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Semiconductor Measurement Technology:

**The Results of an Interlaboratory Study
of Ellipsometric Measurements
of Thin Film Silicon Dioxide on Silicon**

Barbara J. Belzer and David L. Blackburn

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Barbara J. Belzer and David L. Blackburn

Semiconductor Electronics Division
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TABLE OF CONTENTS

	Page
Abstract	1
Key Words	1
Introduction	1
Sample Handling and Preparation	2
Results	3
Discussion	4
Conclusion	5
References	6

LIST OF TABLES

	Page
1 Sample #1019-01	8
2 Sample #1019-02	11
3 Sample #1017-20	14
4 Sample #1017-23	17
5 Sample #1004-14	20
6 Sample #1004-17	23
7 Summary of the Statistical Analysis as Performed by the ASTM E691 Program and as Performed Independently in the Spreadsheet	26

LIST OF FIGURES

	Page
1a Sample 1019-01 Thickness as Reported by the Participant	9
1b Sample 1019-01 Thickness as Calculated by NIST	9
1c Sample 1019-01 Measured Psi Values	10
1d Sample 1019-01 Measured Delta Values	10
2a Sample 1019-02 Thickness as Reported by the Participant	12
2b Sample 1019-02 Thickness as Calculated by NIST	12
2c Sample 1019-02 Measured Psi Values	13
2d Sample 1019-02 Measured Delta Values	13
3a Sample 1017-20 Thickness as Reported by the Participant	15
3b Sample 1017-20 Thickness as Calculated by NIST	15
3c Sample 1017-20 Measured Psi Values	16

LIST OF FIGURES (Cont'd.)

	Page
3d Sample 1017-20 Measured Delta Values	16
4a Sample 1017-23 Thickness as Reported by the Participant	18
4b Sample 1017-23 Thickness as Calculated by NIST	18
4c Sample 1017-23 Measured Psi Values	19
4d Sample 1017-23 Measured Delta Values	19
5a Sample 1004-14 Thickness as Reported by the Participant	21
5b Sample 1004-14 Thickness as Calculated by NIST	21
5c Sample 1004-14 Measured Psi Values	22
5d Sample 1004-14 Measured Delta Values	22
6a Sample 1004-17 Thickness as Reported by the Participant	24
6b Sample 1004-17 Thickness as Calculated by NIST	24
6c Sample 1004-17 Measured Psi Values	25
6d Sample 1004-17 Measured Delta Values	25

Semiconductor Measurement Technology:

**THE RESULTS OF AN INTERLABORATORY STUDY OF ELLIPSOMETRIC
MEASUREMENTS OF THIN FILM SILICON DIOXIDE ON SILICON**

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Abstract

The results of an interlaboratory study for the measurement of thin film oxides on silicon are presented. The study was performed by nine different laboratories using six different types of ellipsometers made by three different manufacturers. The purpose of the study was to establish a baseline of agreement between laboratories for single-wavelength ellipsometric thin-film measurements. The maximum standard deviation of the thickness calculated between laboratories using a common data reduction method was 0.22 nm for 10 nm oxides, 0.43 nm for 50 nm oxides, and 0.32 nm for 100 nm oxides.

Key words: ellipsometry; interlaboratory; metrology; silicon; silicon dioxide; single wavelength; thin films

Introduction

The interlaboratory study consisting of a series of controlled measurements on a selected set of SiO₂ on Si wafers was performed by nine different laboratories using six different types of ellipsometers made by three different manufacturers. The purpose of the study was to establish a baseline of agreement between laboratories for ellipsometric thin-film measurements. National Institute of Standards and Technology (NIST) measurements were made on the High-Accuracy Ellipsometer designed and built at NIST [1]. The measurements were made over a period of approximately 6 months, beginning in mid-August of 1993 and ending in mid-February 1994. The laboratories reported their measured ellipsometric parameters Δ and ψ , the angle of incidence (nominally 70° for all measurements), and the derived thickness of the silicon dioxide using the particular analysis technique usually employed by the laboratory and each laboratory's "preferred" material parameters. All measurements reported here were made at a wavelength of 633 nm. Included in this report are

the derived thicknesses using a common procedure and material parameters. The laboratories participating in the study were Rudolph Research (Flanders, NJ), IBM (Essex Junction, VT), Motorola, Inc. (Mesa, AZ), VLSI Standards (San Jose, CA), SEMATECH (Austin, TX), J.A. Woollam Co. (Lincoln, NE), Digital Equipment Corporation (Hudson, MA), Gaertner Scientific (Chicago, IL), and NIST (Gaithersburg, MD).

Sample Handling and Preparation

The study consisted of measurements made on two wafers each of three different nominal thicknesses (six wafers total) circulated in series from laboratory to laboratory. The nominal thicknesses were 100 nm, 50 nm, and 10 nm. After each laboratory was finished with its measurements, the samples were sent back to NIST where a brief series of measurements were done to assure that there were no significant changes or anomalies in the properties of the samples throughout the study. The only cleaning of the samples was performed at NIST using a procedure that is recommended for the NIST-produced SRMs consisting of an ethanol rinse and nitrogen blow dry, followed by a deionized water rinse and nitrogen blow dry. The samples were routinely cleaned after it was determined that no changes had occurred in the samples and before they were sent to the next participating laboratory. The other participating laboratories did not clean the wafers prior to their measurements. This procedure was used to eliminate any changes or anomalies that might be introduced into the samples due to different cleaning procedures at the different laboratories or due to other unforeseen occurrences. There were no significant changes in the samples observed over the duration of the study.

The oxides on the 10 nm and 50 nm samples were grown at 950 °C in dry oxygen and annealed in nitrogen for approximately 1 h at 1050 °C. The 100 nm oxides were grown at 1050 °C in dry oxygen. The anneal of the 10 nm and 50 nm oxides was done in an attempt to remove differences in the optical properties of the oxide and/or SiO₂-Si interface region due to the different growth temperatures. It was not the intent of this study, though, to investigate any such possible differences.

Each participating laboratory agreed to perform the measurements on the sample set according to the guidelines set forth as follows: The wafers were sent to each laboratory for measurement via overnight mail from NIST only after the participant had confirmed that the wafers could be measured within a day or two of receipt. About a one to one-and-a-half week total time frame for the "receipt-to-return" cycle was requested from each participant. The wafers were returned to NIST via overnight mail after each laboratory had completed its measurements. The wafers were *not to be cleaned by the receiving laboratory*. Four replicate measurements of *each* wafer, with removal and remount of the sample between replications, were to be made at the approximate center of the wafer. There were a total of 24 individual measurements requested from each participant for this study. Each laboratory recorded the following basic information: Δ , ψ , λ (633 nm), angle of incidence (70°), calculated thickness, optical constants (fixed and/or derived), and equipment type. All participants were encouraged to provide any ancillary information which was within the scope of their operation. Information suggested for inclusion were the calculated thickness and refractive

index of the film using the method and constants preferred by the participant and, if possible, the method and constants preferred by NIST, spectroscopic ellipsometry results (Δ , ψ , λ , and thickness calculations), and multiple angle and/or principal angle measurements. The individually preferred method is reported in the tables (As-Reported Thickness Values).

Results

The purpose of the study was to determine a baseline level of agreement in one-layer oxide thickness determinations between the nine participating laboratories using as tight a control as possible on the measurement procedure, sample preparations, and sample handling. The one-layer model of calculated oxide thickness based on ellipsometric measurements assumes a single, homogeneous layer of thermally grown silicon dioxide atop the silicon substrate. It is not to be implied that the measurement, model, or handling procedures that were employed are necessarily the best or yield the most accurate results for these measurements. The goal was to eliminate, as much as possible, variations in results due to differences in procedures and handling. For the sake of this study, NIST calculated individual thicknesses from the data presented by the participants using a math program and the standard ellipsometric equations.

The measured and calculated results for the single wavelength measurements are presented in the tables and graphs which appear at the end of the report. These results are discussed in the next section. Each of the six tables contains the data for one sample from all of the participating laboratories. The tables are divided into sections which list the derived thickness (*As-Reported*) using each laboratory's *preferred* procedure and constants, the derived thickness (*NIST Calculated*) using a common analysis procedure and common, fixed, optical constants for the SiO_2 ($n = 1.461$) and Si ($n = 3.875$, $k = 0.0156$), and the measured ellipsometric parameters Δ and ψ reported by each laboratory, except for lab F which employed an ellipsometer for which individual Δ and ψ values are not indicated to the instrument user.

Also included in Tables 1 through 6 are the average and the standard deviation of each laboratory's set of four measurements on each sample, and the "grand" average and standard deviation of all laboratory average values on each sample. The average and standard deviation values that are highlighted for a few samples for a few laboratories are values that lie far enough from the values of the other laboratories that the data "may require further investigation" according to ASTM Standard Practice E 691 [2]. A cursory examination of these values was carried out, and it was decided to keep all of them for the final analysis. The data in the tables are plotted in Figures 1 through 6, where each laboratory's derived or measured value is shown as a data point, the laboratory's *average* values are connected by a line, and the grand average (*GA*) and $GA \pm 3s$ are shown.

Table 7 summarizes the statistical analysis as performed by the ASTM E 691 Interlaboratory Data Analysis Software [3] and as performed in the spreadsheet used to tabulate the data. The column headings in this table are defined as follows:

<u>Column Heading</u>	<u>Column Contents</u>
Specimen ID	Sample Serial Number
t nom (nm)	Nominal thicknesses expressed in nanometers
Method	Thickness calculation method, i.e., participant vs. NIST's <i>Standard</i> method
t GA (nm)	Thickness Grand Average (ASTM E 691) average of the cell averages
s (nm)	Standard deviation in nanometers of the cell averages as determined from the spreadsheet
Sr	Within-laboratory <i>Repeatability</i> standard deviation (ASTM E 691)
SR	Between-laboratory <i>Reproducibility</i> standard deviation (ASTM E 691)
r (2.8 × Sr)	Repeatability limits, 95% probability (ASTM E 691)
R (2.8 × SR)	Reproducibility limits, 95% probability (ASTM E 691)

Discussion

Tables 1 through 6 summarize each laboratory's reported data. In the following discussion, it should be remembered that this study was undertaken only to determine the baseline agreement between laboratories and not to determine accuracy or preference of measurement methods.

Laboratory B was unable to complete the measurements as originally agreed upon. They were able to complete only three replications on one wafer of each nominal thickness using single-wavelength ellipsometry. A fourth result which was taken from spectroscopic measurements was also provided. The information listed in the tables for Laboratory B is taken from the reported single-wavelength data, with the fourth point being the average of the other three. While this is not as prescribed and almost certainly affected the reproducibility statistic for the study, it was felt that the measurement could not be discounted entirely.

An equipment problem affected the outcome of the measurements for Laboratory D, especially for Sample #1019-01. This participant was unable to use the ellipsometer specified for calibrations and careful scientific procedure, but did have access to one installed in a fabrication environment. Thus, the ellipsometer was not optimized for the type of flexibility necessary for a best scientific effort in that its calibration and operational control were not under the direct control of the individual participant and was therefore, not necessarily known.

Laboratory H used the wavelength, $\lambda = 633$, as selected from a monochromator while all others used a helium-neon laser source for their measurements.

As can be seen in Table 7, agreement between the laboratories generally improves slightly when a common or standard procedure is used in calculating the layer thickness, especially for the 10 nm SiO₂. The repeatability limit, r , is described as the 95% probability limit for each laboratory's measurement repeatability. The r value ranges from a minimum of 0.05 nm for one of the 100 nm samples to 0.25 nm for a 50 nm sample. The reproducibility limit, R , is the 95% probability limit for the reproducible measurement results between all the participants in this study. The R value ranges from 0.51 nm for a 10 nm sample to 1.22 nm for a 50 nm sample. Overall, the calculation method used to analyze the ellipsometry data does not seem to affect the r value and affects the R value only slightly.

The thicknesses that are calculated are dependent upon the method used. The grand average (GA) changes by an average of 0.14 nm (thicker) when using the “standard” or common method. It is not within the scope of this report to give any further interpretation regarding the calculation method other than that differences in the optical constants used significantly affect the calculated thickness.

