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Mechanical Properties of Microelectronics Thin Films: Silicon DiOxide (SiO_2)

Fariborz Maseeh, Sean M. Gelston, and Stephen D. Senturia

Abstract

Mechanical design of microfabricated devices requires knowledge of mechanical material properties. Thin film material properties are sensitively process dependent, and should therefore be organized accordingly. A relational database of material properties is under development as part of a general micro-electro-mechanical CAD environment. A computerized literature search through the published values for Silicon DiOxide (SiO_2) properties under various processing conditions resulted in the following document.

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Microsystems
Technology
Laboratories

Massachusetts
Institute
of Technology

Cambridge
Massachusetts
02139

Room 39-321
Telephone
(617) 253-0292

90 00 21 053

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Author Information

Maseeh and Gelston: Department of Electrical Engineering & Computer Science, Room 39-665, MIT, Cambridge, MA 02139. (617) 253-0725.

Senturia: Department of Electrical Engineering & Computer Science, Room 39-665, MIT, Cambridge, MA 02139. (617) 253-6869.

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PROPERTIES OF MICROELECTRONIC SILICON DIOXIDE (SiO₂)

FARIBORZ MASEEH, SEAN M. GELSTON, STEPHEN D. SENTURIA

MICROSYSTEMS TECHNOLOGY LABORATORIES
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
DEPARTMENT OF ELECTRICAL ENGINEERING & COMPUTER SCIENCE
CAMBRIDGE, MA, USA

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Introduction

There is a growing need for the ability to perform mechanical analysis of microelectronic devices, both in assuring structural reliability against failure of thin film layers, and in evaluating the effects of various external loads including temperature and humidity effects. In addition, with the development of increasingly sophisticated micromechanical devices, including microsensors, pumps, valves, and micromotors, and with the increasing performance demands being placed on these devices, notably in the precision and accuracy of microsensors, there is a critical need for computer-aided-design (CAD) tools which will permit rational design of these devices. The present program is directed towards creation of a suitable CAD environment for micromechanical analysis of microfabricated deformable structures utilized for measuring the mechanical properties of thin films, and static analysis of which can be utilized for reliability investigations.

There are two fundamental problems that confront the designer [*,**]: (1) the need to construct a three- dimensional solid model from a description of the mask set and process sequence to be used in fabrication of a micromechanical device; and (2) the need to be able to predict the mechanical properties of each of the constituent materials in a device, including possible process dependences of these properties. With such a 3-D model in hand, with appropriate properties for each material, prediction of mechanical behavior could be done with existing finite-element modeling (FEM) programs. However, at the present time, there is no CAD system, either mechanical or microelectronic, which successfully addresses these problems in a coherent way. Koppelman [***] has developed a program called OYSTER which permits construction of a 3-D polyhedral-based solid model from a mask set and primitive process description, but as yet, there is no provision for linking to FEM tools or to standardly used layout and process modeling tools, and no database for prediction of mechanical properties from the process sequence.

An architecture for a micro-electro-mechanical CAD system in which these two critical problem areas can be the focus of simultaneous and parallel development work is presented in Fig. 1. The basic idea is to provide three different levels of user interaction: (1) at the conventional microelectronic level, with access to mask layout and process specification; (2) at the mechanical CAD level, for direct construction of 3-D solid models which can then be analyzed with FEM; and (3) at the mechanical-property database level, for entry of mechanical property data as it is acquired and documented. There are then two specific development tasks: (1) development of a 3-D solid modeling tool, which we call the "structure simulator", and which takes mask layout data and a realistic process description and builds a 3-D solid model in a format compatible with the mechanical CAD system (an extension of what OYSTER now does); and (2) the development of a mechanical property database using iterative measurements on deformable micromechanical structures (such as diaphragms, beams, and resonant structures) together with careful FEM studies of the dependence of their behavior on mechanical properties.

We have implemented this architecture in a Sun 4 host, drawing on existing codes wherever possible. The primary interface for mechanical modeling is through PATRAN, a mechanical CAD package which provides for manual construction of 3-D solid models, graphical display, and interfacing with FEM packages (we are using ABAQUS). The 3-D solid model resides in the PATRAN Neutral File, and we have elected to use the material-property format of the Neutral File as a first version of the Mechanical Property Database. Layout is provided through KIC, and process description through the process-flow representation (PFR) is created with a standard text editor. SUPREM III and SAMPLE are installed to provide depth and cross-sectional modeling capabilities. The structure simulator (under development) will accept KIC and PFR files as input, draw on SUPREM

III and SAMPLE as needed, and will output a 3-D solid model in the format of the PATRAN Neutral File. PATRAN will then be able to pick up the model, provide for FEM analysis and graphical display of behavior. The present status is that all of the commercially available codes (solid boxes in Fig. 1) are installed and operating. The first entries into the Mechanical Property Database have been made for silicon dioxide and silicon nitride as a result of the literature review enclosed.

This document is the result of a computerized literature search (done at MIT CLSS) to locate published mechanical property data for silicon dioxide, SiO₂. Investigating some 120+ references, a group of 45 was selected and the mechanical properties of SiO₂ were extracted under both thermal growth and chemical vapor deposition (CVD). The cited values were arranged by different mechanical property headings, and then by the deposition methods as subheadings. The boldface values indicate results of experimental measurements (from references), and the italic values correspond to when a reference cites results from other references without measurements, or when no reference experiment was indicated to support the cited values. Most values were traced to their original measurement (experiment) when possible. Averages of the cited properties have been entered in our mechanical property database.

References

- *. S. D. Senturia, "Microfabricated structures for the measurement of mechanical properties and adhesion of thin films", Transducers '87, Tokyo, 1987, pp. 11-16.
- ** S. D. Senturia, "Can we design microrobotic devices without knowing the mechanical properties of materials?", IEEE MicroRobots and Teleoperators Workshop, Hyannis, 1987.
- ***. G. Koppelman, "OYSTER: a 3D structural simulator for microelectromechanical design," MEMS '89, Salt Lake City, 1989, pp. 88-93.

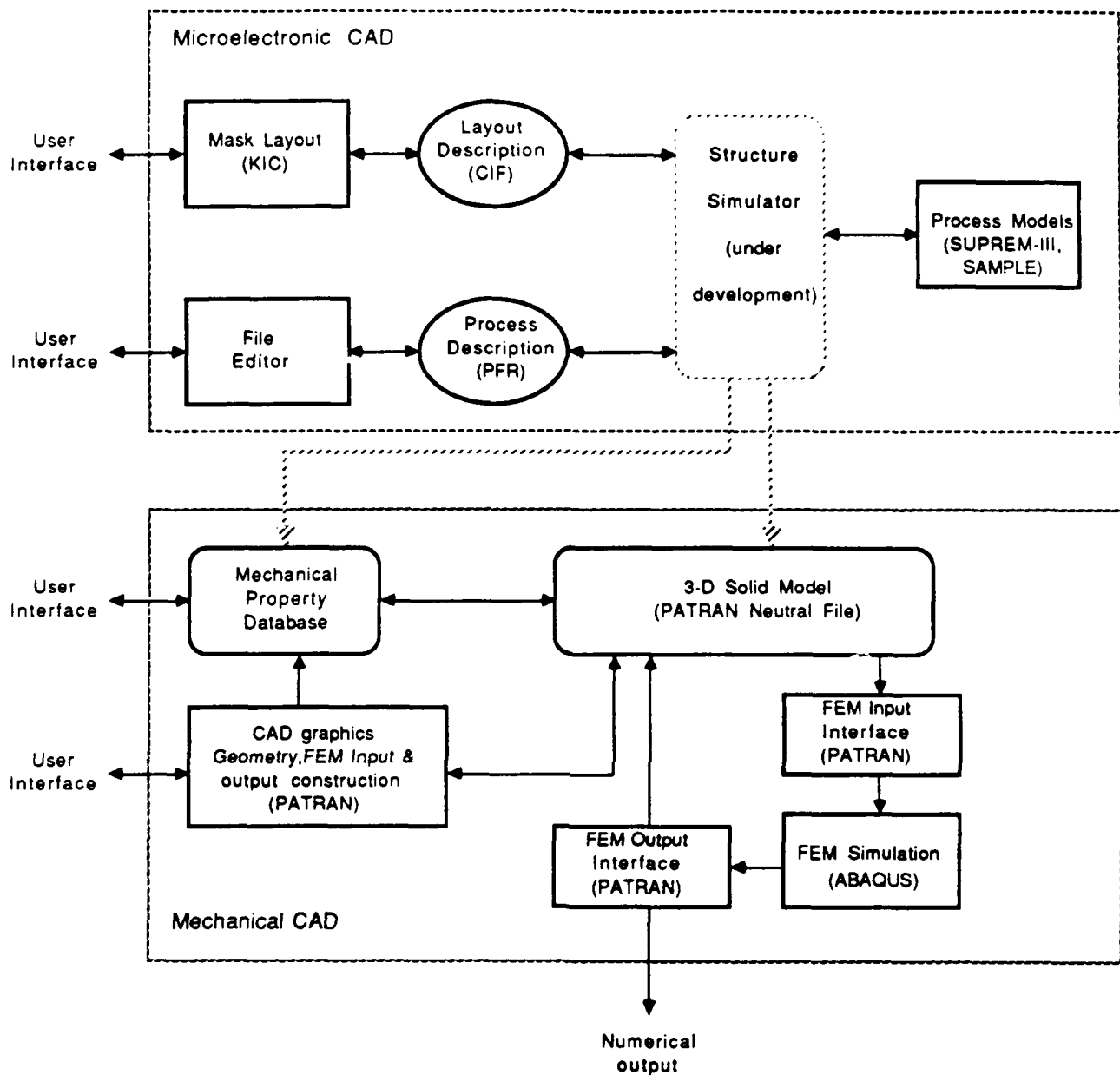


Fig. 1

CAD architecture for micro-electro-mechanical design

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1 Young's Modulus

1.1 Thermal Oxide

- 50 GPa, [3] oxide grown from 550° C to 1000° C.
- 100 GPa, [5].
- 70 GPa, [18].
- 66 GPa [27] for dry oxide grown between 875° C and 1200° C on (100) and (111) oriented Si.
- 76 GPa, [34].
- 57 GPa [36] for wet oxide grown at 960° C.
- 67 GPa [36] for dry oxide grown at 960° C.
- 110×10^5 psi [38].

1.2 PECVD Oxide

No values obtained for the Young's Modulus, E , of PECVD SiO₂

2 Poisson's Ratio

2.1 Thermal Oxide

- 0.15 [3], for oxide grown between 550° C and 1000°
- 0.20 [18], no conditions available.
- 0.17 [21], no conditions available.
- 0.164 [34], no conditions available.

2.2 PECVD Oxide

No data was retrieved for the Poisson's ratio of PECVD silicon oxide.

2.3 Bulk Oxide

- 0.18 [27].

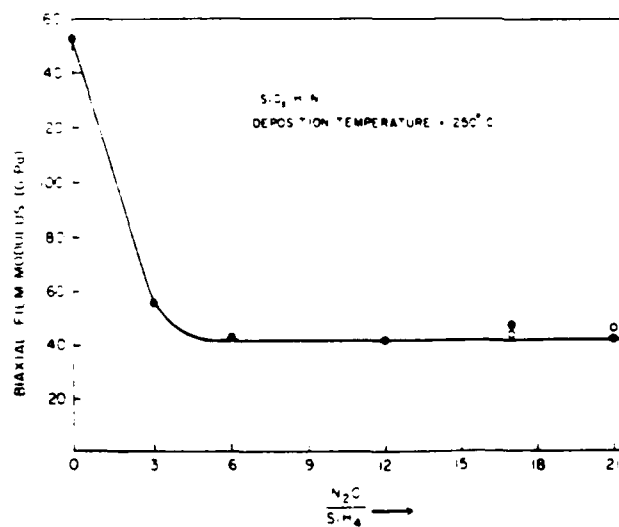
3 Biaxial Modulus

3.1 Thermal Oxide

- 100 GPa [14]
- 63.3 GPa [30] for oxide grown at 1200° C
- 70 GPa [36] for steam oxide
- 82 GPa [36] for dry oxide

3.2 PECVD Oxide

- Biaxial modulus variant with precursor gas ratio.



