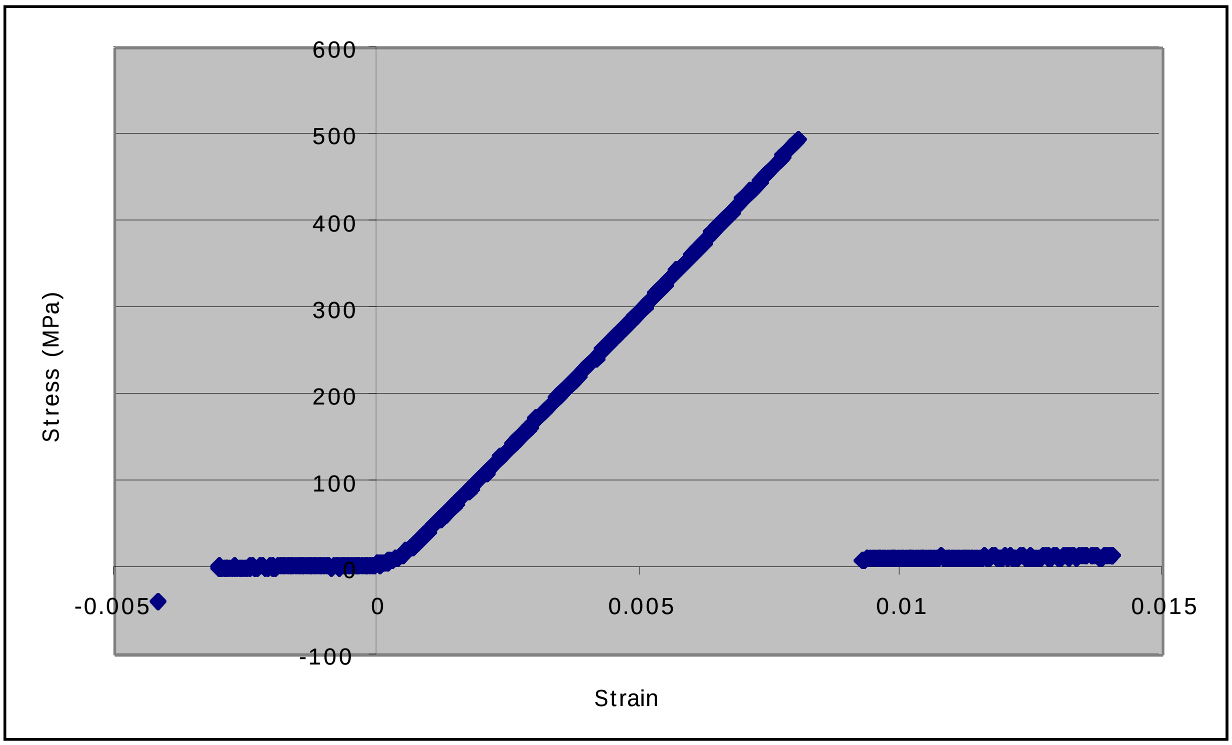


Figure 6. Stress-Strain data from a room temperature microtensile test of a free-standing oxide beam fabricated by TMAH etching. Excellent linear-elastic behavior is observed.

Stage 3: Furnace Demonstration

A high temperature furnace and corresponding pull-rod extensions were designed and fabricated. The system was integrated with the microtensile apparatus after the room temperature testing was complete. The complete system can be seen in Figure 7. The furnace is heated using a current source controlled by a solid state relay and LabVIEWâ code for closed loop feedback. Temperature is measured with a standard thermocouple. The furnace was tested to 800 °C; heating took approximately 5 minutes to reach the target temperature. Minimal overshoot was observed. After a brief period of settling the temperature was controlled to within a range of approximately 1 °C.