The standard deviation of the thickness calculation between laboratories when using the individual laboratory's preferred method ranges from a minimum of 0.29 nm for a 10 nm oxide to a maximum of 0.44 nm for a 50 nm sample. The standard deviation of the thickness calculation between laboratories using a common or *standard* method produces standard deviations which range from a minimum of 0.18 nm for a 10 nm oxide to a maximum of 0.43 nm for one of the 50 nm oxides. The ranges for the standard deviation reported in a recent European-based study [4] using single wavelength ellipsometry (SWE) are from 0.93 nm and 0.99 nm for 10 nm oxides to 0.54 and 0.58 for their 120 nm samples. The spectroscopic ellipsometry results from that study had standard deviations in the calculated thickness from 0.54 nm and 0.74 nm for the 10 nm samples, and 0.48 nm and 0.50 nm for the 50 nm oxides, to 0.29 nm and 0.54 nm for the 120 nm artifacts. It is interesting to note that the European-based study showed a decreasing magnitude in standard deviation between labs as the oxide increased in thickness, whereas this study seems to show no discernible trend in either direction.

Conclusion

The study has demonstrated the within-laboratory and between-laboratory agreement that can be achieved for thin oxide on silicon measurements using single-wavelength ellipsometry. The good agreement that is demonstrated is likely influenced by the carefully controlled and monitored samples (particularly as regards their cleaning), the specific measurement instructions given each participant, and the selection of participants (calibration labs, instrument manufacturers, and others known to understand and make careful measurements). The next task should be to determine if this agreement can be extended into fabrication facilities where excellent tool-to-tool reproducibility is necessary.

The recent study done in Europe concluded that the “results of SWE cannot be trusted for silicon oxide thicknesses below 20 nm” [5]. The results of NIST’s interlaboratory study do not support that conclusion. This study shows that the magnitude in difference between thickness measurements

among laboratories does not need to degrade as oxide thickness decreases down to at least 10 nm. It demonstrates that single-wavelength ellipsometry can be used to reliably measure oxide thicknesses as thin as 10 nm if care is taken.

The Semiconductor Industry Association reported in 1994 in the Semiconductor Technology Workshop Working Group Reports (National Technology Roadmap for Semiconductors or NTRS) that process tolerance requirements for 9 nm gate oxides were ± 0.5 nm (3σ) in 1992 and ± 0.3 nm (3σ) for 6.5 nm gate oxides in 1995. The same report projected a need in 1998 for a ± 0.2 nm (3σ) process tolerance for 4.5 nm gate dielectrics [6]. To best compare these semiconductor-industry-stated requirements to the **between-laboratory agreement** found here, one should look at the **SR** and **R** columns of Table 7 for the 10 nm samples, *Std* (Method) row. The **SR** values are 0.18 nm and 0.23 nm, and the **R** values are 0.51 nm and 0.63 nm for the two 10 nm samples. To best compare the stated requirements to the **within-laboratory agreement** found here, one should look at the **Sr** and **r** columns of Table 7 for the 10 nm samples, *Std* (Method) row. The **Sr** values are 0.03 nm and 0.05 nm, and the **r** values are 0.08 nm and 0.15 nm.

The within-lab measurements here satisfy all the projected NTRS requirements through 1998 (assuming that the "absolute" measurement precision at 10 nm does not degrade upon going to 6 nm thicknesses). The between-lab measurements do not meet the NTRS requirements, with both 10 nm samples falling only slightly outside the 3σ limits stated for 9 nm gate oxides. Further work is needed on thinner samples to determine if this trend continues.

Note should be made of a round robin on oxide-thickness measurements by ellipsometry conducted by ASTM Committee F-1 several years ago [7]. There were 11 laboratories involved in that study, and the samples ranged in nominal thickness from 5 nm to 50 nm. The between-laboratory standard deviations in thickness (Table A 1.4 of reference 7) for that study were comparable to those found in this study.

References

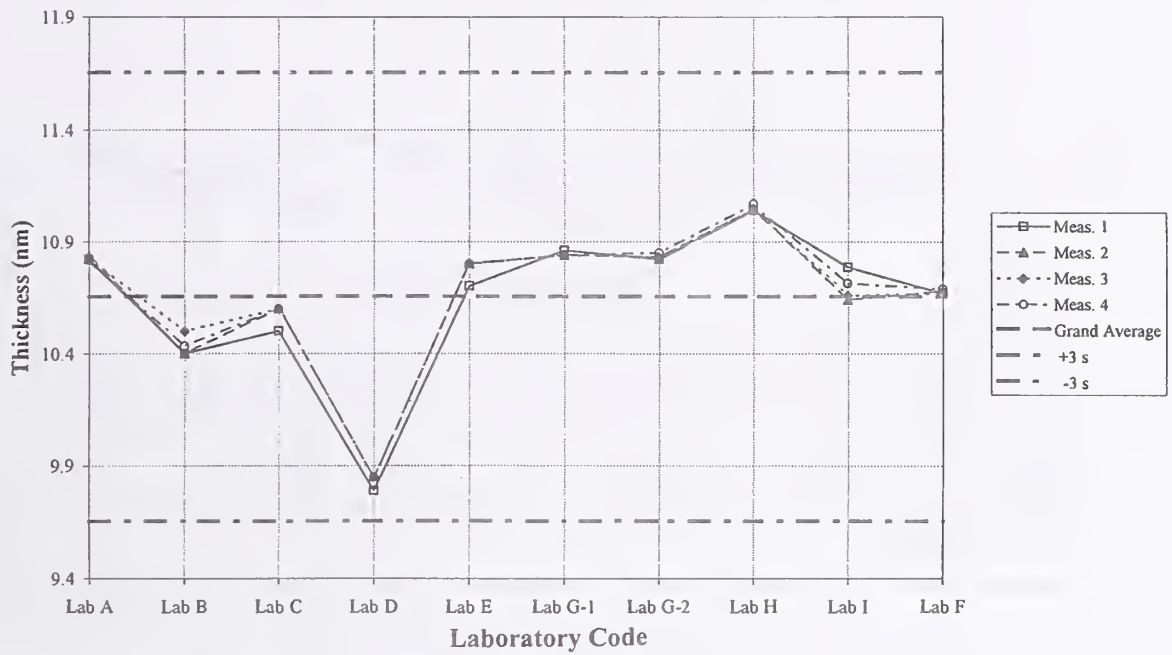
- [1] Candela, G.A. and Chandler-Horowitz, D., An Ellipsometry System for High Accuracy Metrology of Thin Films, Proc. Soc. Photo-Optical Instrum. Engrs., Integrated Circuit Metrology II 480, 2-8 (1984).
- [2] Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method, Designation: E 691-92, American Society for Testing and Materials Committee E-11, May 1992.
- [3] Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

- [4] Vanhellemont, J., et al., Round Robin Investigation of Silicon Oxide on Silicon Reference Materials for Ellipsometry, Appl. Surface Sci., 63, 45-49 (1993).
- [5] Ibid., 50-51 (1993).
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- [7] Standard Test Method for Measurement of Insulator Thickness and Refractive Index on Silicon Substrates by Ellipsometry, Designation: F 576-90, American Society for Testing and Materials Committee F-1, November 1990.

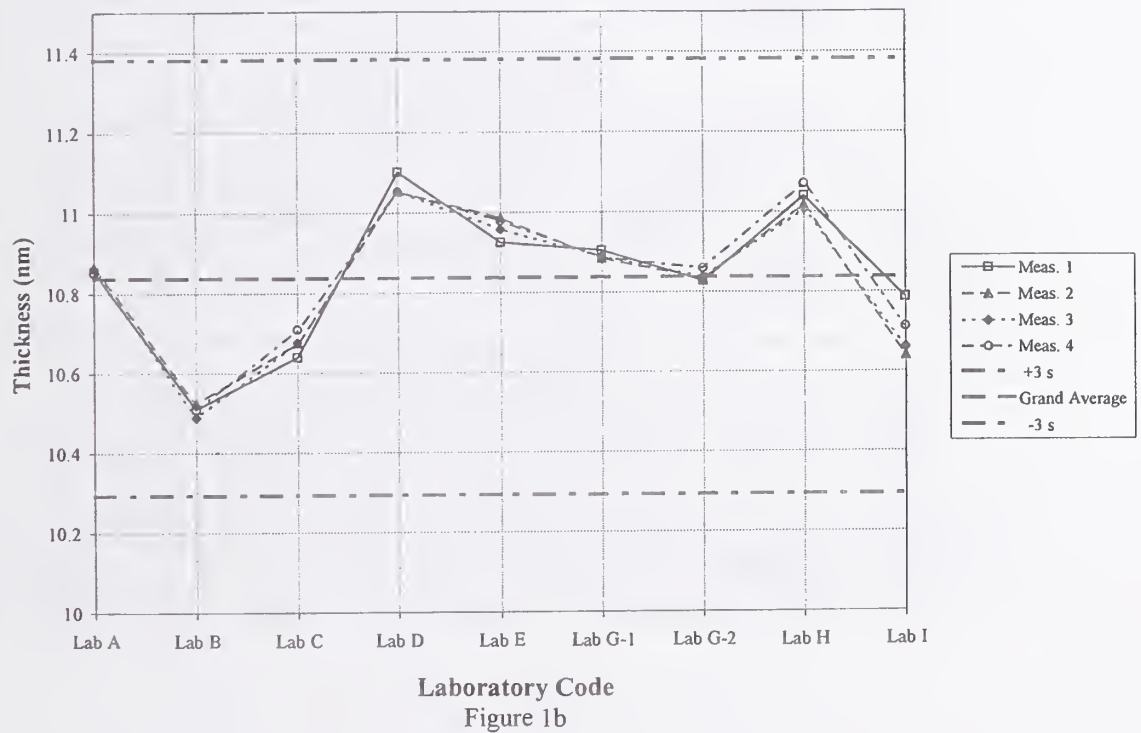
TABLE 1. SAMPLE #1019-01

	<i>Thickness (nm)</i>									
As Reported										
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	Lab F
	10.820	10.400	10.500	9.790	10.700	10.860	10.820	11.040	10.786	10.670
	10.836	10.400	10.600	9.850	10.800	10.840	10.830	11.050	10.640	10.680
	10.830	10.500	10.600	9.850	10.800	10.840	10.830	11.050	10.660	10.670
	10.817	10.433	10.600	9.850	10.800	10.840	10.850	11.070	10.713	10.690
Average	10.826	10.433	10.575	9.835	10.775	10.845	10.833	11.053	10.700	10.678
Std. Dev.	0.009	0.047	0.050	0.030	0.050	0.010	0.013	0.013	0.065	0.010
GrAvg/SD	10.655	0.333								
NIST Calculated (one-layer)	SiO2 n=1.461									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	10.856	10.509	10.640	11.102	10.926	10.905	10.827	11.040	10.786	
	10.867	10.524	10.675	11.053	10.986	10.885	10.832	11.018	10.640	
	10.864	10.489	10.677	11.053	10.958	10.891	10.837	11.006	10.660	
	10.851	10.508	10.709	11.053	10.979	10.887	10.859	11.071	10.713	
Average	10.860	10.508	10.675	11.065	10.962	10.892	10.839	11.034	10.700	
Std. Dev.	0.007	0.014	0.028	0.025	0.027	0.009	0.014	0.029	0.065	
GrAvg/SD	10.837	0.182								
	<i>Psi (degrees)</i>									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	11.512	11.500	11.540	11.400	11.550	11.512	11.538	11.500	11.509	
	11.518	11.470	11.500	11.400	11.510	11.517	11.539	11.480	11.524	
	11.514	11.520	11.540	11.400	11.510	11.517	11.541	11.470	11.548	
	11.510	11.497	11.540	11.400	11.520	11.520	11.544	11.460	11.520	
Average	11.513	11.497	11.530	11.400	11.523	11.517	11.541	11.478	11.525	
Std. Dev.	0.003	0.021	0.020	0.000	0.019	0.003	0.003	0.017	0.016	
GrAVG/SD	11.502	0.043								
	<i>Delta (degrees)</i>									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G2	Lab H	Lab I	
	149.858	150.700	150.380	149.240	149.680	149.728	149.921	149.400	150.020	
	149.823	150.660	150.290	149.360	149.530	149.778	149.909	149.450	150.378	
	149.830	150.750	150.290	149.360	149.600	149.763	149.897	149.480	150.331	
	149.860	150.703	150.210	149.360	149.550	149.773	149.844	149.320	150.198	
Average	149.843	150.703	150.293	149.330	149.590	149.761	149.893	149.413	150.232	
Std. Dev.	0.019	0.037	0.069	0.060	0.067	0.023	0.034	0.070	0.160	
GrAVG/SD	149.895	0.446								