This data reproduced from [23]

Conditions :

Substrates: Glass, Steel, and Quartz

Temperature : 250° C

Pressure : 500 mTorr

Rf Frequency : 13.56 MHz
 Rf Power Density : 0.02 W cm^{-2}

- 42 GPa [23] for the conditions immediately above , when gas ratio exceeds 5.
- Biaxial modulus variant with deposition temperature

<u>Deposition Temperature, ° C</u>	<u>Biaxial Modulus, GPa</u>
250	42
200	42
150	40
100	43

Data reproduced from [23]

Conditions :

N_2O flow : 165 sccm
 $\text{N}_2\text{O}/\text{SiH}_4$ ratio : 12:1
 Pressure : 500 mTorr
 Temperature : variable
 Rf frequency : 13.56 MHz
 Power Density : 0.02 W cm^{-2}
 Substrates : glass, steel, quartz

- 46.6 GPa and 51.5 GPa.

This data collected from [30].

Conditions:

Temperature : 250°C
 Pressure : n/a
 Rf frequency : n/a
 Rf Power : n/a
 SiH_4 (5 % in Ar) : 100 cc min^{-1}
 O_2 : 10 cc min^{-1}
 N_2 flow : 4000 cc min^{-1}
 Substrates : Si and GaAs

- 75 GPa CVD-SiO₂ deposited at 490 A/min

100 GPa CVD SiO₂, deposited at 1900 A/min

Data collected from [37].

Conditions:

Temperature : 450° C

Substrate : (111) Si

Reagents : SiH₄ and O₂

Pressure : n/a:

Deposition rates : 490A/min & 1900A/min.

3.3 Bulk Oxide

- 88 GPa [15].

4 Density

4.1 Thermal Oxides

- Density versus oxidation temperature and pressure for several samples of dry oxide

Growth Temperature, °C	Pressure, atm	Density, g cm ⁻³
800	1	2.47
800	1	2.42
800	500	2.41
1000	500	2.35
1000	1	2.26

This data collected from [8].

Conditions :

Substrates : (111) and (100) Si

Ambient : pure dry O₂, for low pressure, ultradry for high pressure

Initial oxidation for high-pressure oxides : 1000° C, ultradry O₂, for a thickness of 1 nm

- 2.38 g cm⁻³ [10] for dry oxide grown at 500atm, 800° C
- 2.26 g cm⁻³ [10] for dry oxide grown at 1atm, 1000° C
- 2.208 g cm⁻³ [22] for dry oxide grown at 1150° C
- 2.268 g cm⁻³ [22] for dry oxide grown at 700° C
- Density varying with oxidation temperature, for dry oxide.

Temperature, °C	Density, g cm ⁻³
600*	2.286
700*	2.265
750	2.257
800	2.253
900	2.236
1000	2.224
1150	2.208

This data collected from [35].

- The results of anneals on higher density SiO_2 films.

<u>Anneal</u>	<u>Density, g cm^{-3}</u>
None	2.265
N_2 20 min, at 1000°C	2.209
N_2 16 hr, at 600°C	2.209
None	2.270
N_2 16 hr, at 700°C	2.260
$\text{N}_2 \text{ H}_2\text{O}$, 20 hr, 700°C	2.220

This data collected from [35].

Conditions :

Oxidation Temperature : 700°C

Pressure : 5000 psi

Ambient : Dry O_2

- 2.2 g cm^{-3} for wet oxide grown at 960°C [36]
- 2.25 g cm^{-3} for dry oxide grown at 960°C [36]

4.2 PECVD Oxide

- Effects of gas flow rate and annealing on oxide density

<u>Gas Flow Rate</u> <u>(sccm)</u>	<u>ρ</u> <u>(g cm^{-3})</u>	<u>ρ^*</u> <u>(g cm^{-3})</u>
3	2.09	2.22
1	2.07	2.24
0.7	2.07	2.26
0.6	2.02	2.28
05.	1.98	2.30

* Films annealed at 1000°C for 30 min in N_2 ambient

These values collected from [20].

Conditions:SiH₄ (1.5 % in Ar) flow rate : 0.3 sccmO₂ flow rate : variable

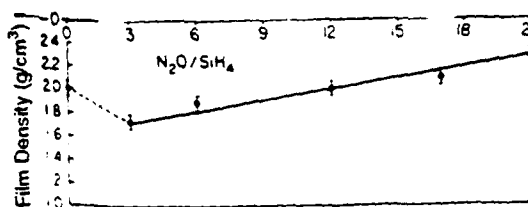
Temperature : 350° C

Pressure : 1.5 Torr

Rf frequency: 13.562 MHz

Rf Power : 50W.

• Density varying with gas ratio



This data reproduced from [23].

Conditions :

Substrates: Glass, Steel, and Quartz

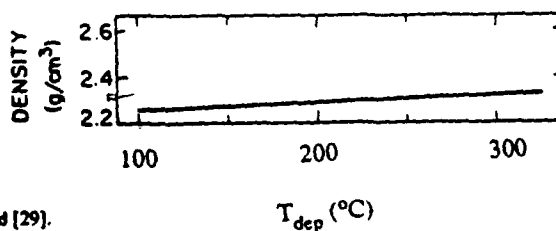
Temperature : 250° C

Pressure : 500 mTorr

Rf Frequency : 13.56 MHz

Rf Power Density : 0.02W cm⁻²N₂O Flow : 165 sccmSiH₄ Flow : variable, to suit gas ratio (see above)

• Density varying with deposition temperature.



This data reproduced [29].

Conditions:

Temperature : variable

Pressure : 1 Torr

Gas Ratio, N₂O / SiH₄ : 65

Rf frequency : 13.56 MHz

Rf Power : 24W.

- $1.97 (+/- 0.02) \text{ g cm}^{-3}$ [32]

Conditions:

$\text{N}_2\text{O} / \text{SiH}_4$ ratio : 12:1

Temperature : variable

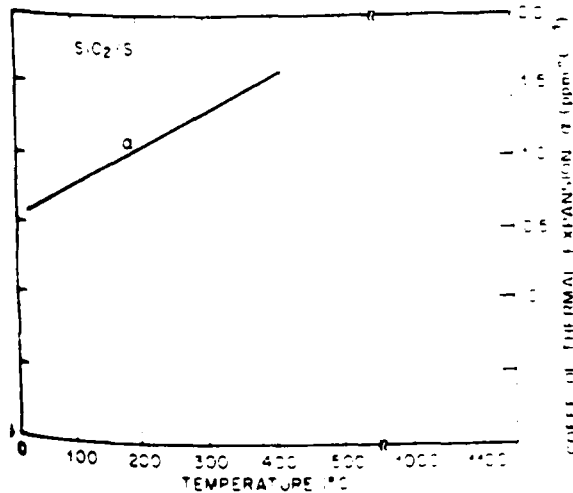
4.3 Bulk Oxide

2.2 g cm^{-3} , [33] at temperature = 300K

5 Coefficient of Thermal Expansion

5.1 Thermal Oxide

- $6 \times 10^{-7} \text{ } ^\circ\text{C}^{-1}$ [14].
- $3.5 \times 10^{-7} \text{ } ^\circ\text{C}^{-1}$ [22] dry grown from 950 - 1150° C.
- $5 \times 10^{-7} \text{ } ^\circ\text{C}^{-1}$ [30] for oxide grown at 1200° C
- $6 \times 10^{-7} \text{ } ^\circ\text{C}^{-1}$ [38] for dry oxide grown at 1200° C
- Thermal expansion coefficient vs. temperature



This data reproduced from [42].

Conditions :

Oxidation Temperature : 1050° C

Measurement Temperature : see above

Oxidation Pressure : n/a

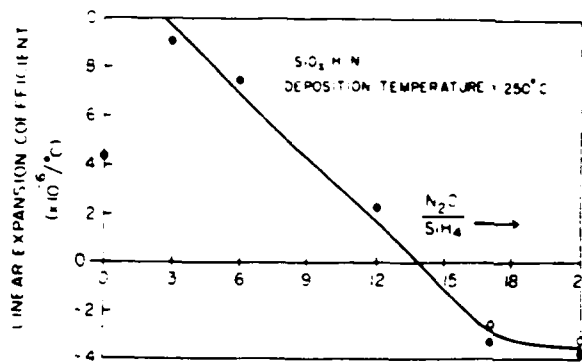
Ambient : Steam

Substrate : (100) oriented Si

Film Thickness : 4000Å

5.2 PECVD Oxide

- Coefficient of thermal expansion, α , variant with gas ratio.



Data reproduced from [23]

Conditions for PECVD:

Precursor gases : SiH₄ and N₂O
 Substrates : quartz, steel, and glass
 Temperature : 250° C
 Pressure : 500 mTorr
 Rf frequency : 13.56 MHz
 Rf Power Density : 0.02 W cm⁻²
 Total Gas flow : 200 sccm.

- Measurements of thermal expansion coefficients for varying deposition temperatures.

Deposition Temperature, ° C	$\alpha, \times 10^{-6} ^\circ C^{-1}$
250	2.3
200	2.6
150	2.2
100	2.2

This data reproduced from [24]

Conditions :

N₂O flow : 165 sccm
 N₂O/SiH₄ ratio : 12:1
 Pressure : 500 mTorr
 Temperature : variable

Rf frequency : 13.56 MHz
Power Density : 0.02 W cm^{-2}
Substrates : glass, steel, quartz.

- $3.9 - 4.1 \times 10^{-6} \text{ }^{\circ} \text{C}^{-1}$ [30]

Conditions :

Temperature : 250°C
 SiH_4 (5 % in Ar) : 100 cc min^{-1}
 O_2 flow : 10 cc min^{-1}
 N_2 flow : 4000 cc min^{-1}

- $5.5 \times 10^{-7} \text{ }^{\circ} \text{C}^{-1}$ [37]

Conditions:

Temperature : 450°C
Substrate : (111) Si
Reagents : SiH_4 and O_2
Pressure : n/a
Deposition rates : 490A/min & 1900A/min.

5.3 Bulk Oxide

- $5.2 \times 10^{-7} \text{ }^{\circ} \text{C}^{-1}$ [15].

6 Thermal Conductivity

6.1 Thermal Oxide

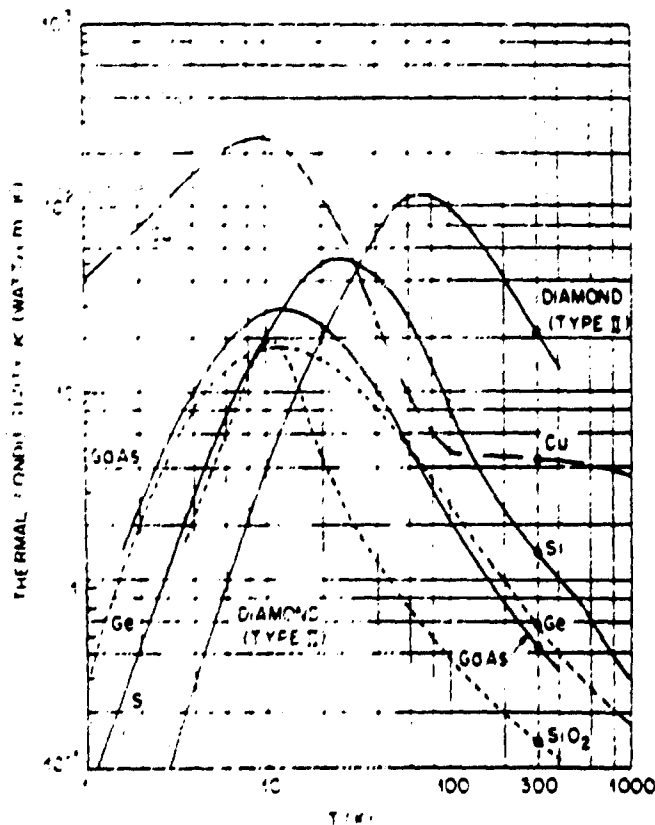
No values obtained for thermal oxide.

6.2 PECVD Oxide

No values obtained for PECVD oxide.

6.3 Bulk SiO_2

- Coefficient of thermal conductivity for bulk SiO_2 , variant with temperature.