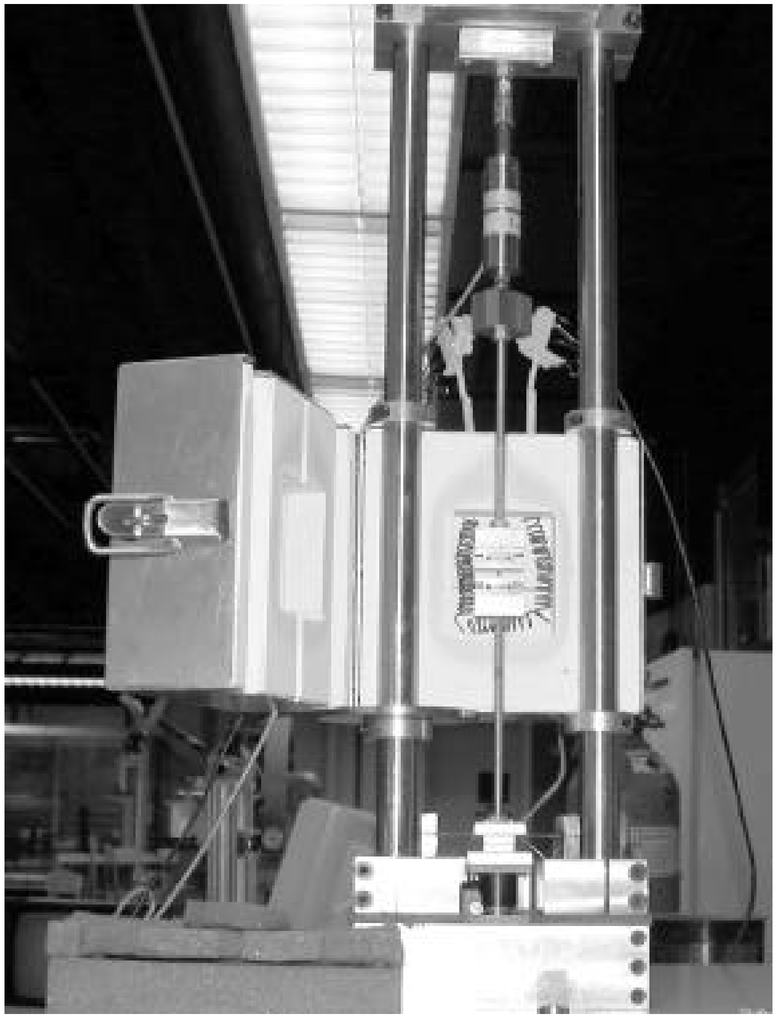


Figure 7. High temperature furnace showing heating coils (center) surrounding pull-rods and grips. A piezoelectric actuator is above the furnace, a capacitance-based load beam below.

Stage 4: High Temperature Testing

Following the room temperature tests only five specimens remained. High temperature tests were attempted but were unsuccessful due to fracture during specimen loading. Alignment of the pull-rod system is more difficult than that of the room temperature system. Furthermore, the highly compliant load beam allows too much vibration when a long pull-rod is attached. These factors greatly increase the likelihood of premature specimen failure.

A stiffer load beam has been designed but has not been fabricated. Stiffer pull-rods and grips have been acquired. Additional specimens are nearing completion. We will make another attempt at high temperature testing before the end of February, after the formal completion date of this project. If successful, as we anticipate, a publishable manuscript will be written and supplied to the ARO.

Technical Reports

No technical reports (other than this final progress report) or peer-reviewed manuscripts have resulted from this work.

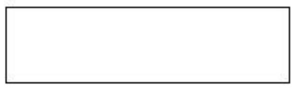
Participating Personnel

Three Co-PI's (Vinci, Delph and Jaccodine) participated in this project but did not receive support. One Ph.D. student, Ming-Tzer Lin, was supported by this grant for the duration of the project. Two other Ph.D. students, Paul El-Deiry and Nicholas Barbosa, were partially supported for a portion of the project. No advanced degrees were awarded.

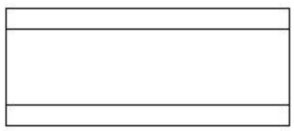
Inventions

No inventions were made.

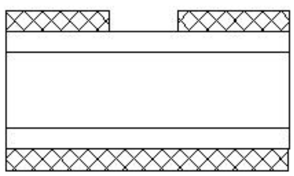
Appendix: Flow diagram for the TMAH fabrication process.



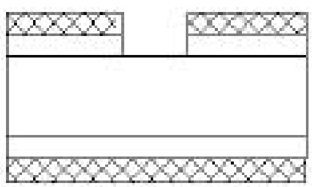
1. Start with 3 in 10 mills (250µm) <100> orientation wafer double side polished.



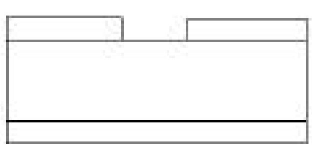
2. Steam Oxide 1100C 2 hr 30 mins for 1µm SiO2 layer on top and back side of the wafer



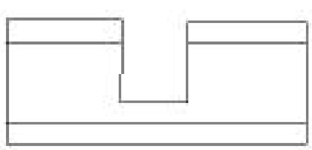
3. Bake photoresist on both sides and use backside mask pattern photoresist on top of Si02 layer by using UV exposure.



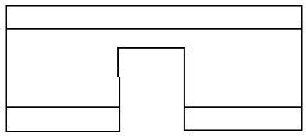
4. Buffer HF (10:1) to etch away SiO2 window for 20min 450A/min



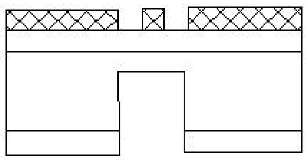
5. Strip away the photoresist on both sides of the wafer



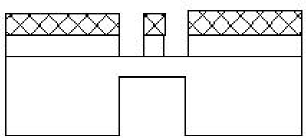
6. TMAH etching (5 % wet TMAH + 1.4 % wet dissolved Si + 0.5 % wet (NH4)2S2O8) 85C etch away 9.2 mill silicon substrate and left 0.8 mill (0.9 um/mins)



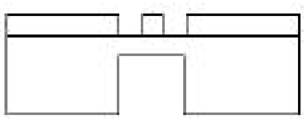
7. Turn over to the other side of the wafer



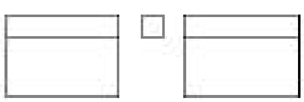
8. Photolithography exposure Pattern SiO_2 with front side mask by using infrared alignment



9. Buffer HF unprotect SiO_2 for 20 mins till hydrophobic on the other side



10. Strip away photoresist



11. TMAH etching (5 % wet TMAH + 1.4 % wet dissolved Si + 0.5 % wet $(NH_4)_2S_2O_8$) 85C etch away left silicon substrate. (0.9 um/mins)