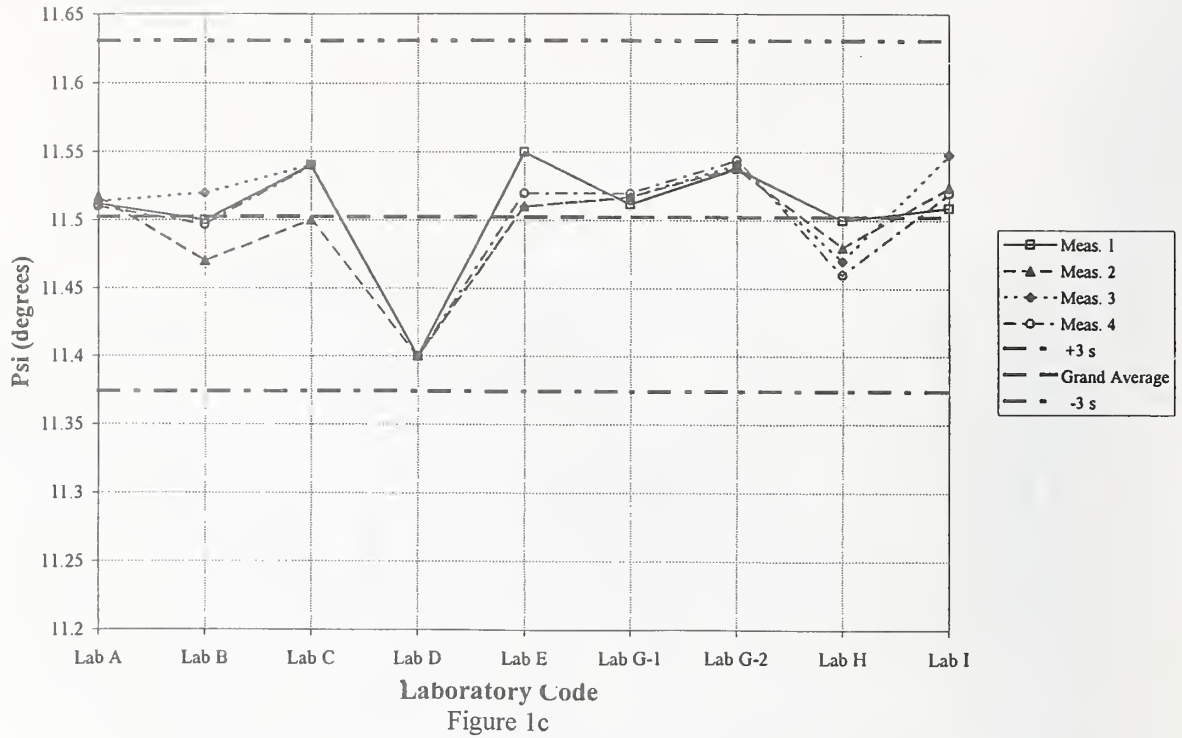
Sample 1019-01 Thickness as Reported by the Participant



Sample 1019-01 Thickness as Calculated by NIST



Sample 1019-01 Measured Psi Values



Sample 1019-01 Measured Delta Values

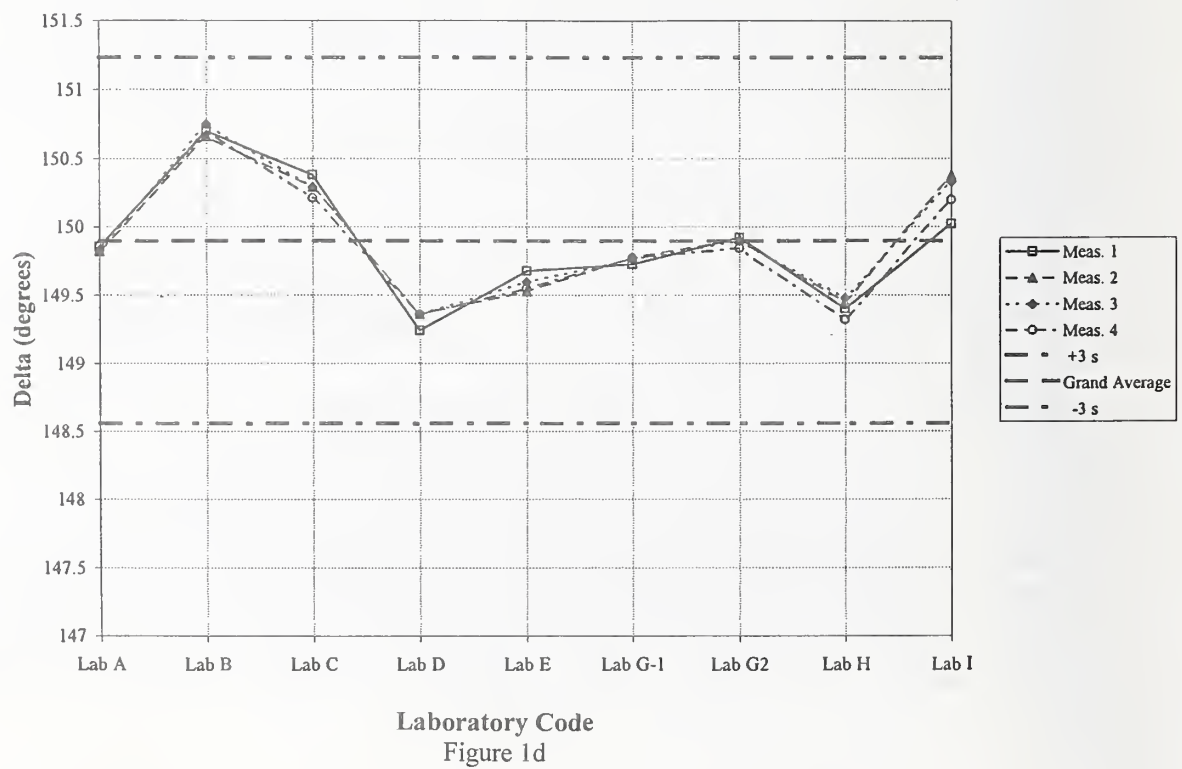
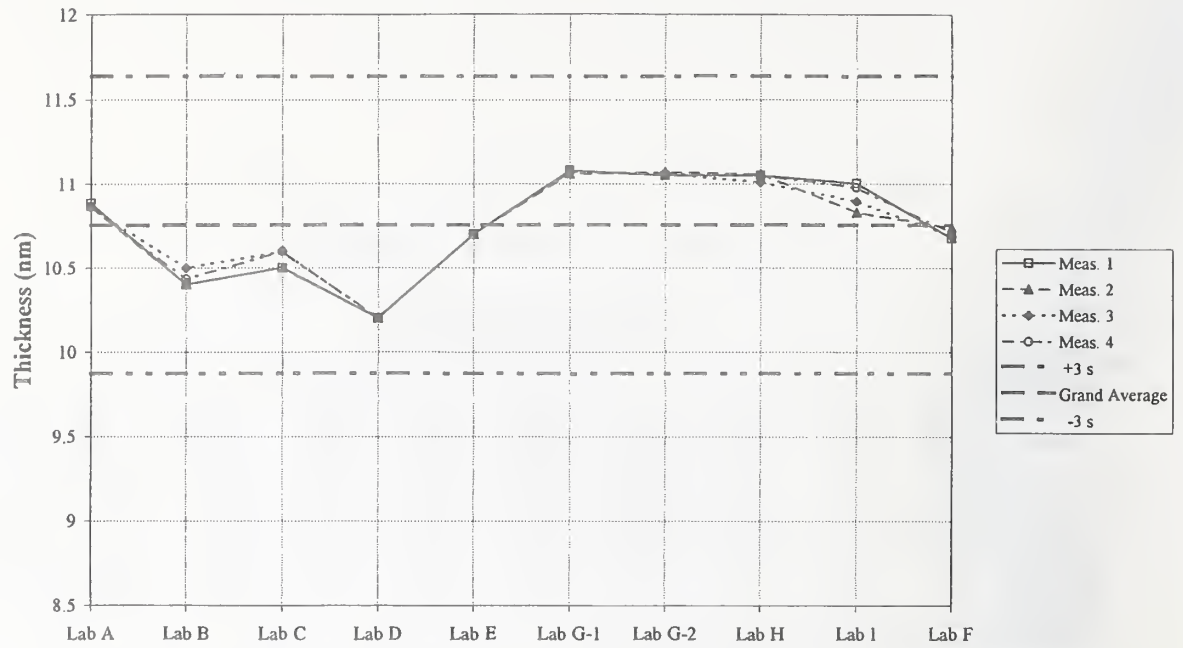


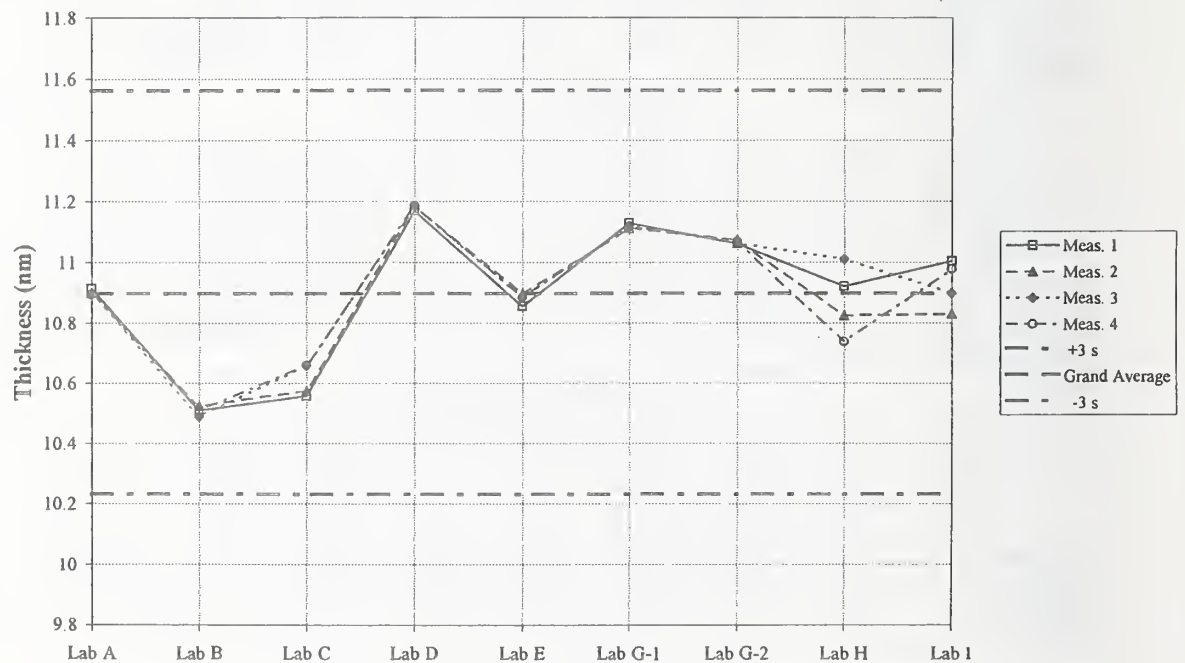
TABLE 2. SAMPLE #1019-02

	<i>Thickness (nm)</i>									
As-Reported	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	Lab F
	10.885	10.400	10.500	10.200	10.700	11.080	11.050	11.050	11.004	10.680
	10.870	10.400	10.500	10.200	10.700	11.060	11.070	11.060	10.830	10.740
	10.862	10.500	10.600	10.200	10.700	11.070	11.060	11.010	10.898	10.690
	10.866	10.433	10.600	10.200	10.700	11.070	11.060	11.050	10.979	10.710
Average	10.871	10.433	10.550	10.200	10.700	11.070	11.060	11.043	10.928	10.705
Std. Dev.	0.010	0.047	0.058	0.000	0.000	0.008	0.008	0.022	0.079	0.026
GrAvg/S	10.756	0.294								
NIST Calculated (one-layer)	SiO ₂ n = 1.461									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	10.914	10.509	10.557	11.169	10.855	11.129	11.061	10.921	11.004	
	10.899	10.524	10.574	11.186	10.897	11.112	11.074	10.824	10.830	
	10.894	10.489	10.659	11.186	10.884	11.121	11.064	11.011	10.898	
	10.894	10.508	10.660	11.186	10.885	11.118	11.069	10.737	10.979	
Average	10.900	10.508	10.613	11.182	10.880	11.120	11.067	10.873	10.928	
Std. Dev.	0.009	0.014	0.055	0.009	0.018	0.007	0.006	0.119	0.079	
GrAvg/S	10.897	0.222								
	<i>Psi (degrees)</i>									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	11.529	11.500	11.500	11.440	11.530	11.557	11.583	11.520	11.567	
	11.526	11.470	11.500	11.440	11.540	11.562	11.590	11.540	11.590	
	11.521	11.520	11.500	11.440	11.520	11.562	11.580	11.510	11.593	
	11.528	11.497	11.540	11.440	11.550	11.562	11.584	11.390	11.564	
Average	11.526	11.497	11.510	11.440	11.535	11.561	11.584	11.490	11.579	
Std. Dev.	0.003	0.021	0.020	0.000	0.013	0.003	0.004	0.068	0.015	
GrAvg/S	11.525	0.046								
1019-02	<i>Delta (degrees)</i>									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	149.708	150.700	150.580	149.080	149.780	149.188	149.355	149.690	149.491	
	149.745	150.660	150.540	149.040	149.750	149.228	149.324	149.930	149.917	
	149.758	150.750	150.330	149.040	149.780	149.208	149.347	149.470	149.752	
	149.758	150.700	150.330	149.040	149.780	149.213	149.336	150.130	149.553	
Average	149.742	150.703	150.445	149.050	149.773	149.209	149.341	149.805	149.678	
Std. Dev.	0.024	0.037	0.134	0.020	0.015	0.017	0.013	0.287	0.194	
GrAvg/S	149.749	0.542								