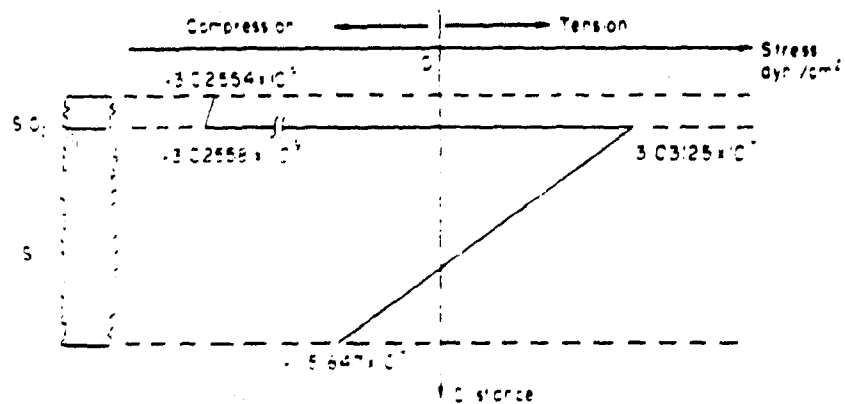


This data reproduced from [33].

7 Stress

7.1 Thermal Oxide

- 600 MPa, [3] oxide grown at 700° C.
- Schematic distribution of stress in the oxide film and its substrate.



This data reproduced from [6].

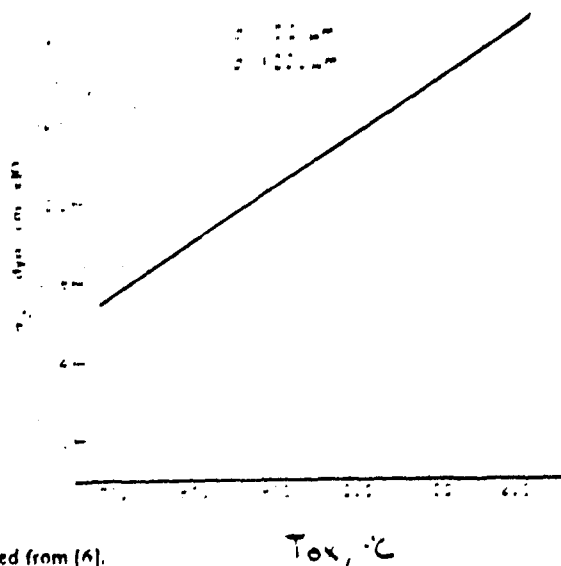
Conditions :

Oxidation Temperature : 1200° C

Growth Substrate : 2-6 Ω -cm p-type (111) Si

Anneal : 400° C. in N_2 and H_2 Annealing Duration : n/a

- Calculated stress in SiO_2 variant with oxidation temperature.



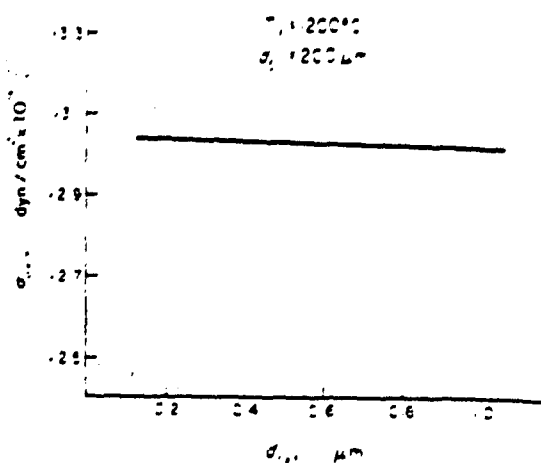
This data reproduced from [6].

Conditions :

Substrate : 2-6 $\Omega\text{-cm}$ p-type (111) Si

Anneal : 400° C. in N_2 and H_2 , duration n/a

- Calculated variation of stress with oxide thickness



This data reproduced from [6].

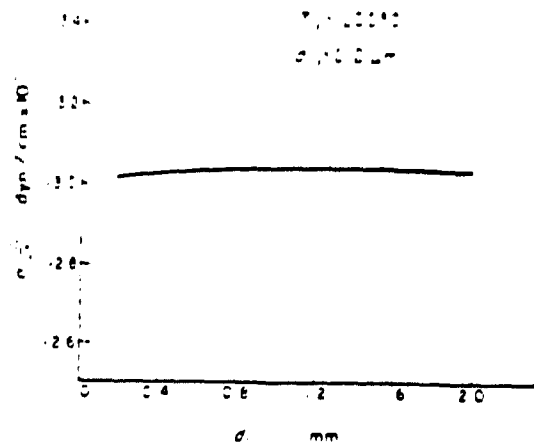
Conditions :

Growth Temperature : 1200° C

Substrate : 2-6 $\Omega\text{-cm}$ p-type (111) Si

Anneal : 400° C. in N_2 and H_2 , duration n/a

- Calculated variation of stress with substrate thickness



This data reproduced from [6].

Conditions :

Growth Temperature : 1200° C

Substrate : 2-6 Ω-cm p-type (111) Si

Anneal : 400° C. in N₂ and H₂, duration n/a

- Residual Stress Measurements for Pressure-Oxides, and Normal 1 atm Oxide.

<u>Sample Type</u>	<u>Stress, MPa</u>
Pressure Oxide,	
500 atm, 800° C	150
	400
	400
	230
	400
	280
Average:	310
Controls,	
1 atm, 1000° C	410
	420

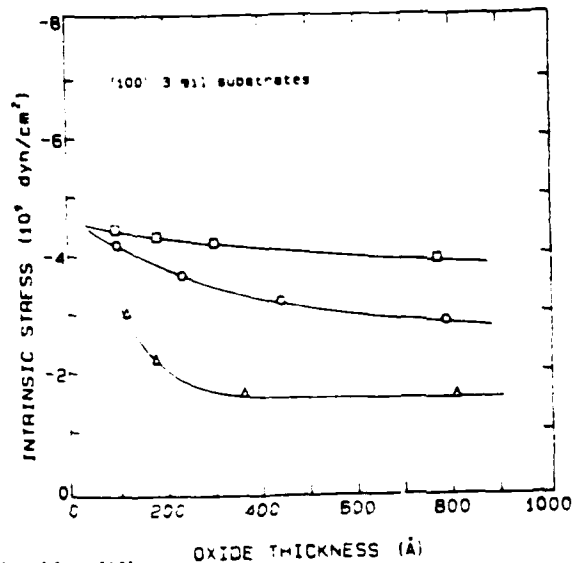
This data collected from [8].

Conditions :

Sustrates : (100) and (111) Si

H₂O content : < 1ppm

- 350 MPa [9], average compressive stress at room temperature for dry oxides grown at 1000° C.
- 310 - 340 MPa [11], average compressive stress for dry oxide grown at 500 atm, 800° C.
- Intrinsic stress variant with film thickness.



Oxidation Temperatures

700° C (□)

800° C (○)

1000° C (△)

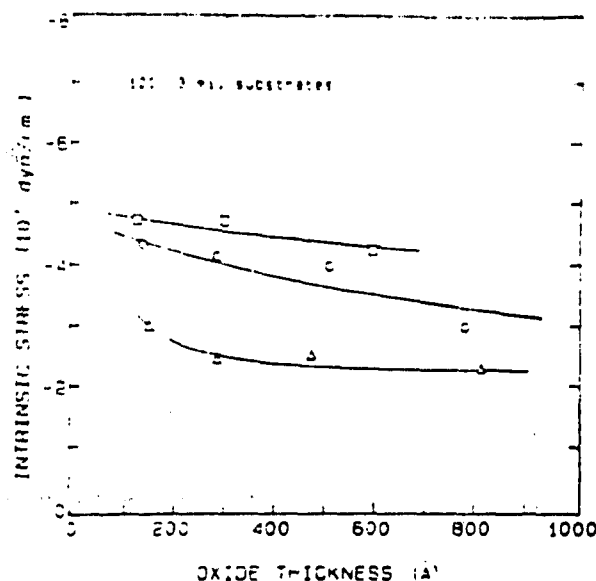
This data reproduced from [12]

Conditions :

Substrate : (100) p-type Si

Oxidation Ambient : dry O₂ with <5 ppm H₂O and <0.5 ppm hydrocarbons.

- Intrinsic stress variant with film thickness for etched oxide.



This data reproduced from [12].

Conditions :

Substrate : (100) p-type Si

Oxidation Ambient : dry O_2 with $<5 \text{ ppm H}_2\text{O}$ and $<0.5 \text{ ppm}$

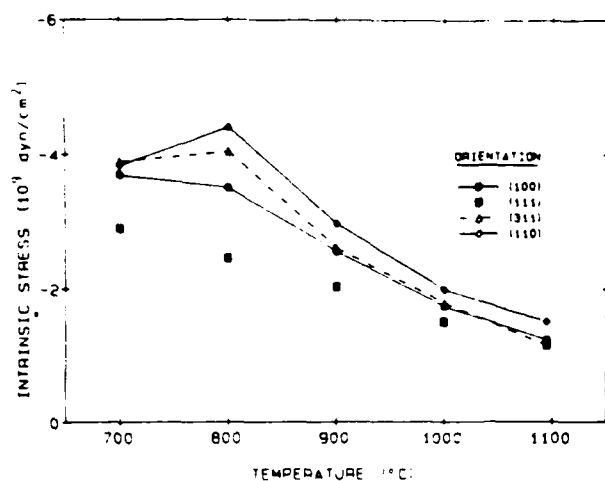
Etching : in HF solution, ($\text{NH}_4:\text{HF} = 50:1$) hydrocarbons.

- 450 MPa, [12] maximum stress for a limit of a film with zero thickness.
- Total stress for dry oxides grown on two Si substrates at different temperatures.

<u>Substrate</u>	<u>Temperature, ° C</u>	<u>Total Stress, MPa</u>
p+ $10^{-2} \Omega\text{cm}$ Si	850	150
p+ $10^{-2} \Omega\text{cm}$ Si	1090	290
p 1 Ωcm Si	850	80
p 1 Ωcm Si	1090	260

This data collected from [13].

- Stress varying with oxidation temperature for four Si orientations.



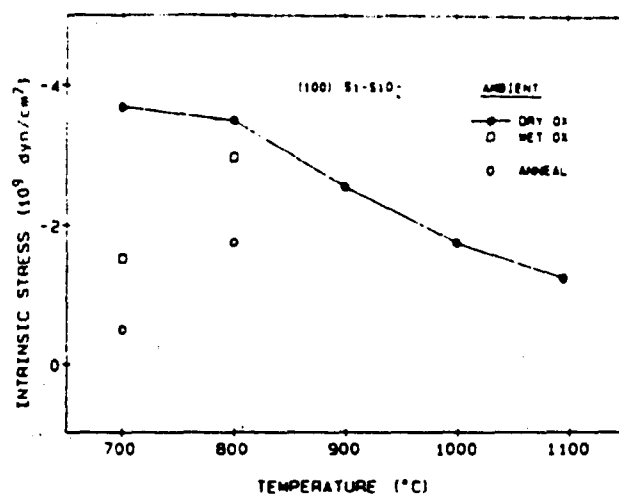
This data reproduced from [15].

Conditions :

Ambient : dry O_2 , with $<5 \text{ ppm H}_2\text{O}$, and $<0.5 \text{ ppm hydrocarbons}$

Pressure : 1 atm

- Stress varying with oxidation temperature for (100) Si.



This data reproduced from [15].

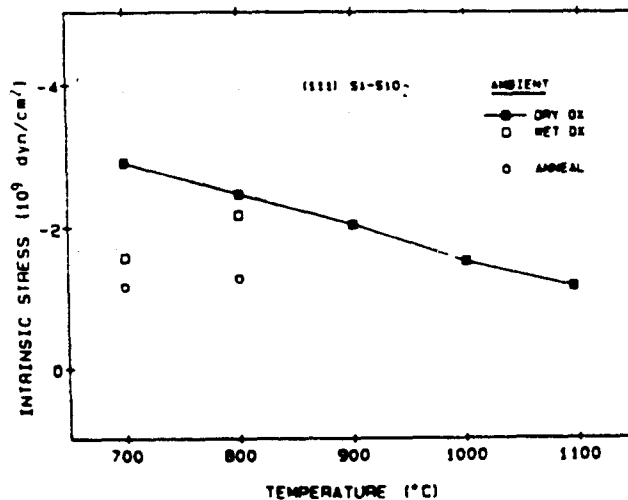
Conditions :

Ambient : dry O₂, with <5 ppm H₂O, and <0.5 ppm hydrocarbons

Pressure : 1 atm

Annealed : 1000° C. in N₂

- Stress varying with oxidation temperature for (111) Si.



This data reproduced from [15].

