Stylus Profilometer Round-Robin Study for Depth Measurements

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A round-robin study was organized among Japanese SIMS users to evaluate the repeatability and reproducibility of depth measurements by stylus profilometry. The uncertainty of depth in stylus profilometry was found to be small, 3% or less in the depth range 10^{-2} to 10^{0} μm

1. Introduction

Depth profiling is an important mode of materials analysis in secondary ion mass spectrometry (SIMS), Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS). To evaluate material composition below the surface, a crater is formed on the sample surface by the rastered ion beam. Then, the material composition is measured as a function of etching time. Accurate correlation between etching time and crater depth is currently done by using a stylus profilometer. In the comparison of depth profile between laboratories, depth scale uncertainties affect not only profile shape but also the measured dose of an ion implant, especially in SIMS. Deviation in SIMS quantitative values based on the depth profiling of ion-implanted reference materials were of 5-10% [1]. On the other hand, the round robin test organized by Simons[2,3] demonstrated that the uncertainties of SIMS crater depth measurements are within 1.3% for a crater of 2μm, 2.4% for 0.5μm and 4.7% for 0.1μm. We therefore independently examined the precision of stylus profilometry, in conjunction with the previous SIMS depth profiling round robin test [1], using certified standard samples.

2. Experimental

The test sample used was a step calibration standard from Taylor Hobson Ltd. The grooves of the standard had depths of 0.029 (standard deviation: 0.0006 μm), 0.27 (0.003 μm) and 2.33 (0.006 μm) μm. Eighteen laboratories participated in the tests. Most of them were participants of a previous SIMS round robin test [1]. The stylus profilometers used were seven models from three manufacturers. The participants and the profilometer models used are listed in Table 1. Most of the profilometers had embedded microprocessors and video display screens, and had features such as automatic software leveling, autoscaling, and depth averaging. Four older models had strip chart output that required manual depth measurement. The profilometers were calibrated with laboratory calibration standards. The analysis protocol requested measurements of three scans at each groove.

3. Results

The measured depths of each laboratory are summarized in Table 2, and shown graphically for each of the 3 grooves in Figs. 1, 2, and 3, respectively (data for the 0.0029 μm groove was not available for labs 2, 9, 16, 17 and 20). The error bars in the figures are standard

Table 1 Round-robin participants and profilometer models with laboratory calibration standards

Paticipating laboratories	Stylus profilometry	Lab. Standard (nm)
Camaca Instruments Japan K.K.	DEKTAK 3ST Auto	473.0
Fuji Electric	DEKTAK 2A	980.0
Fujtsu Ltd.	DEKTAK 3030	878.0
Furukawa Electric	TalyStep	
Kawasaki Steel Corp.	Alphastep 200	-
Kobelco Research Institute,Inc.	DEKTAK 3030	909.0
Koukan Keisoku K.K.	DEKTAK 2A	880.0
Matsushita Technoresearch Inc.	DEKTAK 2A	840.0 450.0
NEC Corporation	DEKTAK 3ST	192.6
Nippon Steel Corp.	DEKTAK 3030	980.0
NTT	TalyStep	2670.0 450.0
Sanyo Electric Co., Ltd.	DEKTAK 3ST	962.0
Sharp Corporation	TalyStep	2210.0
	DEKTAK 3ST	94.1
Sony Corporation	TalyStep	2590.0
	TalyStep	350.0
Toray Research Center	DEKTAK 3030ST	949.0
Toshiba Corporation	DEKTAK 3030	894.0
Toyota Central R&D Labs., Inc.	DEKTAK 3ST	929.9
anonymous	DEKTAK 3030ST	86.3

deviations of the mean values. These data clearly show that the values for most of the laboratories lie within ±2% of the weighted mean of each groove. The data scattering is very small, within ±1% for most data, for the deepest groove of 2.318µm. The difference between the mean and the standard value is within the statistical error for all the grooves.

These measured values were statistically analyzed according to ISO protocol, ISO 5725-2 [4], to obtain the repeatability and reproducibility of the measurements. Cochran's test, Grubb's test and the graphical consistency technique were applied independently to the data according to ISO 5725-2. An outlier indicated by all the tests was excluded from the analysis (lab 18 for 0.029 µm groove).

The results are presented in Table 3 and Fig. 4. The repeatability s_r is very small, less than 3%, for all the grooves. The reproducibility s_R is less than 1% for the deepest groove. It gradually decreases with