Sample 1019-02 Thickness as Reported by the Participant



Sample 1019-02 Thickness as Calculated by NIST



Sample 1019-02 Measured Psi Values

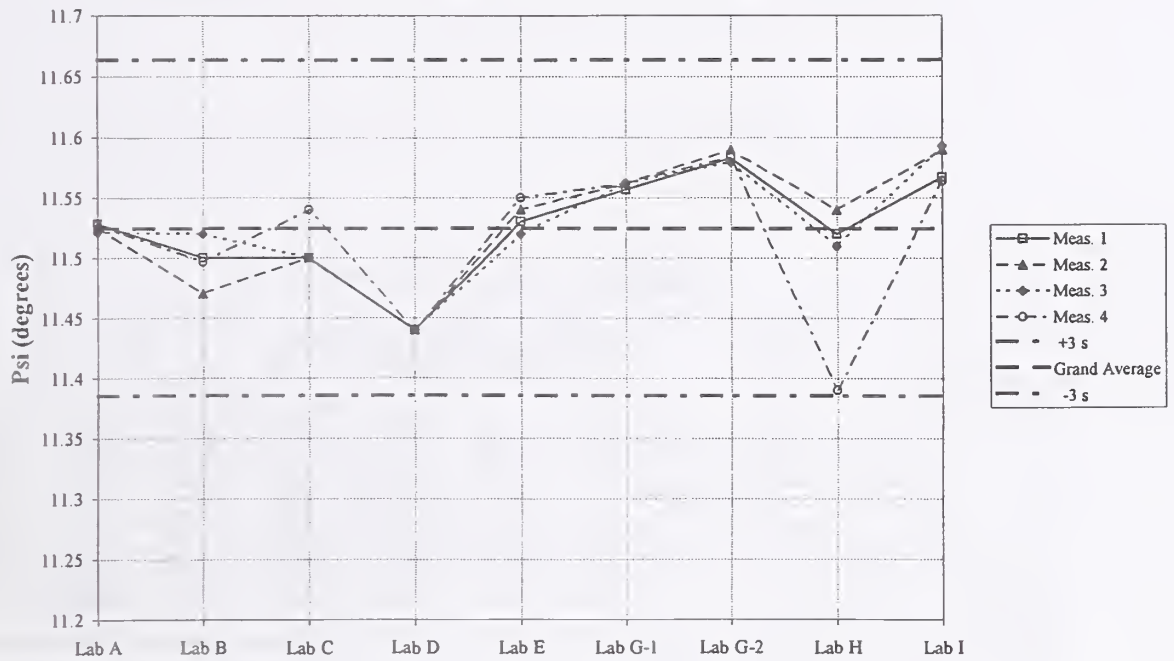


Figure 2c

Sample 1019-02 Measured Delta Values

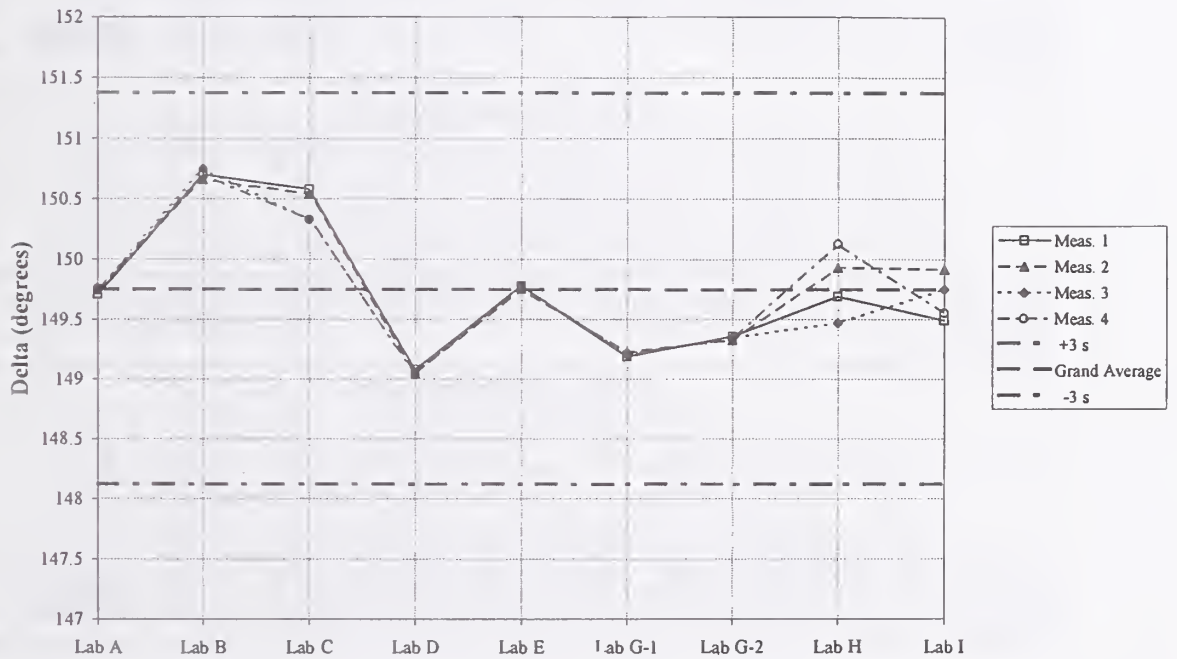
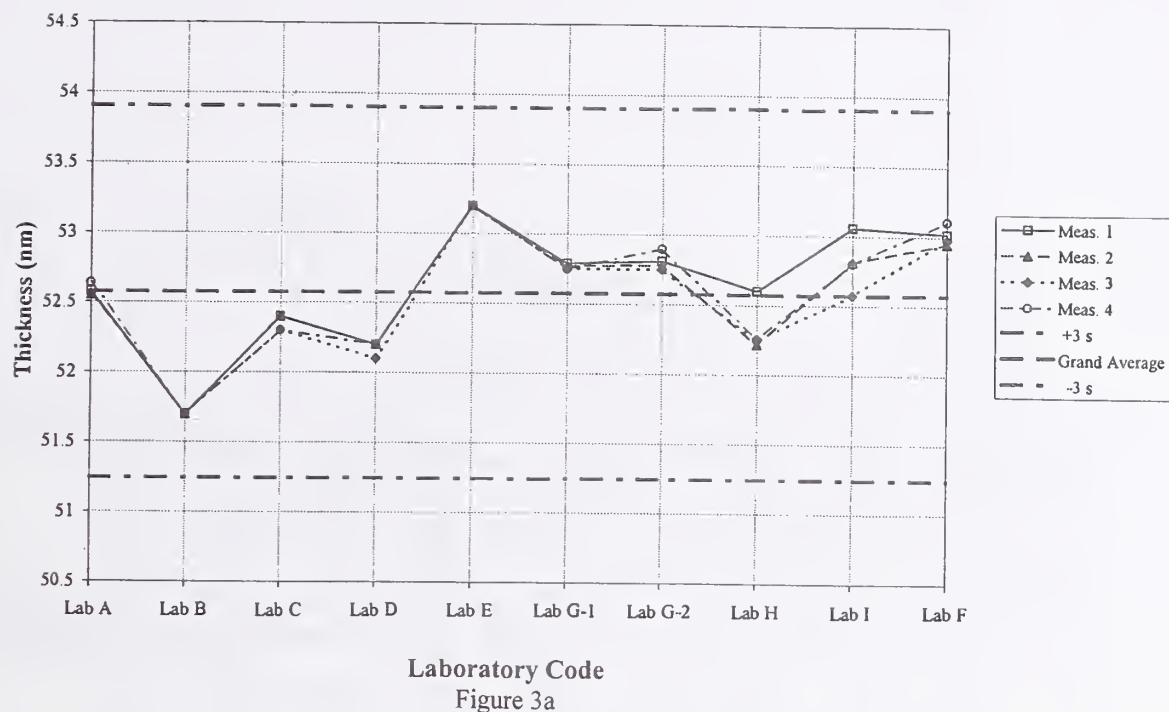