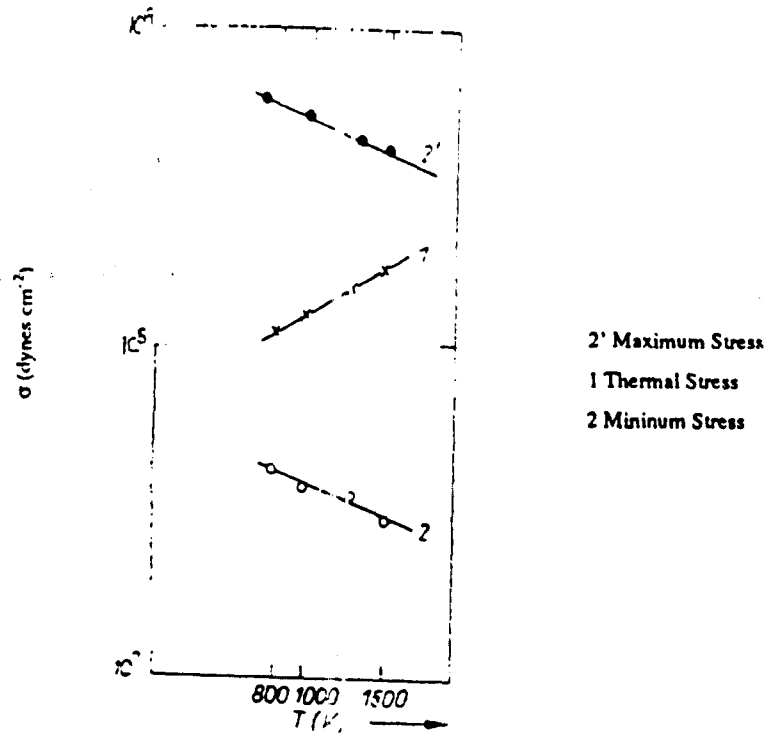
Conditions :

Ambient : dry O₂, with <5 ppm H₂O, and <0.5 ppm hydrocarbons

Pressure : 1 atm

Annealed : 1000° C. in N₂

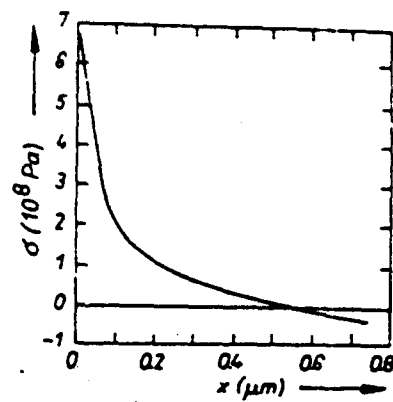
- Stress varying with oxidation temperature for dry oxide.



This data reproduced from [21].

Conditions : n/a.

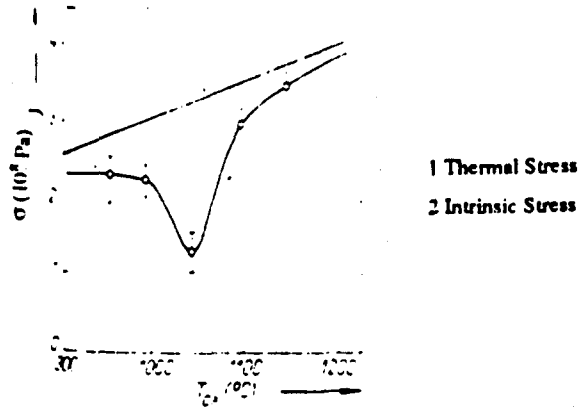
- Stress distribution over the thickness for dry oxide.



This data reproduced from [21].

Conditions : n/a.

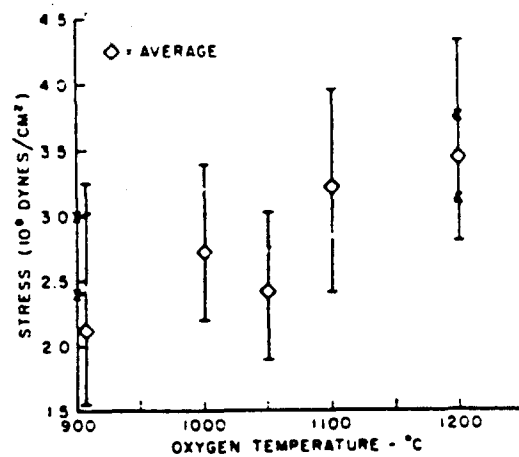
- Intrinsic and thermal stresses varying with oxidation temperature for dry oxide.



This data reproduced from [21].

Conditions : n/a.

- Stress measurements from beam experiment.



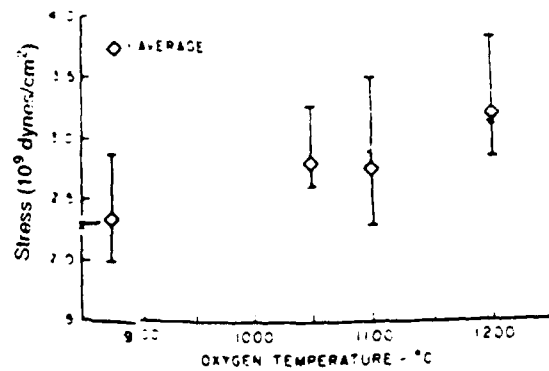
This data reproduced from [27].

Conditions :

Substrates : (111) and (100) - oriented Si

Oxidation : Dry

- Stress measurements from balloon experiment.

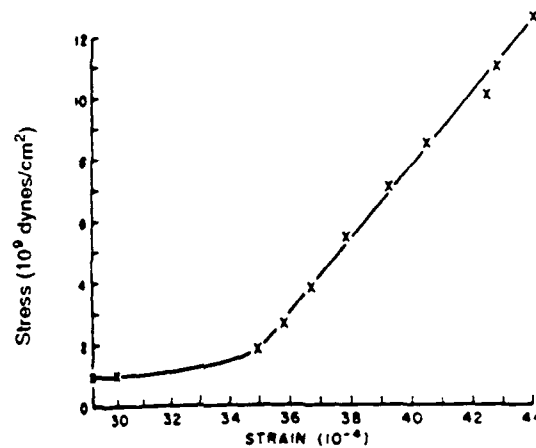


This data reproduced from [27].

Substrates : (111) and (100) - oriented Si

Oxidation : Dry

- Stress vs. strain for typical oxide balloon



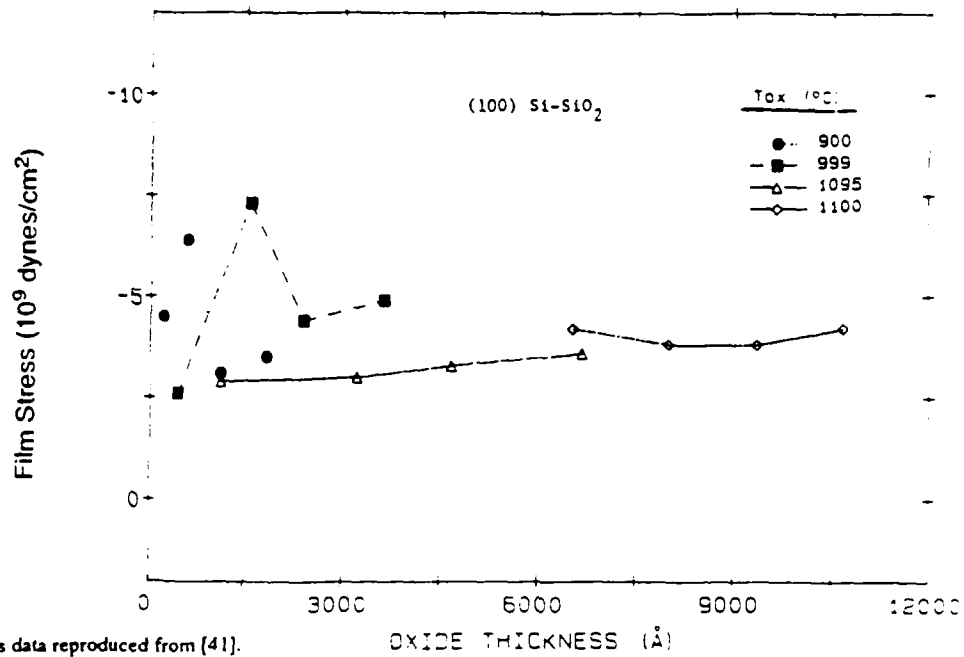
Substrates : (111) and (100) - oriented Si

Oxidation : Dry

This data reproduced from [27].

- 340 MPa [30] measured at room temp., grown at 700° C.
- 400 MPa compressive [35], grown at 700° C.
- 50 MPa tensile [35], grown at 1150° C.

- 220 - 360 MPa compressive, [37] measured at room temp., grown at 1100° C.
- Total film stress, measured at room temperature, vs. oxide thickness



Conditions :

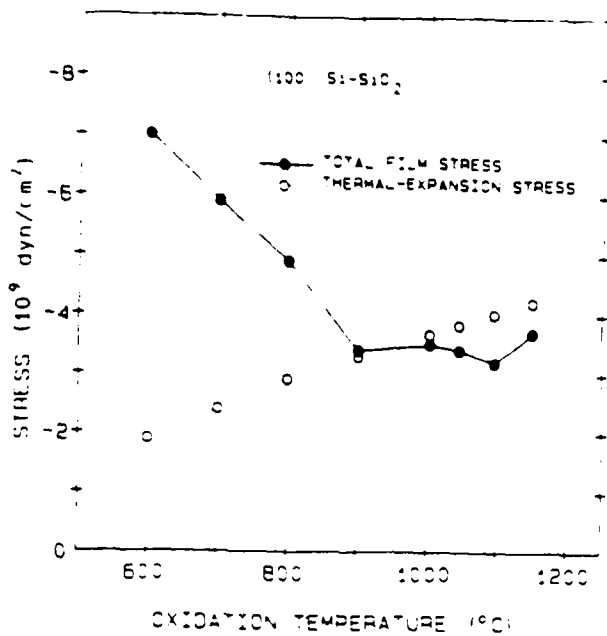
Oxidation Temperature : see above.

Oxidation Pressure : 1 atm

Ambient : Dry O₂

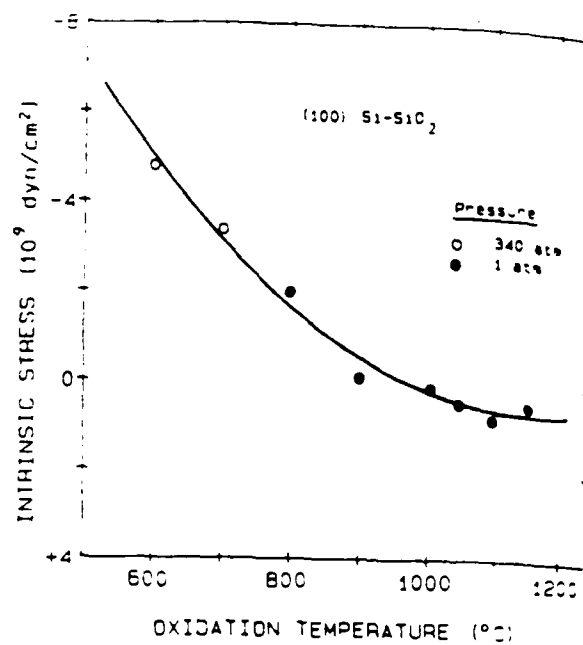
Substrate : *p*-type (100) oriented Si

Components of stress vs.
oxidation temperature



This data reproduced from [41].