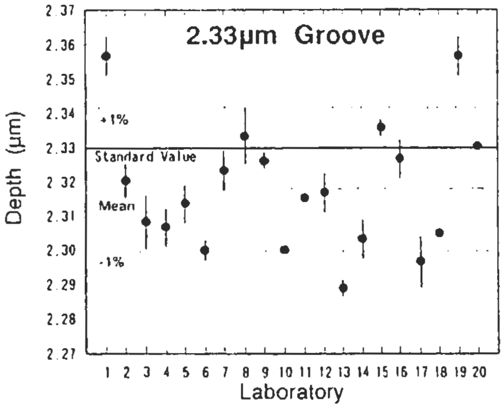

Figure 1 Individual laboratory groove depth measurements for 2.33µm

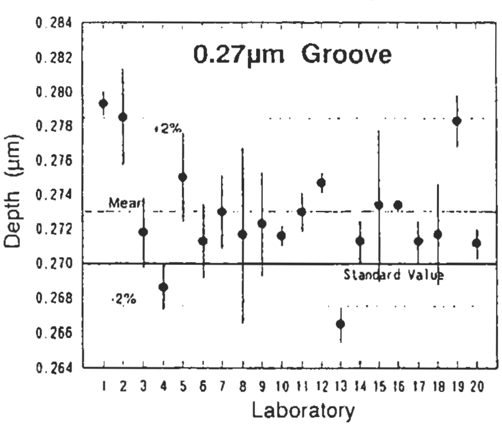

Figure 2 Individual laboratory groove depth measurements for 0.27µm

Table 2 Results of the round-robin test

	2.33μm groove		0.27μm groove		0.029μm groove	
	Mean	RSD (%)	Mean	RSD (%)	Mean	RSD (%)
1	2.357	0.245	0.279	0.235	0.0294	2.700
2	2.320	0.246	0.279	1.016		
3	2.308	0.331	0.272	0.743	0.0287	2.238
4	2.307	0.154	0.269	0.475	0.0279	1.897
5	2.314	0.246	0.275	0.962	0.0288	0.873
6	2.300	0.138	0.271	0.774	0.0285	5.611
7	2.323	0.249	0.273	0.751	0.0288	0.919
8	2.333	0.353	0.272	1.891	0.0300	1.764
9	2.326	0.111	0.272	1.115		
10	2.300	0.000	0.272	0.205	0.0285	0.535
11	2.315	0.056	0.273	0.412	0.0285	2.654
12	2.317	0.249	0.275	0.207	0.0293	3.361
13	2.289	0.108	0.267	0.378	0.0290	2.614
14	2.303	0.251	0.271	0.426	0.0291	0.396
15	2.336	0.106	0.273	1.613	0.0290	2.763
16	2.327	0.248	0.273	0.110		
17	2.297	0.333	0.271	0.426		
18	2.305	0.000	0.272	1.063	0.0283	10.189
19	2.357	0.245	0.278	0.549	0.0293	3.936
20	2.330	0.061	0.271	0.338		
Mean value	2.318	0.795	0.273	1.147	0.0289	1.783

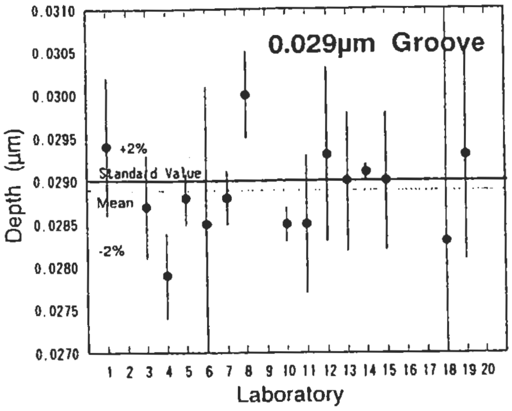

Figure 3 Individual laboratory groove depth measurements for 0.029μm

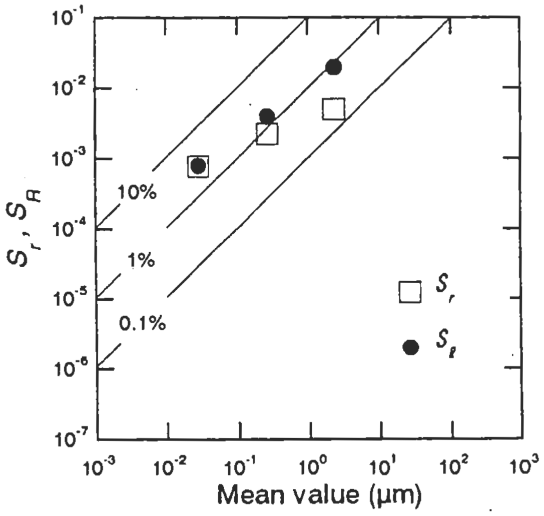

Figure 4 Relationship between mean value and repeatability (S_r), reproducibility (S_R)

Table 3 Repeatability and reproducibility of groove depths (in μm)

Level	Number of labs	Mean value	S_r	S_R
1	20	2.32	4.980E-03	1.990E-02
2	20	0.27	2.260E-03	3.940E-03
3	14	0.029	7.760E-04	7.980E-04

decrease in the depth, but stays less than 3% even for the 0.029 μm groove.

Those results confirm the high accuracy of stylus profilometry in the depth range from 10^{-2} to 10^{0} μm. There were no differences in precision between manual and computerized data handling.

4. Conclusion

The present results confirm that the uncertainty of depth in stylus profilometry is 3% or less in the depth range from 10^{-2} to 10^{0} μm. Thus, the deviation in quantitative value in SIMS depth profiling is mainly influenced by fluctuations in SIMS measurement itself, and not by stylus profilometry. However, measurement of real SIMS craters may have higher uncertainty because they are not as ideal as this test sample.

References

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