Figure 2d

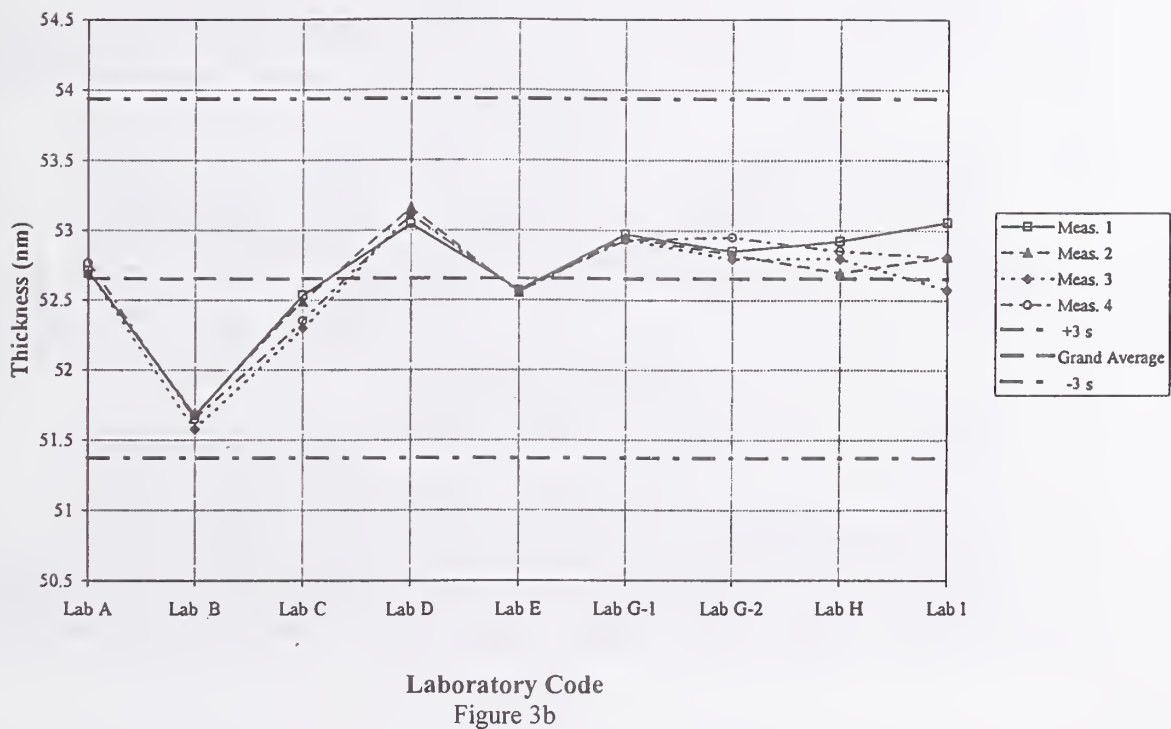
TABLE 3. SAMPLE #1017-20

<i>Thickness (nm)</i>										
As Reported										
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	Lab F
	52.574	51.700	52.400	52.200	53.200	52.790	52.810	52.600	53.059	53.020
	52.550	51.700	52.400	52.200	53.200	52.770	52.780	52.220	52.812	52.950
	52.557	51.700	52.300	52.100	53.200	52.750	52.750	52.250	52.577	52.980
	52.636	51.700	52.300	52.200	53.200	52.750	52.900	52.260	52.805	53.110
Average	52.579	51.700	52.350	52.175	53.200	52.765	52.810	52.333	52.813	53.015
Std. Dev	0.039	0.000	0.058	0.050	0.000	0.019	0.065	0.179	0.197	0.070
GrAVG/S	52.574	0.443								
NIST Calculated (one-layer) SiO₂ n = 1.461										
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	52.709	51.675	52.536	53.044	52.573	52.972	52.852	52.923	53.059	
	52.694	51.697	52.487	53.166	52.555	52.953	52.827	52.695	52.812	
	52.691	51.578	52.301	53.117	52.555	52.938	52.795	52.797	52.577	
	52.764	51.650	52.350	53.117	52.567	52.932	52.952	52.852	52.805	
Average	52.715	51.650	52.419	53.111	52.563	52.949	52.857	52.817	52.813	
Std. Dev	0.034	0.052	0.111	0.050	0.009	0.018	0.068	0.096	0.197	
GrAVG/S	52.655	0.428								
<i>Psi (degrees)</i>										
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	23.601	23.430	23.630	23.600	23.630	23.682	23.715	23.730	23.570	
	23.591	23.470	23.630	23.600	23.620	23.682	23.710	23.630	23.580	
	23.596	23.450	23.580	23.600	23.620	23.677	23.701	23.660	23.536	
	23.623	23.450	23.580	23.600	23.620	23.677	23.747	23.670	23.560	
Average	23.603	23.450	23.605	23.600	23.623	23.680	23.718	23.673	23.562	
Std. Dev	0.014	0.016	0.029	0.000	0.005	0.003	0.020	0.042	0.019	
GrAVG/S	23.621	0.078								
<i>Delta (degrees)</i>										
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	93.635	94.410	93.790	93.400	93.760	93.458	93.571	93.520	93.334	
	93.643	94.410	93.880	93.400	93.770	93.473	93.589	93.660	93.540	
	93.648	94.500	93.960	93.360	93.770	93.483	93.611	93.590	93.713	
	93.600	94.410	93.920	93.400	93.760	93.488	93.505	93.550	93.540	
Average	93.631	94.433	93.888	93.390	93.765	93.476	93.569	93.580	93.532	
Std. Dev	0.021	0.045	0.073	0.020	0.006	0.013	0.046	0.061	0.155	
GrAVG/S	93.696	0.314								

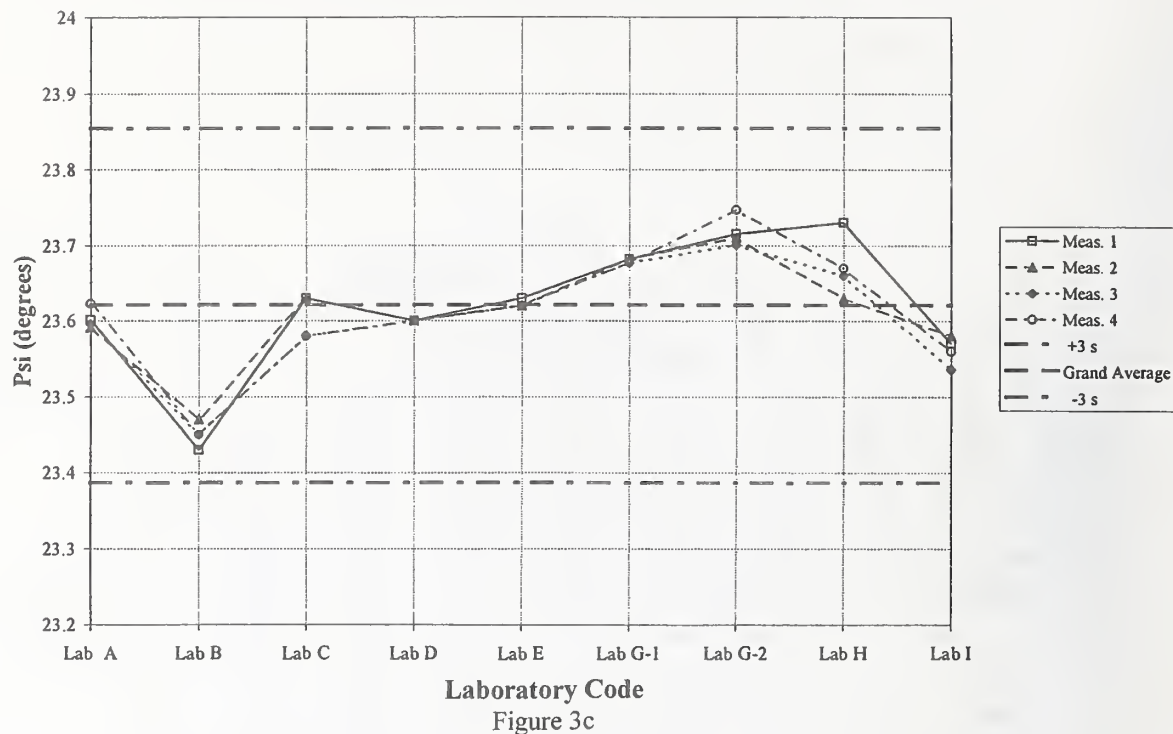
Sample 1017-20 Thickness as Reported by the Participant



Sample 1017-20 Thickness as Calculated by NIST



Sample 1017-20 Measured Psi Values



Sample 1017-20 Measured Delta Values

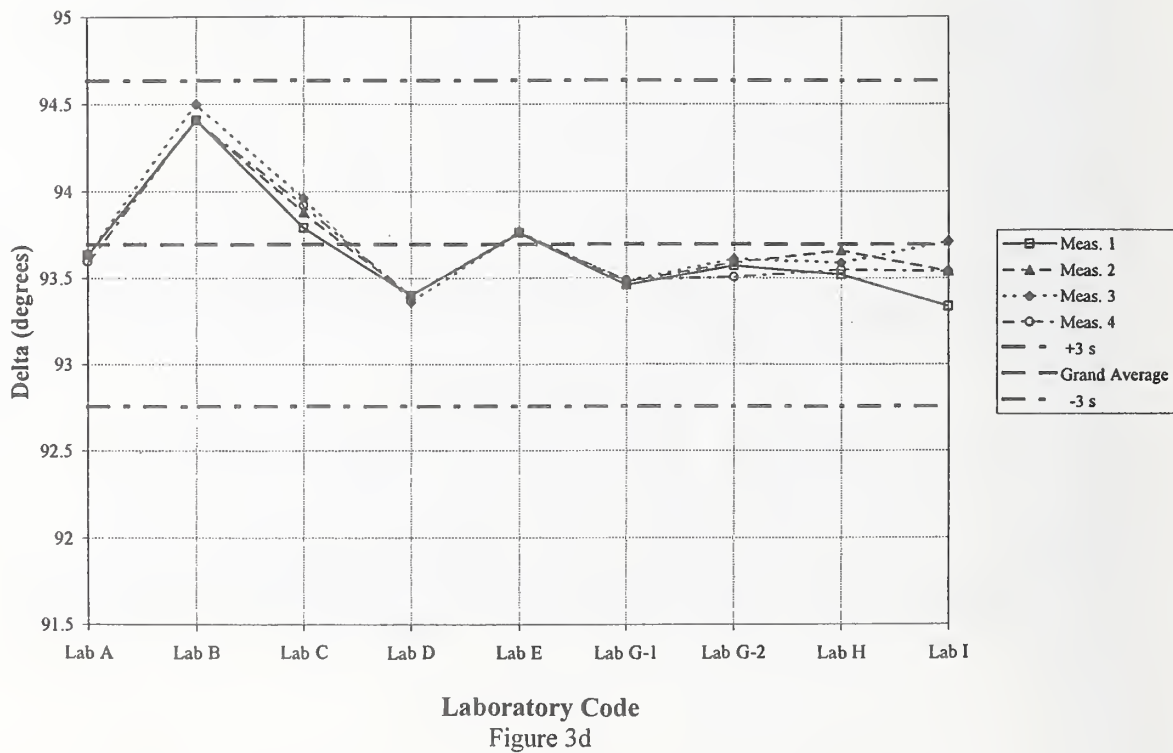
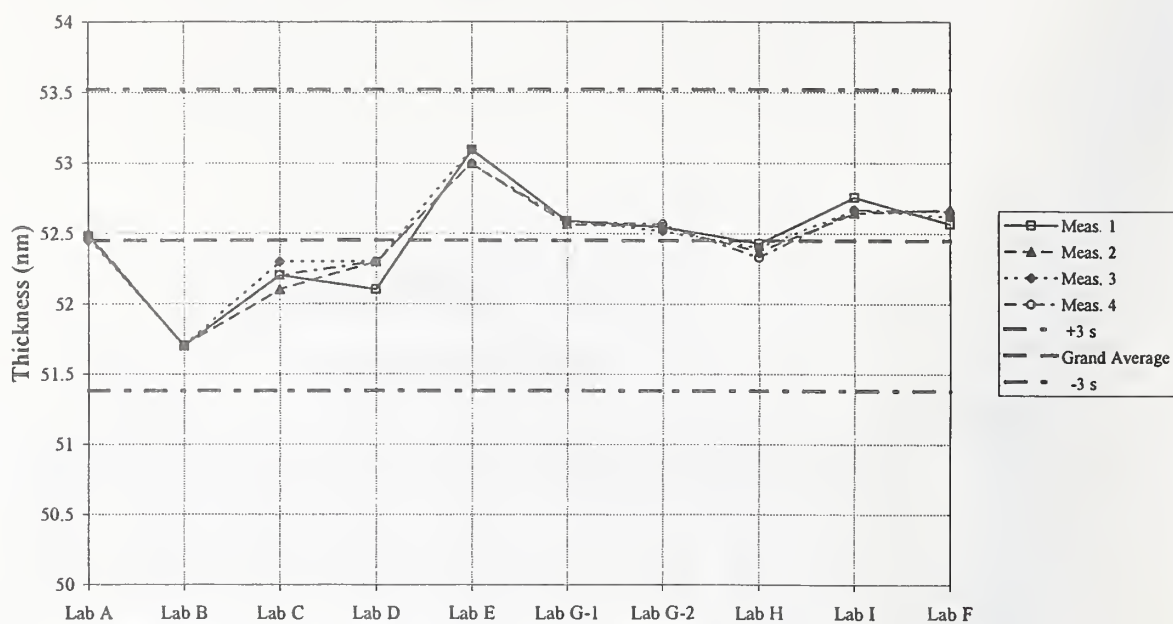