Intrinsic stress vs.
oxidation temperature



Conditions For Both Graphs:

Oxidation Temperature : see above

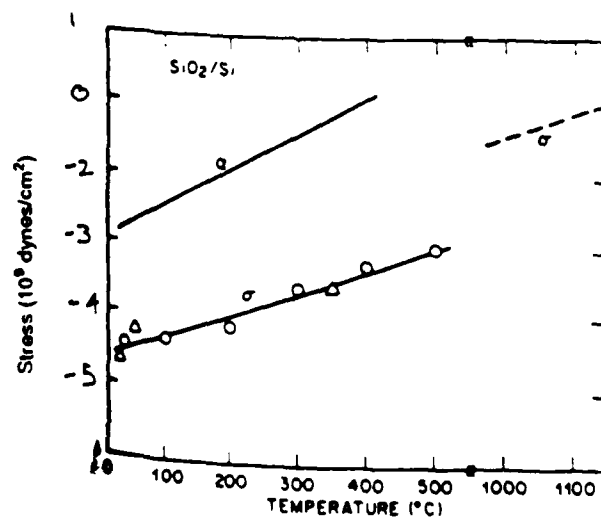
Measurement Temperature : room temperature

Oxidation Pressure : 1 atm (unless otherwise specified)

Ambient : Dry O₂

Substrate : *p*-type (100) oriented Si

• Stress vs. temperature

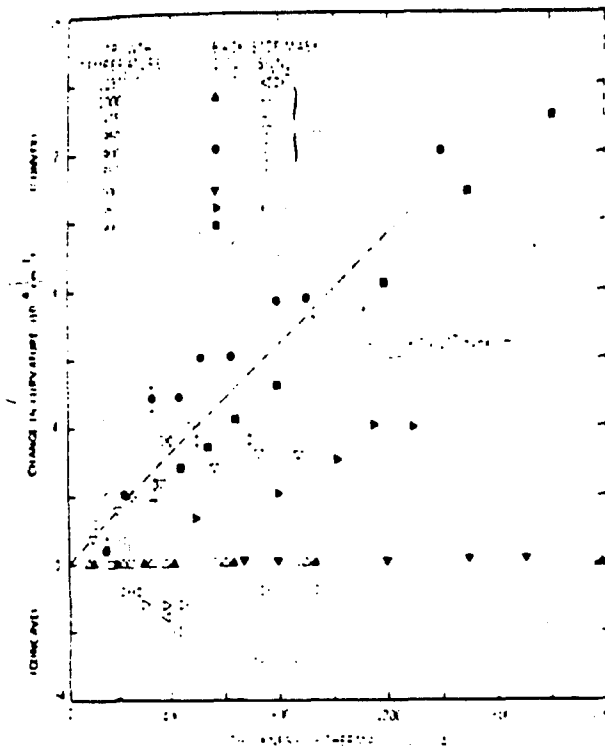


This data reproduced from [42].

Conditions :

Oxidation Temperature : 1050° C
 Measurement Temperature : see above
 Oxidation Pressure : n/a
 Ambient : Steam
 Substrate : (100) oriented Si
 Film Thickness : 4000A

- Change in curvature induced by stress, vs. oxide thickness



This data reproduced from [44].

Dashe line represents the expected curvature change for SiO₂ stress level of 700MPa.

Conditions :

Oxidation Temperature : see above
 Measurement Temperature : ox. temp.
 Oxidation Pressure : n/a
 Ambient : wet O₂
 Substrate : 1-Ω-cm P-doped (111) and (100) Si
 Film Thickness : 4000A

• Interfacial stress at room temperature for various substrates.

<u>P₂O₅</u> <u>Diffusion</u>	<u>Direction of</u> <u>Stress Measurement</u>	<u>Si</u> <u>Orientation</u>	<u>Stress,</u> <u>MPa</u>
No	(110)	(111)	47
No	(211)	(111)	450
Yes	(110)	(111)	250
No	(110)	(110)	390
No	(100)	(110)	390
Yes	(110)	(110)	260
No	(110)	(100)	400
No	(110)	(100)	380
Yes	(110)	(100)	240

This data reproduced from [45].

Conditions :

Oxidation Temperature : 1200° C

Measurement Temperature : room temperature >

Oxidation Pressure : n/a

Ambient : wet O₂ (dew point temperature 90° C)

Substrate : 5 Ω-cm (111), (110), and (100) Si

Film Thickness : 8400 Å

P₂O₅ Diffusion : in N₂ for 30 min. at 920° C

• Oxide stress at room temperature for quickly and slowly cooled films

<u>Sample Type and Cooling</u>	<u>Film Stress, MPa</u>
Dry Oxide	
Slowly Cooled	270
Quickly Cooled	370
Wet Oxide	
Slowly Cooled	160
Quickly Cooled	280

This data reproduced from [45].

Conditions :

Oxidation Temperature : 1200° C

Measurement Temperature : room temperature

Measurement Direction : (110) direction

Oxidation Pressure : n/a

Ambient : wet oxide, dew point temperature = 90° C

dry oxide, dew point temperature = -40° C

Substrate : (110) - oriented P-doped Si. Film Thickness : 8600-9200 Å

Cooling Duration : for quick cooling, immediate exposure to air

Cooling Duration : for quick cooling, immediate exposure to air
for slow cooling, duration = 5hr

- Oxide stress at room temperature for various doping levels of Si

Slice Resistivity, $\Omega\text{-cm}$	Doping Level, cm^{-3}	Oxide Stress, MPa
1000	4×10^{12}	280
5-10	$8.5 \times 10^{14} - 4 \times 10^{14}$	270
0.37	1.6×10^{16}	200
0.035	4.5×10^{17}	260
0.0015	5.5×10^{19}	300

This data reproduced from [45].

Conditions :

Oxidation Temperature : 1200° C

Measurement Temperature : room temperature

Measurement Direction : (200) direction

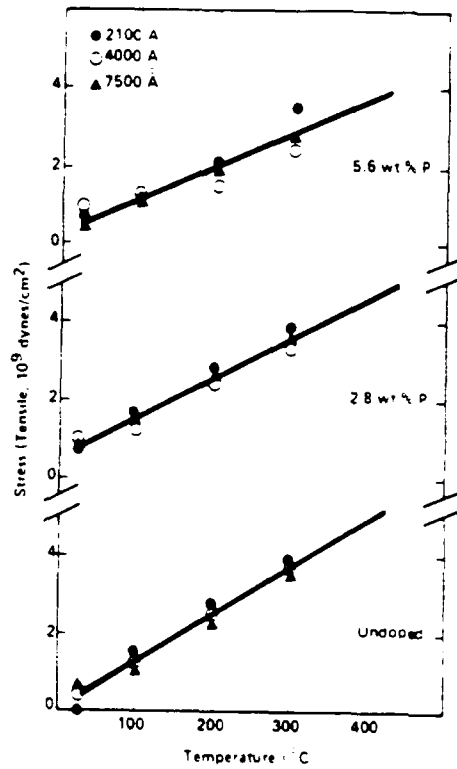
Oxidation Pressure : n/a

Ambient : wet oxide, dew point temperature = 90° C

Substrate : (111) - oriented P-doped Si. Film Thickness : 9200Å

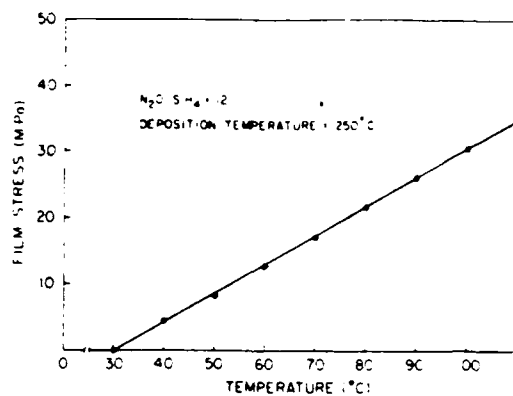
7.2 PECVD Oxide

- Stress varying with temperature (not oxidation temperature).



This data reproduced from [14].

- Thermally induced stress in the oxide, as a function of the measured temperature.



This data reproduced from [23] and [24]

Conditions :

Temperature : 250° C

Pressure : 500 mTorr

N_2O/SiH_4 : 12:1

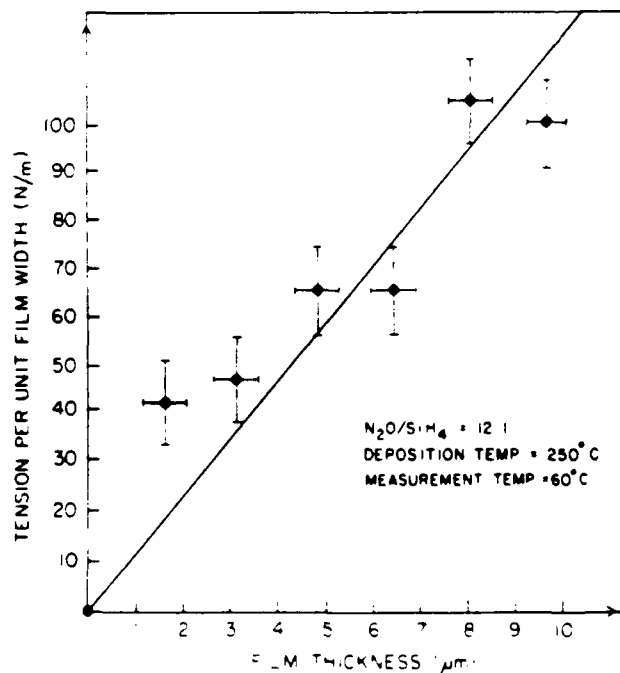
Total gas flow rate : 200 sccm

Rf frequency : 13.56 MHz

Rf Power Density : 0.02 W cm⁻²

Substrates : quartz, steel, glass.

- Tension in the film as a function of film thickness.



This data reproduced from [24]

Conditions :

Temperature : 250° C

Pressure : 500 mTorr

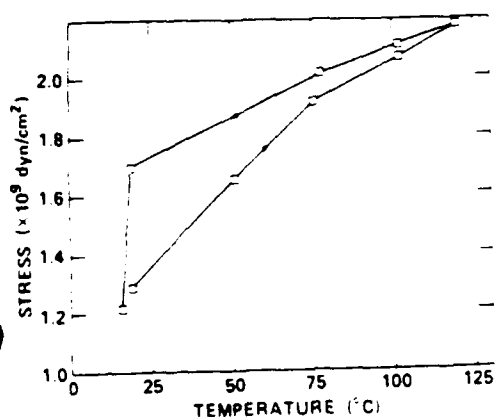
Rf frequency : 13.56 MHz

Rf Power Density : 0.02 W cm⁻²N₂O / SiH₄ : 12:1SiH₄ flow rate : 200 sccm

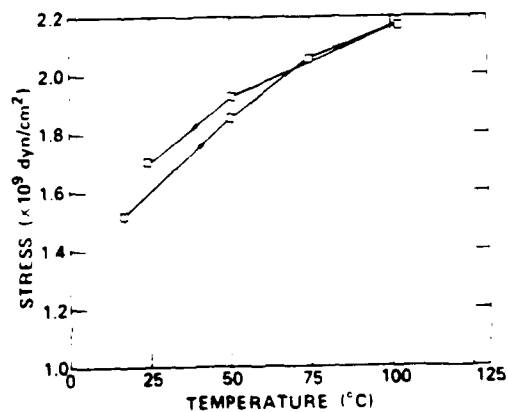
Substrates : glass, steel, quartz

- Stress in a CVD SiO₂ film as a function of temperature.

Air



Dry Air



This data reproduced from [30].

Conditions :

Deposition Temperature : 250° C

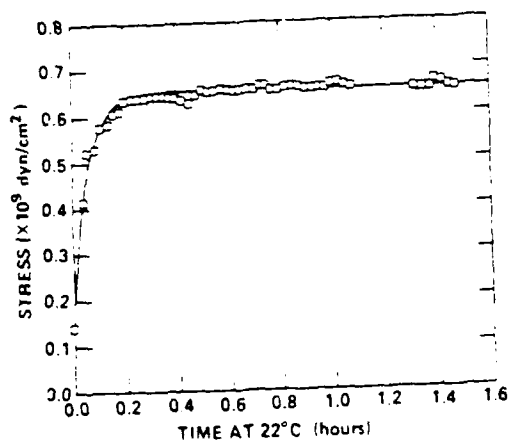
SiH₄ (5% in Ar) flow : 100 cc/minO₂ flow : 10 cc/minN₂ flow : 4000 cc/min

Deposition Thickness : 0.65 μm

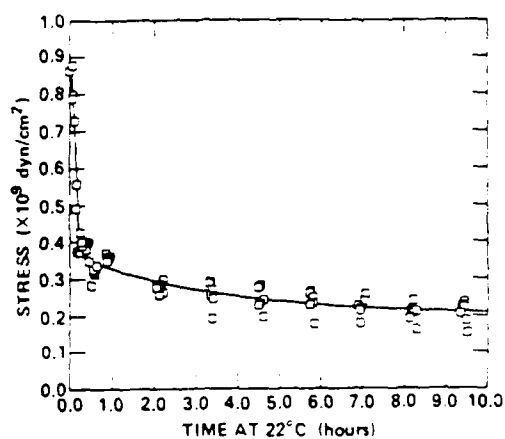
Substrates : GaAs and Si

• Time variation of stress in CVD SiO_2

Kept in air, 49% rel. humid.,
exposure to a dry N_2
ambient at 22° C.



Kept in dry N_2 , upon exposure
to air, 60% RH, at 22° C.



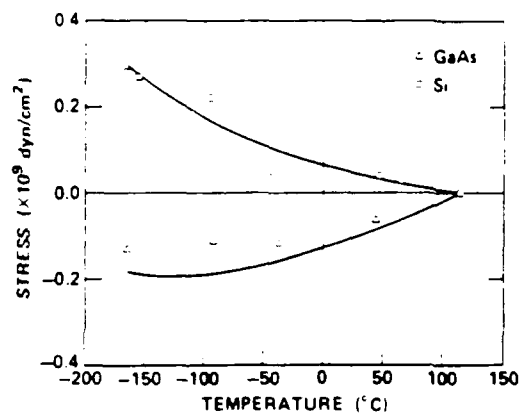
This data reproduced from [30].

Conditions :

Deposition Temperature : 250° C
 SiH_4 (5% in Ar) flow : 100 cc/min
 O_2 flow : 10 cc/min
 N_2 flow : 4000 cc/min
 Depositoin Thickness : 0.65 μm
 Substrates : GaAs and Si

- $\sigma(T) - \sigma(115^\circ \text{C})$ as a function of temperature.

Solid curves represent calculated values. Measurements done in vacuum.

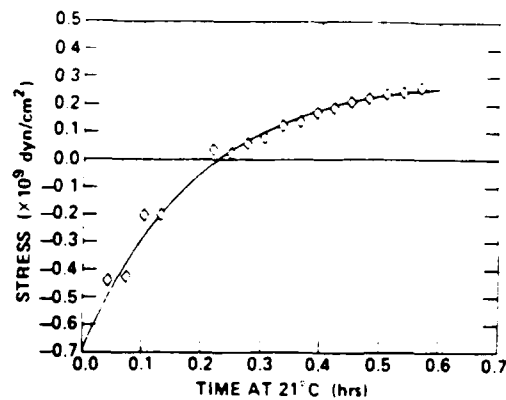


This data reproduced from [30].

Conditions :

Deposition Temperature : 250°C
 SiH_4 (5% in Ar) flow : 100 cc/min
 O_2 flow : 10 cc/min
 N_2 flow : 4000 cc/min
 Deposition Thickness : $0.47 \mu\text{m}$
 Substrates : GaAs and Si

- Time varying stress for a CVD SiO_2 film, previously kept in air, 48% RH, at 21°C , upon exposure to $40\ \mu\text{m Hg}$.

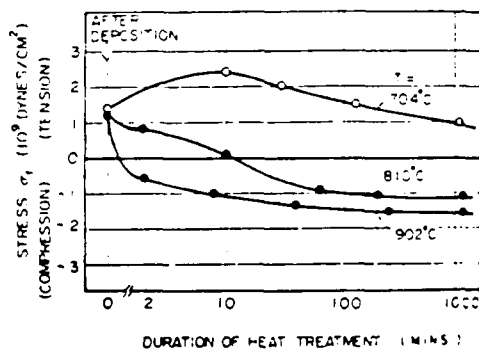


This data reproduced from [30].

Conditions :

Deposition Temperature : 250°C
 SiH_4 (5% in Ar) flow : 100 cc/min
 O_2 flow : 10 cc/min
 N_2 flow : 4000 cc/min
 Deposition Thickness : $0.47\ \mu\text{m}$
 Substrates : GaAs

- Stress change in CVD SiO_2 after heat treatments.



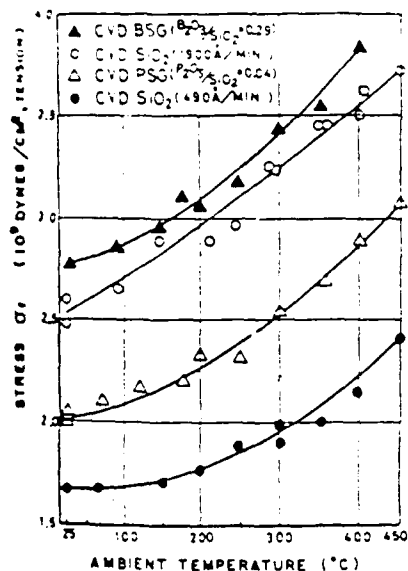
This data reproduced from [37].

Conditions :

Deposition Temperature : $400^\circ\text{C} - 450^\circ\text{C}$
 Deposition Pressure : n/l
 Reagent : SiH_4
 Substrate : (111) Si, $200\ \mu\text{m}$ thick

Heat Treatment Temperature : see above

• Stress in CVD films vs. ambient temperature



This data reproduced from [37].

Conditions :

Deposition Temperature : 400° C - 450° C

Deposition Pressure : n/l

Reagent : SiH₄

Substrate : (111) Si, 200μm thick

• Stress in CVD SiO₂ films vs. deposition rate

Deposition Rate (A/min)	Stress after Deposition, MPa	Intrinsic Stress, MPa
1900 A/min	220	370
490 A/min	170	240

This data collected from [37].

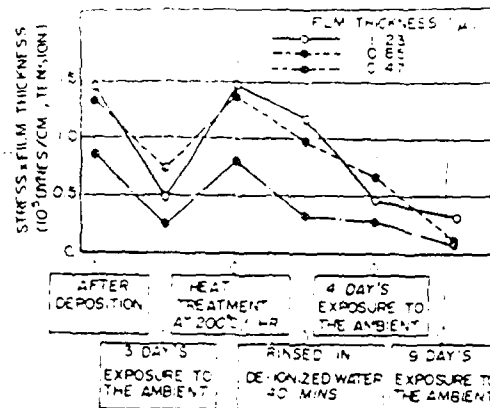
Deposition Temperature : 400° C - 450° C

Deposition Pressure : n/l

Reagent : SiH₄

Substrate : (111) Si, 200μm thick

- Stress change of CVD SiO_2 film after various treatments.



This data collected from [37].

Conditions :

Deposition Temperature : 400° C - 450° C

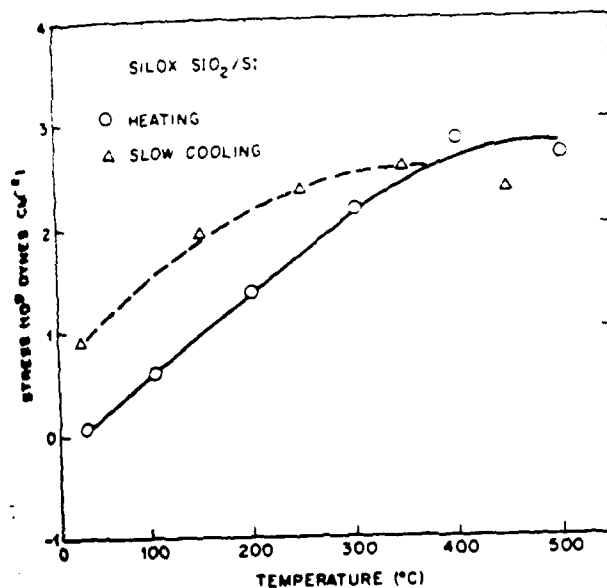
Deposition Pressure : n/l

Reagent : SiH_4

Substrate : (111) Si, 200μm thick

Film Thickness : see above

- Temperature dependence of CVD SiO_2 stress on Si



This data reproduced from [42]

Conditions :

Deposition Temperature : 480° C

Deposition Pressure : n/a

Reagents : SiH_4 and O_2

Film Thickness : 6000Å

8 Stress Relaxation Time

8.1 Thermal Oxide

- Stress relaxation time variant with temperature.

<u>Temperature, ° C</u>	<u>Stress Relaxation Time, hrs</u>
800	>1000
900	>1000
950	-
1000	~25-60
1050	~6-20
1100	~1-3
1180	<<0.3

This data collected from [5]

Conditions :

Oxidation Temperature : see above

Oxidation Pressure : n/a.

Ambient : dry oxide.

- Stress relaxation time variant with temperature.

<u>Temperature, ° C</u>	<u>Stress Relaxation Time, hrs</u>
700	5278
800	175
1000	0.2

This data collected from [12].

Conditions :

Substrate : (100) p-type Si

Oxidation Ambient : dry O₂ with <5 ppm H₂O and <0.5 ppm hydrocarbons.

- Stress relaxation time variant with temperature.

<u>Temperature, ° C</u>	<u>Stress Relaxation Time, hrs</u>
800	5100
900	21
1000	0.2
1100	(10 seconds)

This data collected from [35].

Conditions :

Oxidation Temperature : see above

Pressure : 1 atm

Substrate : (111) and (100) oriented Si crystals

8.2 PECVD Oxide

No information available for PECVD oxide films.

8.3 Bulk Oxide

- Stress Relaxation times for I. R. Vitreosil.

<u>Temperature, ° C</u>	<u>τ, hrs.</u>
900	>50,000
1000	>1000
1100	170
1200	24
1300	6
1400	2

This data collected from [39].

9 Viscosity

9.1 Thermal Oxide

- Viscosity variant with temperature.

<u>Temperature, ° C</u>	<u>Stress Relaxation Time, hrs</u>
800	$>>10^{18}$
900	$>10^{18}$
950	$\sim 10^{18}$
1000	7×10^{16}
1050	6×10^{15}
1100	5×10^{14}
1180	$\sim 10^{13}$

This data collected from [5]

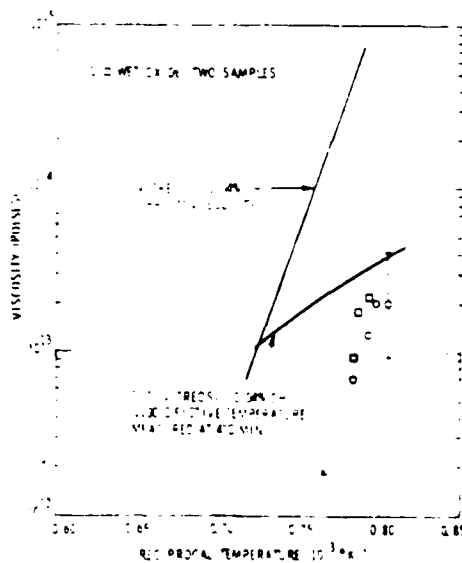
Conditions :

Oxidation Temperature : see above

Oxidation Pressure : n/a.

Ambient : dry oxide.

- Viscosity (Poise), as a function of temperature.



This data reproduced from [40]

Conditions

Oxidation Temperature : 1100° C

Oxidation Ambient : steam

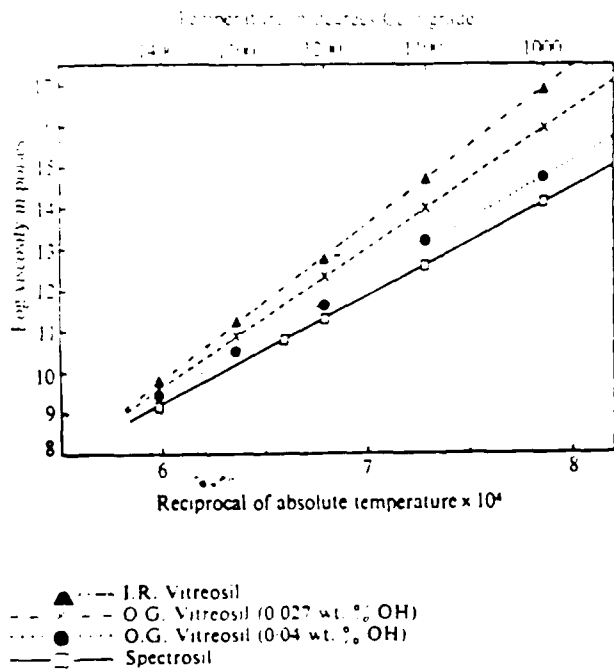
Substrate : standard *n*-type P-doped 1.0 Ω -cm Si wafers of (100) orientation

9.2 PECVD Oxide

No information available for PECVD Oxides.

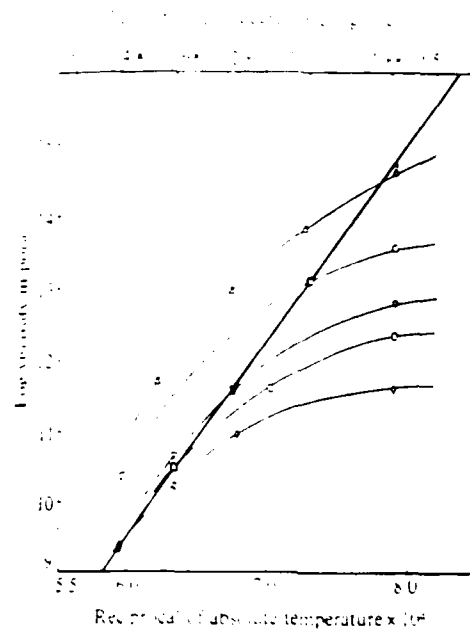
9.3 Bulk Oxide

- Equilibrium viscosities for different types of vitreous silica.