TABLE 4. SAMPLE #1017-23

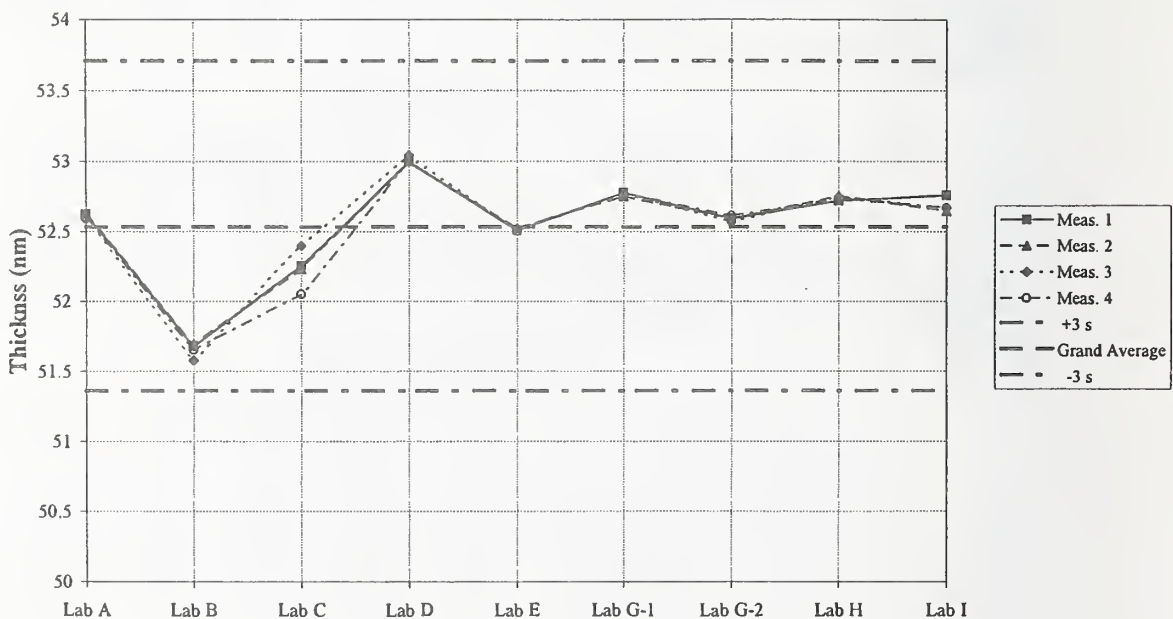
	<i>Thickness (nm)</i>									
As-Reported										
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	Lab F
	52.484	51.700	52.200	52.100	53.100	52.590	52.550	52.430	52.758	52.570
	52.496	51.700	52.100	52.300	53.000	52.570	52.550	52.370	52.646	52.670
	52.452	51.700	52.300	52.300	53.100	52.590	52.520	52.400	52.672	52.620
	52.480	51.700	52.200	52.300	53.000	52.590	52.570	52.330	52.670	52.660
Average	52.478	51.700	52.200	52.250	53.050	52.585	52.548	52.383	52.687	52.630
GrAvg/S	52.451	0.357								
NIST Calculated (one-layer)	SiO2 n = 1.461									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	52.625	51.675	52.253	52.995	52.507	52.775	52.594	52.718	52.758	
	52.628	51.697	52.230	52.995	52.526	52.752	52.592	52.755	52.646	
	52.589	51.578	52.398	53.044	52.507	52.772	52.570	52.748	52.672	
	52.597	51.650	52.051	52.995	52.517	52.775	52.614	52.737	52.670	
Average	52.610	51.650	52.233	53.007	52.514	52.769	52.593	52.740	52.687	
Std. Dev	0.020	0.052	0.142	0.024	0.009	0.011	0.018	0.016	0.049	
GrAvg/S	52.533	0.391								
	<i>Psi (degrees)</i>									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	23.571	23.430	23.580	23.600	23.630	23.617	23.631	23.650	23.554	
	23.578	23.470	23.540	23.640	23.620	23.620	23.636	23.650	23.540	
	23.563	23.450	23.580	23.640	23.620	23.622	23.631	23.660	23.543	
	23.576	23.450	23.500	23.640	23.620	23.627	23.634	23.640	23.537	
Average	23.572	23.450	23.550	23.630	23.623	23.622	23.633	23.650	23.544	
Std. Dev	0.007	0.016	0.038	0.020	0.005	0.004	0.002	0.008	0.007	
GrAvg/S	23.586	0.064								
	<i>Delta (degrees)</i>									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	93.690	94.410	94.000	93.360	93.800	93.588	93.743	93.650	93.563	
	93.690	94.410	94.000	93.280	93.780	93.608	93.747	93.620	93.657	
	93.715	94.500	93.880	93.320	93.800	93.593	93.763	93.630	93.638	
	93.715	94.440	94.130	93.320	93.790	93.593	93.728	93.630	93.635	
Average	93.703	94.440	94.003	93.320	93.793	93.596	93.745	93.633	93.623	
Std. Dev	0.014	0.042	0.102	0.033	0.010	0.009	0.014	0.013	0.041	
GrAvg/S	93.762	0.313								