This data reproduced from [39].

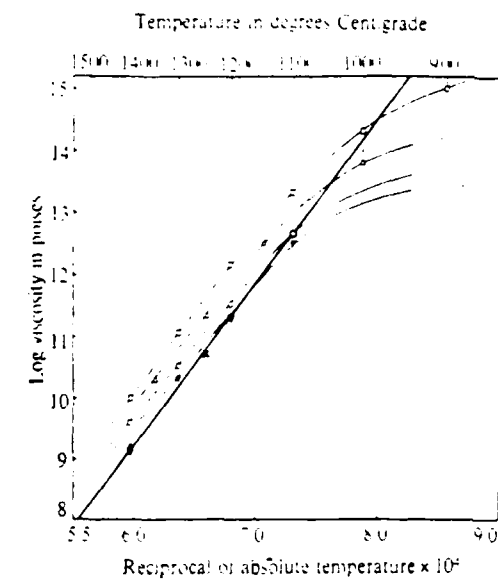
• Variation of viscosity with temperature for Vitreosil.



- Δ Fictive temperature 1000 C
- $\square$ Fictive temperature 1100 C
- ∇ Fictive temperature 1200 C
- $+$ Fictive temperature 1300 C
- $\diamond$ Fictive temperature 1400 C
- Equilibrium viscosity curve—bold symbols

This data reproduced from [39].

• Variation of Viscosity with temperature for Spectrosil.



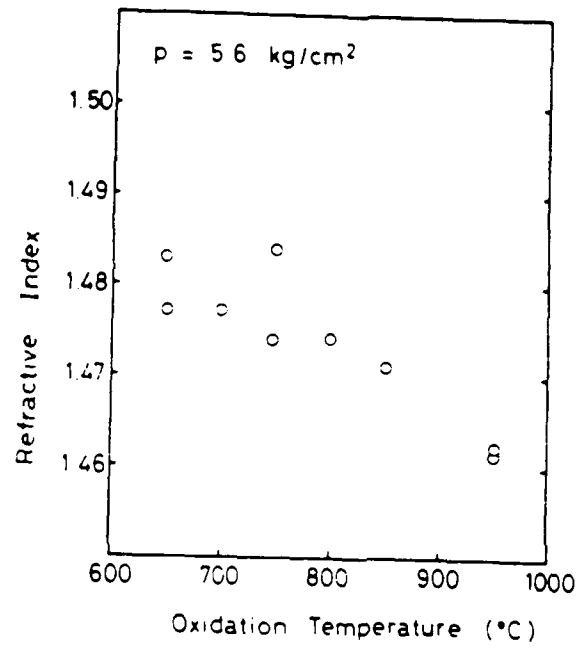
- Fictive temperature 900° C
- Δ Fictive temperature 1000° C
- Fictive temperature 1100° C
- ▽ Fictive temperature 1200° C
- ▲ Fictive temperature 1250° C
- △ Fictive temperature 1400° C
- Equilibrium viscosity curve—bold symbols

This data reproduced from [39].

10 Refractive Index

10.1 Thermal Oxide

- Refractive Index varying with oxidation temperature.



This data reproduced from [1].

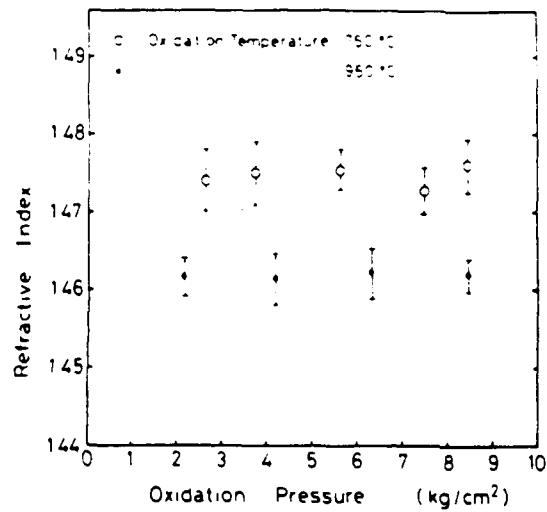
Conditions :

H_2 / O_2 flow ratio : 1.8

Ambient : dry O_2

Pressure : 5.6 kg cm^{-2}

- Refractive Index varying with oxidation pressure.



This data reproduced from [1]

Conditions :

H_2 / O_2 flow ratio : 1.8

Ambient : dry O_2

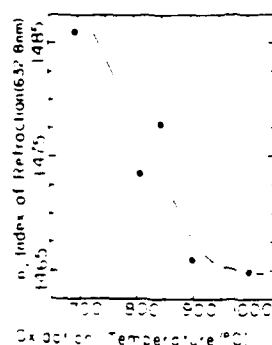
Pressure : 5.6 kg cm⁻².

- Effect of oxidation temperature and pressure on refractive index, for dry oxide.

Oxygen Pressure, atm	(Oxidation Temperature,) °C	Index of Refraction
1	700	1.486
1	800	1.474
1	850	1.478
1	900	1.466
1	1000	1.465
(high pressure)		
136	550	...
207	550	1.476
136	600	1.474
212	600	1.484
136	700	1.475
207	700	1.487
212	700	1.476
306	700	1.474
211	800	1.473

This data taken from [3]

- Index of refraction as a function of oxidation temperature.



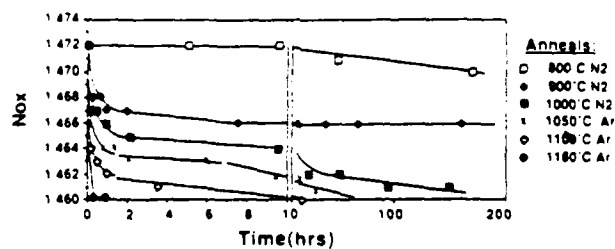
This data reproduced from [3].

Conditions :

Pressure : 1 atm

Ambient : dry O_2 .

- Refractive index versus anneal time for dry - oxide grown thermally on (111) Si at 800° C.



This data reproduced from [5].

Conditions :

Pressure : n/a.

- 1.460 for fully relaxed SiO_2 thin film. [5]
- 1.466 for an oxide, dry-grown at 1000° C. [5]

- Refractive Index values before and after relaxation.

<u>Growth Temperature, ° C</u>	<u>Refractive Index</u>	<u>Relaxation Treatment</u>	<u>Ref. Index After Relaxation</u>
800	1.472	none	
800	1.472	1000° C 1 hr N ₂	1.467
800	1.472	1000° C 16 hr N ₂	1.464
800	1.472	1180° C 40 min N ₂	1.460
1180	1.460	none	
800	1.472	1204° C 5 min O ₂	1.460
800	1.472	none	
800	1.472	1180° C 40 min	1.460
800	1.472	Corona*	
1000	1.467	800° C 20 min O ₂	1.460
1000	1.467	none	
		Corona*	1.460
1000	1.467	900° C 4 hr O ₂	
		none	

* Stress relaxation performed by a Corona discharge device.

This data taken from [7].

- Refractive Index measurements for Pressure Oxide, Normal Oxide, and Low Temperature Oxide.

<u>Sample Type</u>	<u>Film Thickness, nm</u>	<u>Refractive Index</u>
Pressure Oxide,		
500 atm, 800° C	947.6	1.476
	153.3	1.475
	983.0	1.473
	941.1	1.473
	685.7	1.475
	129.8	1.478
	960.0	1.475
	967.2	1.467
	684.6	1.477
Average:		
Controls,		
1 atm, 1000° C	959.0	1.461
	951.4	1.461
	1293.0	1.462
Average :		1.461
Low Temperature Oxide,		
1 atm, 800° C	...	1.468
	...	1.476

This data reproduced from [8].

Conditions :

Sustrates : (100) and (111) Si

H₂O content : < 1ppm

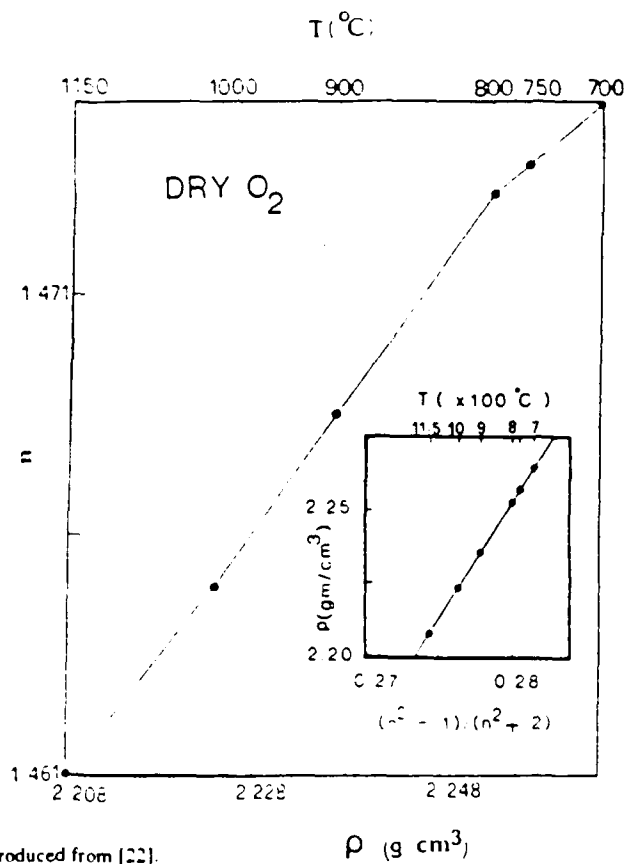
- 1.475 for oxide prepared at 500 atm. and 800° C. [10]
- 1.461 for oxide prepared at 1 atm. and 1000° C. [10]
- 1.462 for thermal oxide prepared at 1100° C. [11]
- The effect of oxidation time and annealing on refractive index and film thickness

Duration of Second Oxidation, hrs	Thickness of Unannealed Film, Å	η of Unannealed Film	Thickness of Annealed Film, Å	η of Annealed Film
1	9	1.474	23	1.463
2	15	1.475	44	1.465
4	30	1.475	83	1.465
9	78	1.474	163	1.464
19	178	1.473	267	1.464

Data taken from [17].

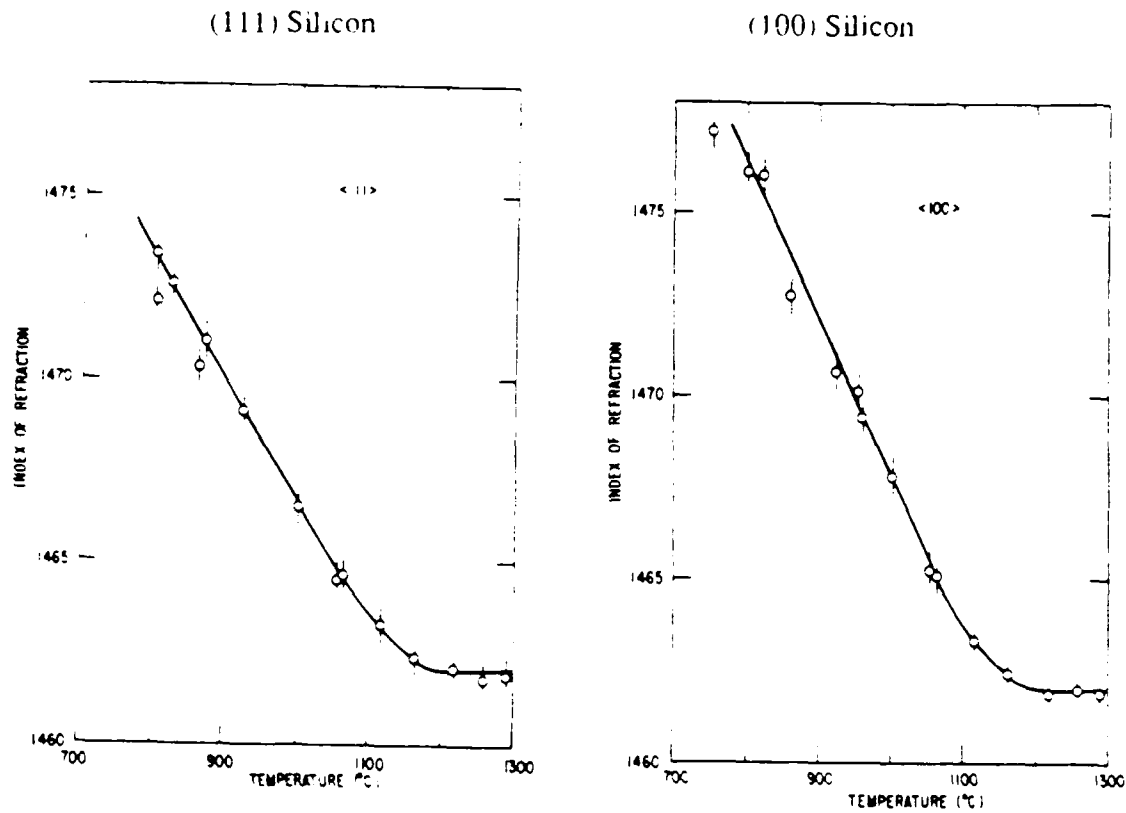
Both oxidations were conducted in pure dry O₂ at 800° C. The annealing was performed for 1 hour at 1000° C in pure Ar.

- Refractive Index as it depends upon density.



Reproduced from [22].

- Refractive Index versus deposition temperature for thermal oxides grown on two different silicon substrates.



Reproduced from [34].

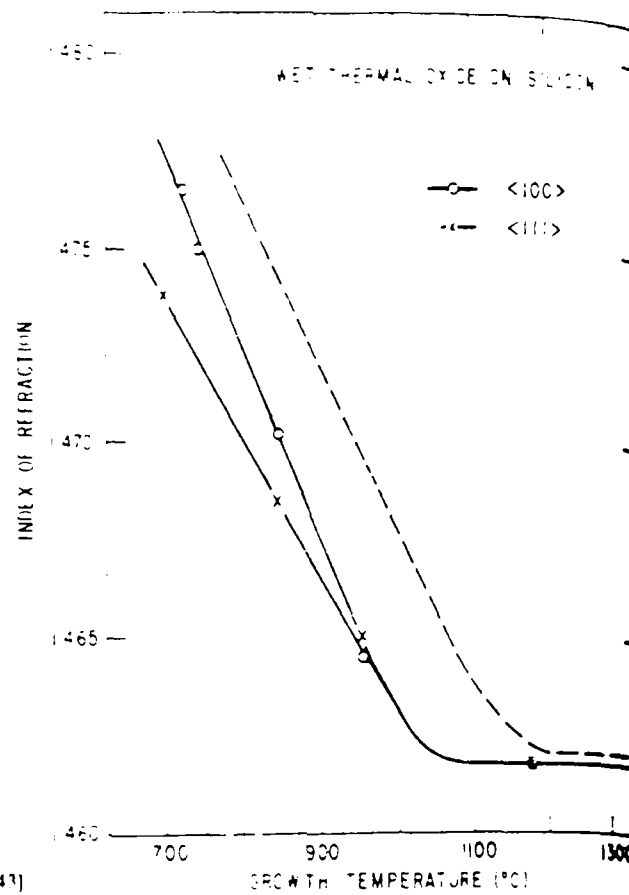
- Variation of refractive index with oxidation temperature for thermally grown SiO_2 films.

Temperature, °C	Refractive Index
600*	1.480
700*	1.475
750	1.473
800	1.472
900	1.468
1000	1.465
1150	1.461

* These samples grown at 5000 psi O_2 , all others at 1 atm O_2 .

Reproduced from [35]

• Index of refraction vs. oxidation temperature



This data reproduced from [43]

Conditions :

Oxidation Temperature : see above

Ambient : 95° C saturated water vapor

Substrates : (111) and (100) lightly doped Si

10.2 PECVD Oxide

- Table of values of refractive index, and deposition parameters.

<u>Deposition Temperature, ° C</u>	<u>Flow Rates N₂O:SiH₄:He, sccm</u>	<u>Deposition Rate, Å/min.</u>	<u>Refractive Index</u>
350	500:200:0	510	1.462
350	500:200:0	510	1.465
350	200:80:1000	150	1.469
350	100:40:2000	60	1.472
350	100:40:2000	60	1.464
350	100:40:2000	50	1.471
350	100:40:2000	80	1.471
350	100:40:2000	80	1.471
350	100:40:2000	60	1.471
275	100:40:2000	60	1.474

This data collected from [4].

Conditions :

Temperature : see above

Pressure : 1 Torr

Flow Rates : see above

Rf Power : 25 W (0.03 W cm⁻²)

Rf Freq. : 13.56 MHz

- Refractive Index dependent upon Deposition Temperature, Pressure, Rate, and Annealing.

<u>Deposition Temperature, ° C</u>	<u>Deposition Pressure</u>	<u>Deposition Rate, Å/min</u>	<u>Anneal Performed</u>	<u>Refractive Index</u>
350	1 Torr	60	No	1.471 (+/- 0.001)
350	1 Torr	60	Yes	1.463 (+/- 0.002)
275	1 Torr	60	No	1.473 (+/- 0.002)
275	1 Torr	60	Yes	1.463 (+/- 0.002)
350	1 Torr	520	No	1.467 (+/- 0.004)
350	1 Torr	520	Yes	1.463 (+/- 0.003)
700	1 atm	50	No	1.444 (+/- 0.001)
700	1 atm	50	Yes	1.454 (+/- 0.001)

Data collected from reference N° 4.

Deposition Conditions

Temperature : see above

Pressure : see above

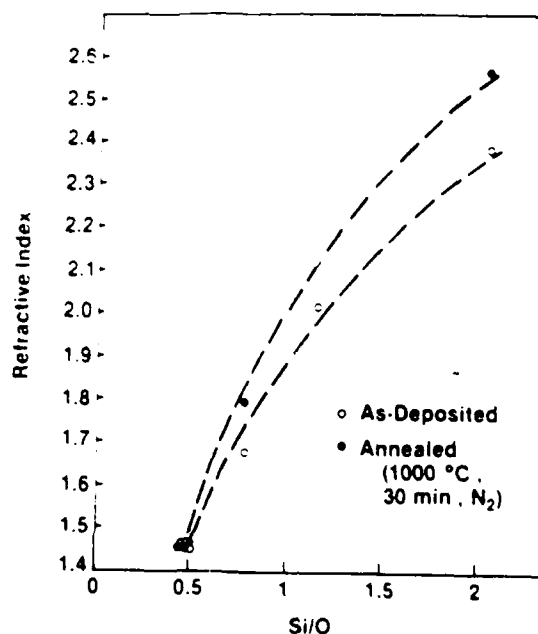
N₂O / SiH₄ ratio : 125 (for atmosphere pressure deposition only)

Rf frequency : 13.56 MHz.
 Rf Power : 25W (0.03 W cm⁻²)

Annealing Conditions

Temperature : 1000° C
 Ambient : N₂
 Duration : 30 min.

- 1.461 - 1.465 for oxide prepared at 500° C, 10 mTorr, Rf frequency of 0.5-3.0 MHz, and Rf Power of 1 kW. Gas data : n/a. [16]
- Refractive index, before and after annealing for PECVD oxide.



This data reproduced from [20]

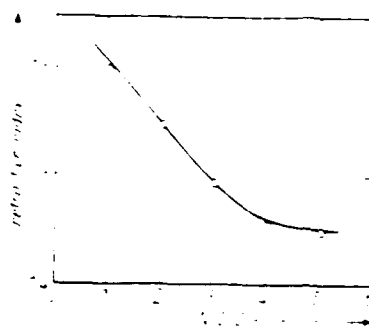
Deposition Conditions

Reagents: SiH₄, O₂, Ar
 Rf frequency : 13.562 MHz
 Rf Power : 50 W
 Temperature : 350° C
 Pressure : 1.5 Torr
 SiH₄ flow : 0.3 sccm
 O₂ flow rate : variable
 Substrate : Al.

Annealing Conditions :

30 min. in N₂, at 1000° C.

- Refractive index as it varies with reagent gas ratio, for PECVD oxide.



Graph reproduced from [25]

Conditions

Temperature : n/a

Pressure : n/a

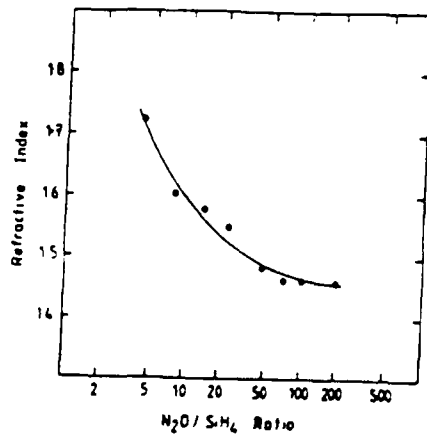
Rf frequency : 13.56 MHz

Rf Power : n/a

Substrate : Si wafer

Gas ratio : variable.

- Refractive index vs. gas ratio for photo-enhanced CVD SiO_2 .



Graph reproduced from [26].

Conditions :

SiH_4 flow rate : 1 sccm

N_2O flow rate : 70 sccm

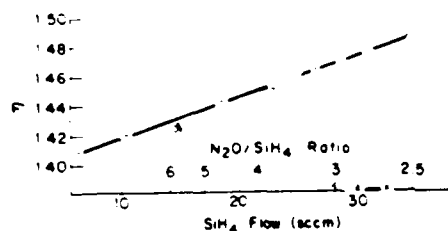
Pressure : 1 mbar

Temperature : 275°C

Power density : 0.1 mW cm^{-2}

N_2 flow rate : 30 sccm.

- Refractive index vs. gas ratio for glow-discharge deposited SiO_2 .



Graph reproduced from [28].

Conditions:

Temperature : $<40^\circ\text{C}$

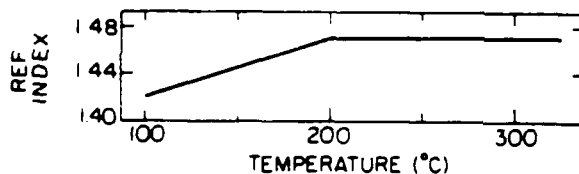
Pressure : 45 mTorr

Rf frequency : 13.56 MHz

Power Density : $0.2\text{--}0.5\text{ W cm}^{-2}$

Reagents : N_2O and SiH_4 .

- Refractive index vs. deposition temperature for PECVD SiO_2 .



Reproduced from [29]

Conditions:

Temperature : variable

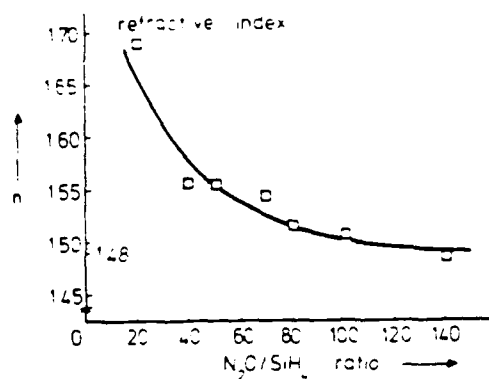
Pressure : 1 Torr

Gas Ratio, $\text{N}_2\text{O} / \text{SiH}_4$: 6.5

Rf frequency : 13.56 MHz

Rf Power : 24W

• Refractive index vs. gas ratio for PECVD SiO₂.



Reproduced from [31].

Conditions :

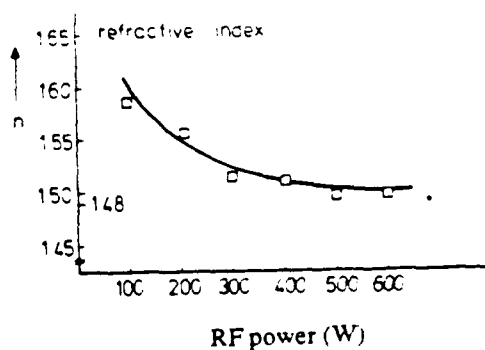
Temperature : 300° C

Pressure : 53 Pa

Rf frequency : 57 kHz

Rf Power Density : 0.05 W cm⁻².

• Refractive index vs. rf power, for PECVD SiO₂.



Reproduced from [31].

Conditions :

Temperature : 300° C

Pressure : 53 Pa

Rf frequency : 57 kHz

Gas Composition, N₂O : 98%, SiH₄ : 2%.

10.3 Bulk Oxide

1.46 for bulk SiO_2 . [33]

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