Sample 1017-23 Thickness as Reported by the Participant



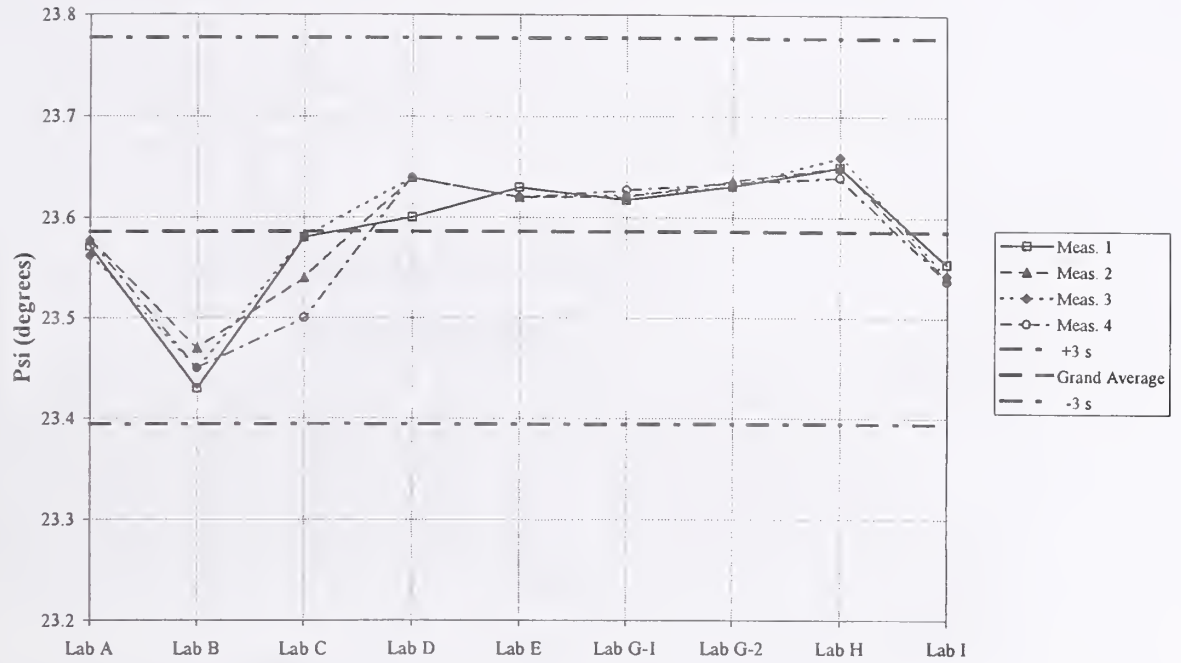
Laboratory Code
Figure 4a

Sample 1017-23 Thickness as Calculated by NIST



Laboratory Code
Figure 4b

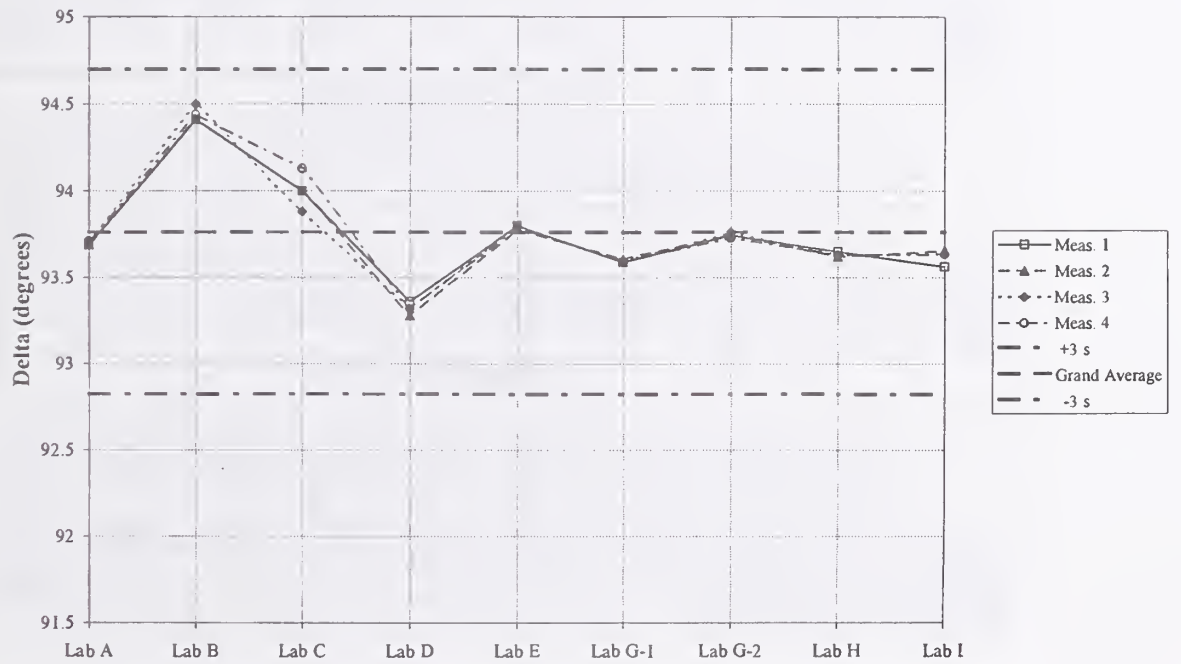
Sample 1017-23 Measured Psi Values



Laboratory Code

Figure 4c

Sample 1017-23 Measured Delta Values



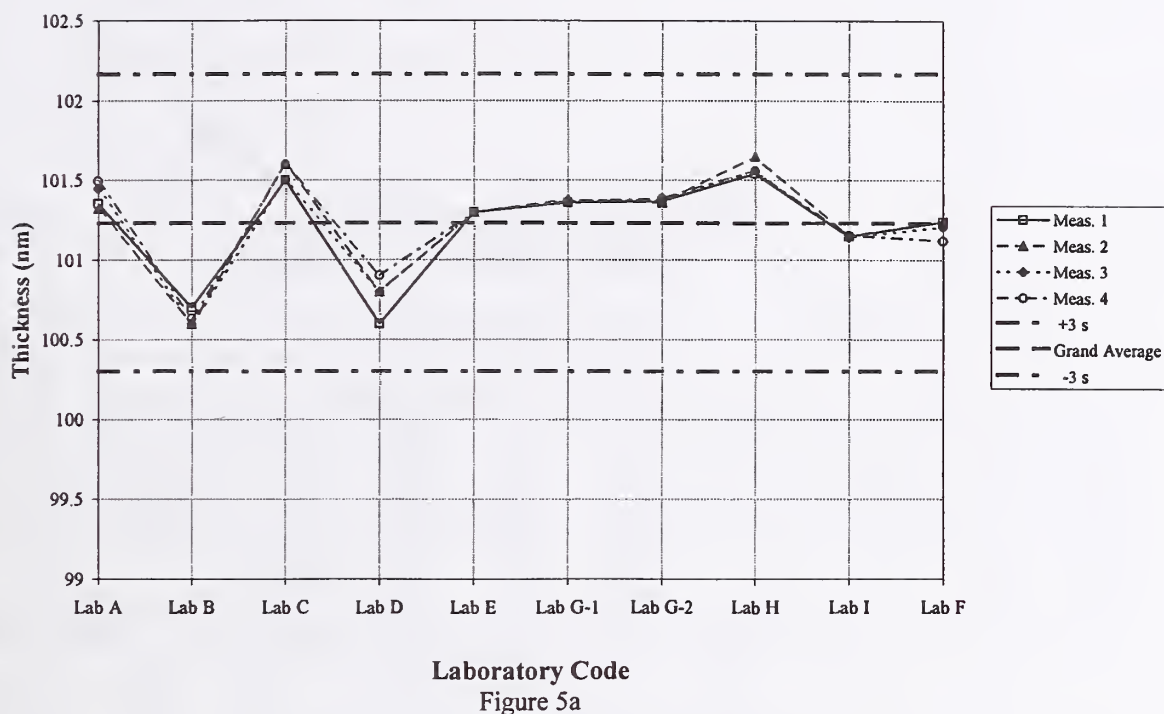
Laboratory Code

Figure 4d

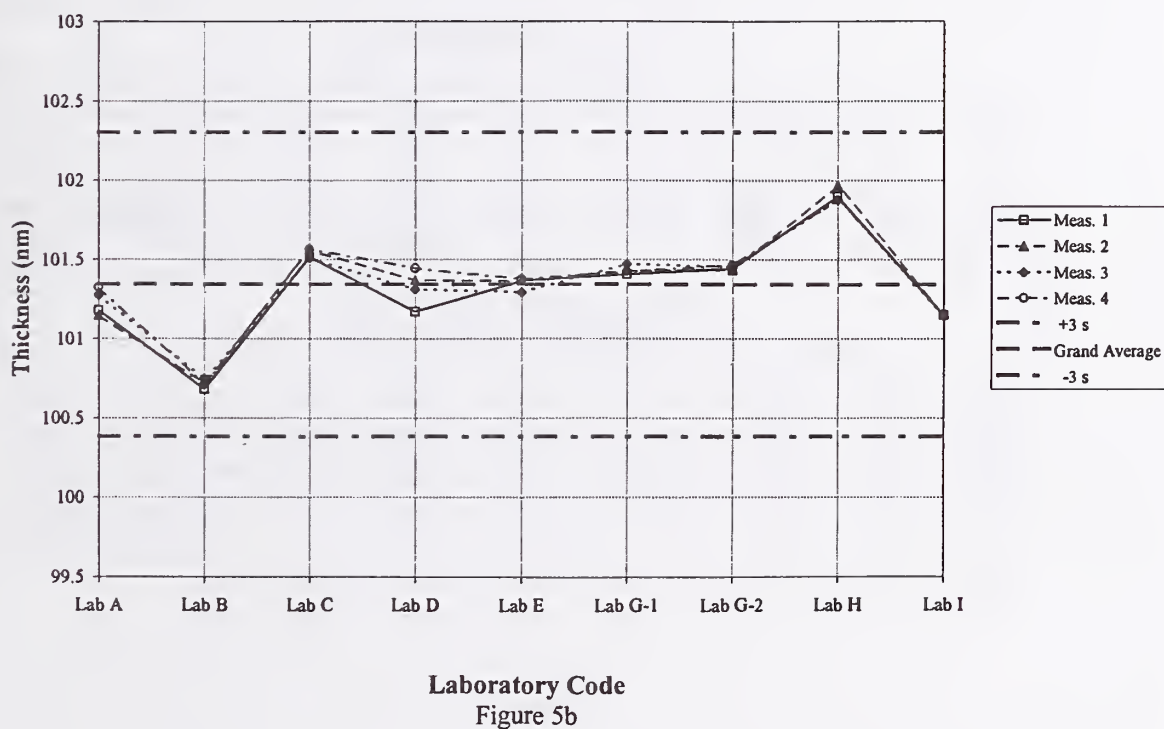
TABLE 5. SAMPLE #1004-14

	<i>Thickness (nm)</i>									
As-Reported										
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	Lab F
	101.356	100.700	101.500	100.600	101.300	101.360	101.370	101.540	101.149	101.240
	101.323	100.600	101.600	100.800	101.300	101.380	101.360	101.650	101.154	101.240
	101.448	100.600	101.500	100.800	101.300	101.360	101.380	101.560	101.145	101.210
	101.496	100.633	101.600	100.900	101.300	101.370	101.390	101.560	101.156	101.120
Average	101.406	100.633	101.550	100.775	101.300	101.368	101.375	101.578	101.151	101.203
Std. Dev	0.080	0.047	0.058	0.126	0.000	0.010	0.013	0.049	0.005	0.057
GrAvg/S	101.234	0.311								
NIST Calculated (one-layer)	SiO₂ n = 1.461									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	101.178	100.681	101.516	101.170	101.364	101.407	101.444	101.902	101.149	
	101.146	100.716	101.558	101.370	101.364	101.430	101.435	101.970	101.154	
	101.275	100.749	101.508	101.312	101.294	101.472	101.457	101.880	101.145	
	101.324	100.710	101.566	101.445	101.378	101.424	101.466	101.880	101.156	
Average	101.231	100.714	101.537	101.324	101.350	101.433	101.451	101.908	101.151	
Std. Dev	0.083	0.028	0.029	0.116	0.038	0.028	0.014	0.043	0.005	
GrAvg/S	101.344	0.321								
	<i>Psi (degrees)</i>									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	41.970	41.640	42.130	42.000	42.070	42.112	42.117	42.390	41.984	
	41.952	41.660	42.170	42.120	42.070	42.123	42.113	42.430	41.987	
	42.023	41.660	42.130	42.080	42.030	42.115	42.123	42.370	41.983	
	42.050	41.653	42.170	42.160	42.080	42.120	42.130	42.370	41.989	
Average	41.999	41.653	42.150	42.090	42.063	42.118	42.121	42.390	41.986	
Std. Dev	0.045	0.009	0.023	0.068	0.022	0.005	0.007	0.028	0.003	
GrAvg/S	42.063	0.193								
	<i>Delta (degrees)</i>									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	79.008	79.370	79.330	78.720	79.090	78.963	79.094	79.110	78.755	
	79.010	79.370	79.210	78.720	79.090	78.983	79.087	79.120	78.755	
	79.048	79.540	79.290	78.760	79.080	78.968	79.109	79.160	78.739	
	79.063	79.427	79.250	78.760	79.080	78.978	79.097	79.160	78.744	
Average	79.032	79.427	79.270	78.740	79.085	78.973	79.097	79.138	78.748	
Std. Dev	0.027	0.080	0.052	0.023	0.006	0.009	0.009	0.026	0.008	
GrAvg/S	79.057	0.222								

Sample 1004-14 Thickness as Reported by the Participant



Sample 1004-14 Thickness as Calculated by NIST



Sample 1004-14 Measured Psi Values

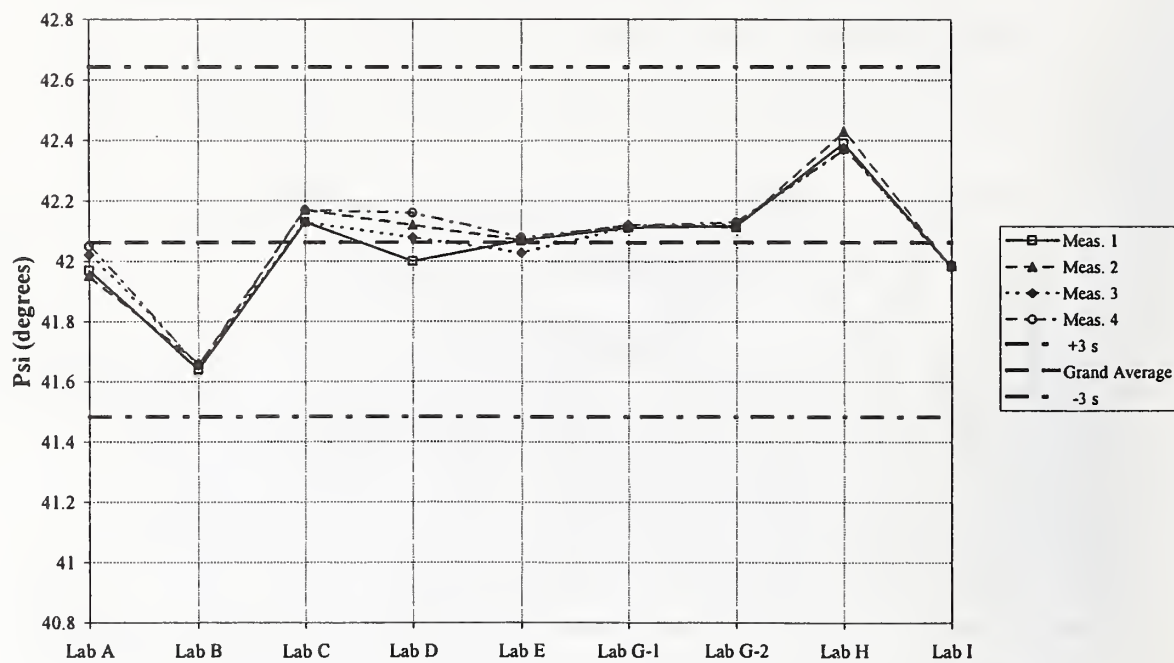


Figure 5c

Sample 1004-14 Measured Delta Values

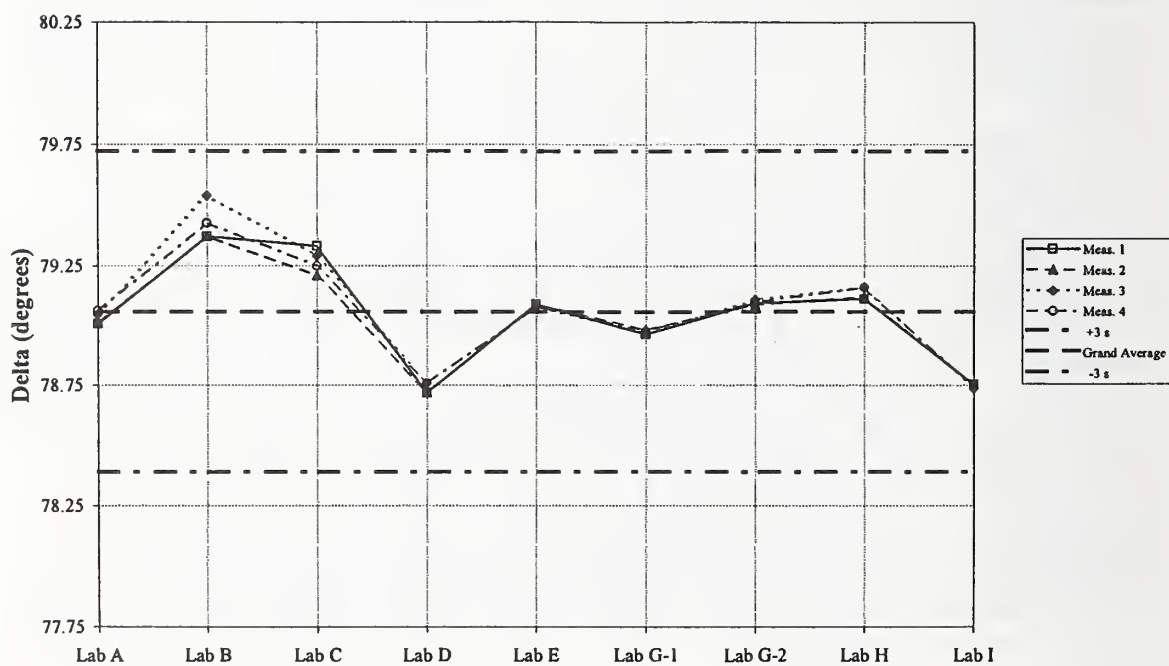
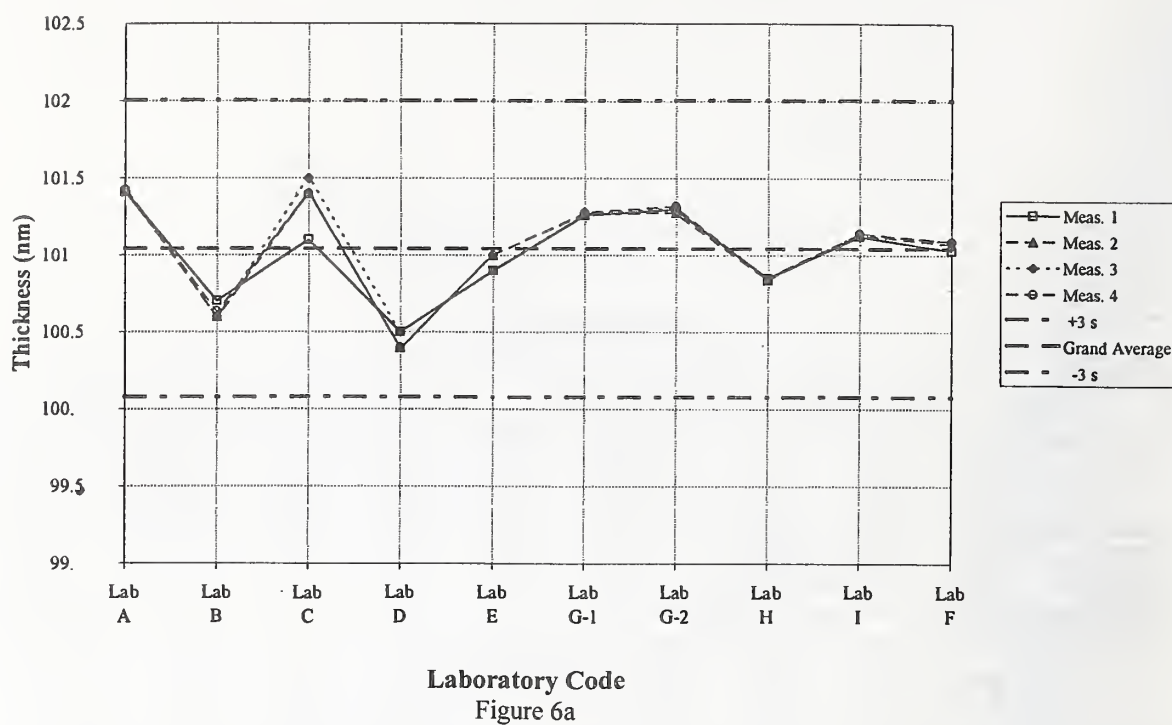


Figure 5d

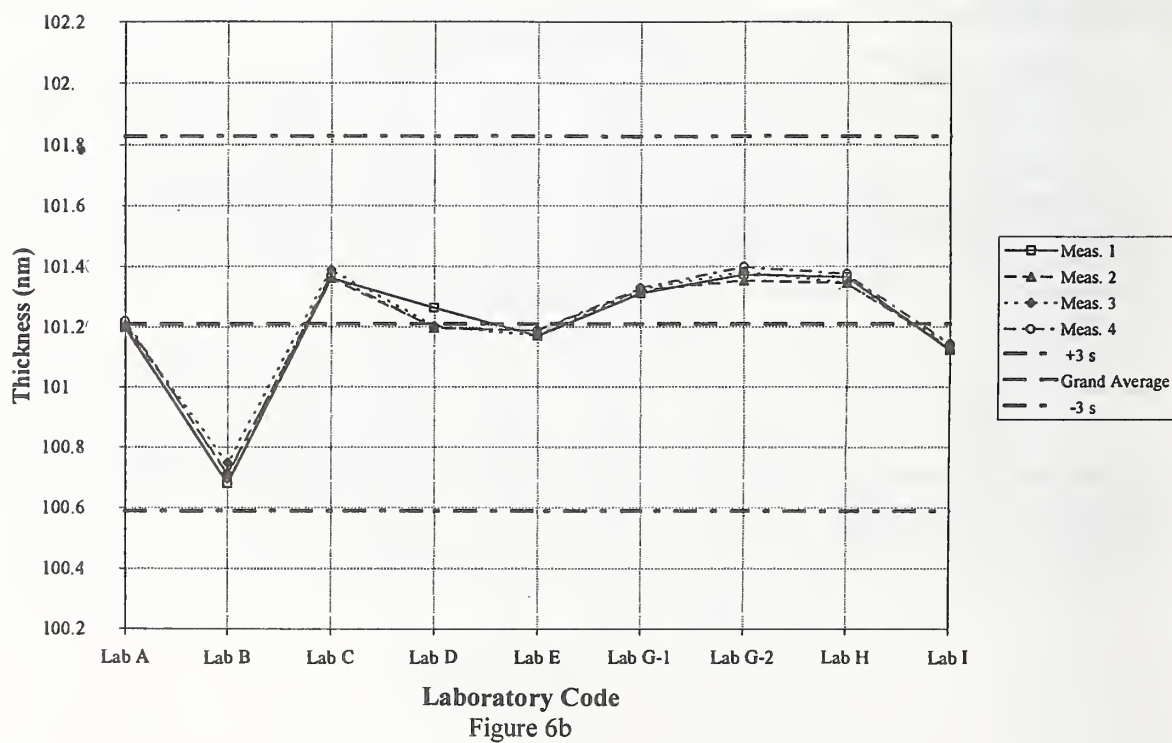
TABLE 6. SAMPLE #1004-17

	<i>Thickness (nm)</i>									
As-Reported										
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	Lab F
	101.409	100.700	101.100	100.500	100.900	101.260	101.300	100.850	101.122	101.030
	101.418	100.600	101.400	100.400	101.000	101.270	101.280	100.840	101.132	101.070
	101.412	100.600	101.500	100.500	100.900	101.270	101.310	100.850	101.143	101.090
	101.423	100.633	101.400	100.400	101.000	101.280	101.320	100.850	101.143	101.090
Average	101.416	100.633	101.350	100.450	100.950	101.270	101.303	100.848	101.135	101.070
Std. Dev.	0.006	0.047	0.173	0.058	0.058	0.008	0.017	0.005	0.010	0.028
GrAvg/SD	101.042	0.321								
NIST Calculated (one-layer)	SiO ₂ n= 1.461									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	101.198	100.681	101.361	101.262	101.169	101.311	101.373	101.364	101.122	
	101.207	100.716	101.361	101.196	101.187	101.321	101.354	101.345	101.132	
	101.204	100.749	101.389	101.204	101.171	101.324	101.382	101.345	101.143	
	101.219	100.710	101.370	101.196	101.187	101.329	101.399	101.376	101.143	
Average	101.207	100.714	101.370	101.215	101.179	101.321	101.377	101.358	101.135	
Std. Dev.	0.009	0.028	0.013	0.032	0.010	0.008	0.019	0.015	0.010	
GrAvg/SD	101.208	0.206								
	<i>Psi (degrees)</i>									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	42.005	41.640	42.080	42.080	41.980	42.077	42.099	42.090	41.989	
	42.010	41.660	42.080	42.040	41.990	42.082	42.089	42.080	41.995	
	42.006	41.660	42.080	42.040	41.980	42.085	42.102	42.080	42.000	
	42.013	41.653	42.080	42.040	41.990	42.087	42.112	42.100	41.998	
Average	42.008	41.653	42.080	42.050	41.985	42.083	42.101	42.088	41.996	
Std. Dev.	0.003	0.009	0.000	0.020	0.006	0.004	0.009	0.010	0.005	
GrAvg/SD	42.005	0.139								
	<i>Delta (degrees)</i>									
	Lab A	Lab B	Lab C	Lab D	Lab E	Lab G-1	Lab G-2	Lab H	Lab I	
	78.818	79.370	79.000	78.520	78.880	78.783	78.905	78.930	78.571	
	78.823	79.370	79.000	78.520	78.890	78.788	78.891	78.920	78.571	
	78.838	79.540	79.130	78.560	78.890	78.778	78.922	78.920	78.586	
	78.860	79.427	79.040	78.520	78.890	78.788	78.921	78.910	78.603	
Average	78.834	79.427	79.043	78.530	78.888	78.784	78.910	78.920	78.583	
Std. Dev.	0.019	0.080	0.061	0.020	0.005	0.005	0.015	0.008	0.015	
GrAvg/SD	78.880	0.262								

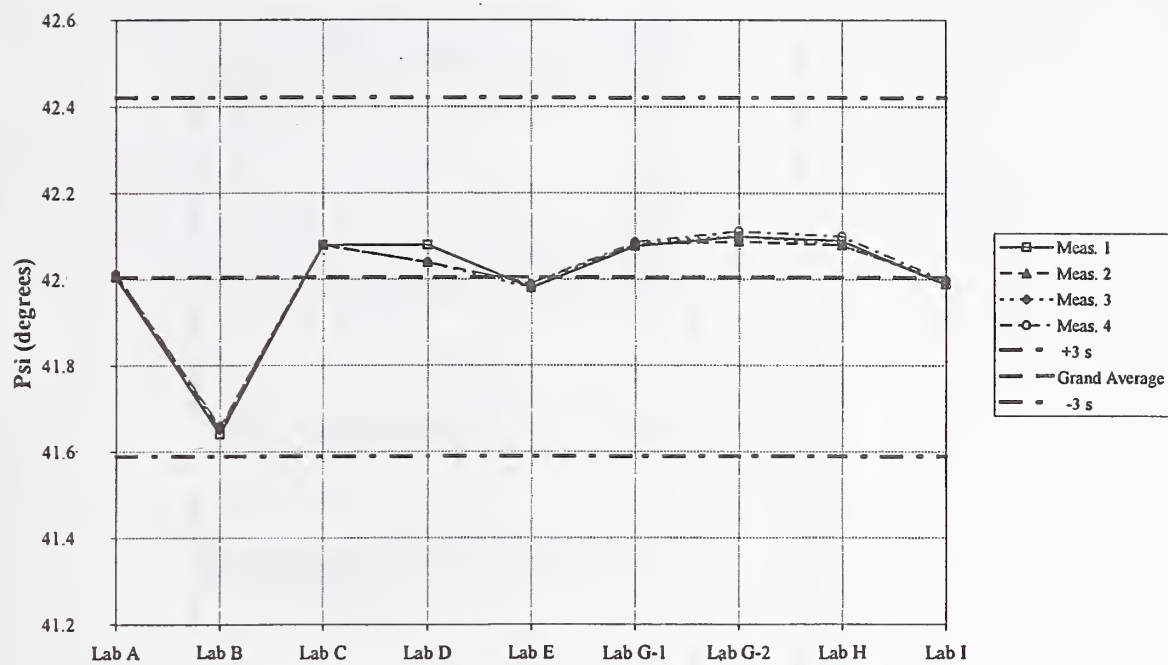
Sample 1004-17 Thickness as Reported by the Participant



Sample 1004-17 Thickness as Calculated by NIST

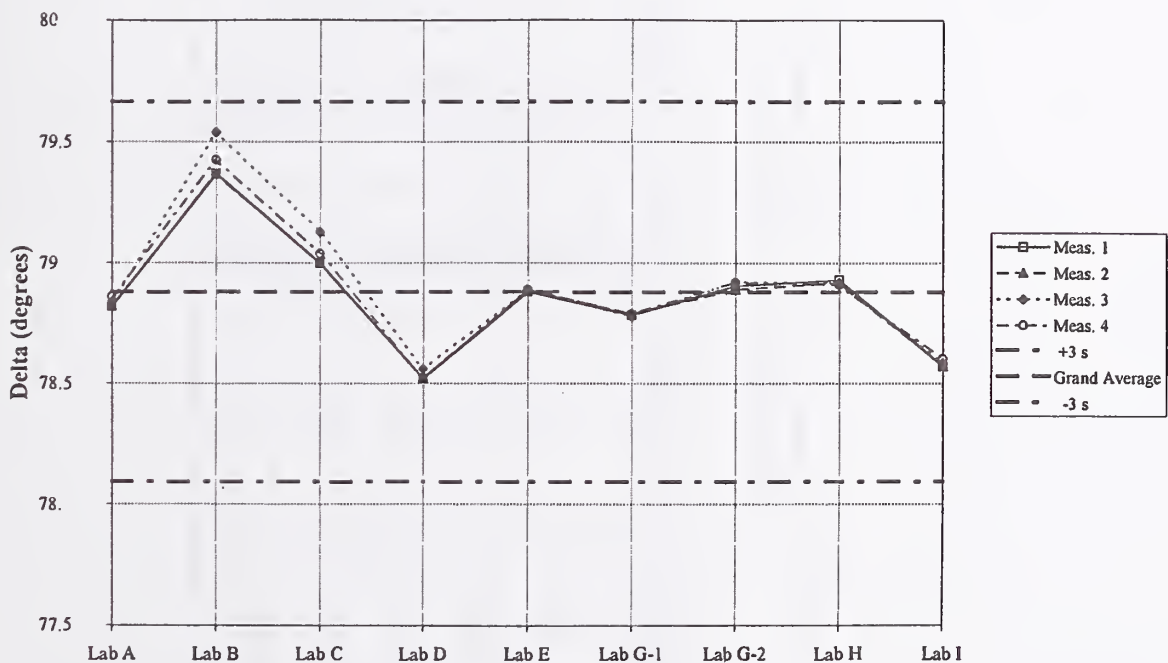


Sample 1004-17 Measured Psi Values



Laboratory Code
Figure 6c

Sample 1004-17 Measured Delta Values



Laboratory Code
Figure 6d

TABLE 7. Summary of the Statistical Analysis as Performed by the ASTM E691 Program and as Performed Independently in the Spreadsheet

Specimen ID	t nom (nm)	Method	t GA (nm)	s (nm)	Sr	SR	r (2.8xSr)	R (2.8xSR)
1019-01	10	Lab	10.66	0.33	0.04	0.33	0.10	0.94
		Std	10.84	0.18	0.03	0.18	0.08	0.51
1019-02	10	Lab	10.76	0.29	0.04	0.30	0.10	0.83
		Std	10.90	0.22	0.05	0.23	0.15	0.63
1017-20	50	Lab	52.57	0.44	0.09	0.45	0.26	1.26
		Std	52.66	0.43	0.09	0.43	0.25	1.22
1017-23	50	Lab	52.45	0.36	0.06	0.33	0.18	0.91
		Std	52.53	0.39	0.05	0.39	0.15	1.10
1004-14	100	Lab	101.23	0.31	0.06	0.31	0.16	0.88
		Std	101.34	0.32	0.05	0.32	0.15	0.91
1004-17	100	Lab	101.04	0.32	0.06	0.33	0.18	0.91
		Std	101.20	0.21	0.02	0.21	0.05	0.58

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