Long term stability of silicon roughness standards

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Abstract

In 1986 the first roughness reference standards made of silicon were produced at the Laboratory for Precise Measurements of Length (LFSB), which is now a part of Croatian Metrology Institute (HMI) designated as HMI/FSB-LPMD. Until then, roughness standards were made either from steel or glass.

After 27 years of use we decided to conduct a research of metrological features on two silicon roughness standards, used as primary standards for roughness in Croatia. The analysis relies on the roughness parameters measurement results (*Ra*, *Rz* and *RSm*) from calibration certificates provided by several national metrology institutes. Results of the Birge ratio test showed a good statistical consistency within the declared levels of measurement uncertainties.

1. HMI/FSB-LPMD roughness reference standards

HMI/FSB-LPMD standards were made from silicon mono-crystal processed by planar technology, obtaining rectangular grooves in the SiO_2 layer (Figure 1). Standards have two measurement surfaces with the same nominal values of the groove depths:

- surface with a repeating pattern of SiO_2 rectangular groove;
- surface with three wide SiO_2 rectangular grooves. [1,2]

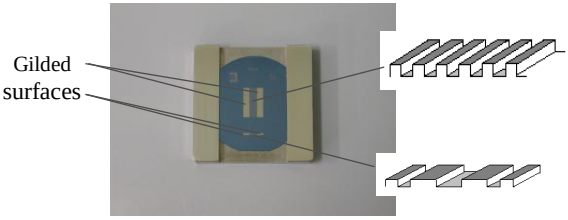

Figure 1: HMI/FSB-LPMD roughness reference standard

The research of metrological features was conducted on two roughness standards, Ref. No. 0-6 and 0-8. For 27 years these standards were continuously in use as national roughness standards and were regularly calibrated. The first calibration certificate dates from 1994. In order to assure traceability to secondary standards and stylus instruments the standards were measured more than 400 times using contact method. In each measurement, at least 6 tracks were made on the measuring surface using different probes, i.e. more than 2400 tracks were made on the standards measuring surface.

2. Analysis of roughness parameters stability

In order to conclude about stability of metrological characteristics, this research was focused on calibration results provided by several national metrology institutes. Roughness parameters (*Ra*, *Rz* and *RSm*) were measured in accordance with the edition of ISO 4287 which was applicable at the time. In all certificates, under common measurement conditions, the following parameters were stated: Gaussian filter, stylus tip radius r = 2 µm, reported results obtained on 12 profiles. Tables 1 and 2 shows the results of measured roughness parameters for standards 0-6 and 0-8 as reported in certificates, with expanded uncertainties expressed with coverage factor k = 2, P = 95%.

Table 1: Calibration results on standard 0-6

Year	λ_c, **mm**	***Ra*, µm**	U_{95}	***Rz*, µm**	U_{95}	***RSm*, µm**	U_{95}
1994	0.8	0.77	5 %	1.75	8 %	-	-
1997	0.8	0.83	4 %	1.79	6 %	-	-
1999	0.8	0.83	4 %	1.79	6 %	-	-
2004	0.8	0.81	4 %	1.78	6 %		
2008	0.25	0.827	2 %	1.76	2 %	76.03	0.25 µm
2012	0.8	0.82	0.03 µm	1.76	0.09 µm	75.97	0.50 µm

Table 2: Calibration results on standard 0-8

Year	λ_c, **mm**	***Ra*, µm**	U_{95}	***Rz*, µm**	U_{95}	***RSm*, µm**	U_{95}
1994	0.8	1.76	5 %	3.91	8 %	-	-
1997	0.8	1.85	4 %	4.03	6 %	-	-
1999	0.8	1.85	4 %	4.01	6 %	-	-
2004	0.8	1.81	4 %	4.00	6 %		
2008	0.25	1.84	2 %	3.96	2 %	75.95	0.25 µm
2012	0.8	1.84	0.07 µm	3.97	0.20 µm	76.00	0.50 µm

The weighted mean was used as the reference value in the comparison of roughness parameters measurement results. The statistical consistency of the results with the uncertainties given by calibration certificates was checked by the *En* value for each institute. In order to compare the observed spread of the results with the spread expected from the individual reported uncertainties a Birge ratio test was applied. Results calculated from reported values on analysed standards are given in table 3.

Table 3: Results calculated from reported values

		1994	1997	1999	2004	2008	2012
Ra on Ref. No. 0-6	x_i-x_w	-0.050	0.010	0.010	-0.010	0.007	0.000
	En	-1.250	0.284	0.284	-0.294	0.349	-0.002
	u_{int} = 0.006 µm	u_{ext} = 0.007 µm		R_B = 1.31	$R_{B,crit}$= 1.50		x_w = 0.820 µm
Ra on Ref. No. 0-6 *	x_i-x_w	-0.055	0.005	0.005	-0.015	0.002	-0.005
	En	-1.357	0.155	0.155	-0.423	0.122	-0.141
	u_{int} = 0.006 µm	u_{ext} = 0.003 µm		R_B = 0.49	$R_{B,crit}$= 1.55		x_w = 0.824 µm
Rz on Ref. No. 0-6	x_i-x_w	-0.015	0.025	0.025	0.015	-0.005	-0.005
	En	-0.106	0.223	0.223	0.134	-0.115	-0.055
	u_{int} = 0.014 µm	u_{ext} = 0.005 µm		R_B = 0.36	$R_{B,crit}$= 1.50		x_w = 1.765 µm
Ra on Ref. No. 0-8	x_i-x_w	-0.072	0.018	0.018	-0.022	0.008	0.008
	En	-0.792	0.226	0.226	-0.292	0.172	0.103
	u_{int} = 0.012 µm	u_{ext} = 0.011 µm		R_B = 0.87	$R_{B,crit}$= 1.50		x_w = 1.832 µm
Rz on Ref. No. 0-8	x_i-x_w	-0.06	0.06	0.04	0.03	-0.01	0.000
	En	-0.188	0.239	0.160	0.120	-0.100	-0.001
	u_{int} = 0.032 µm	u_{ext} = 0.011 µm		R_B = 0.36	$R_{B,crit}$= 1.50		x_w = 3.97 µm

*Results calculated from largest consistent subset (Excluded result-yellow)

Overall, the calibration results obtained through last 18 years showed good statistical consistency within the declared levels of measurement uncertainties, with the exception of the result for *Ra* parameter measured in 1994.

In calibration certificates institutes have also expressed values of estimated standard deviations for measured roughness parameters obtained on 12 profiles. Reported values of standard deviations indicates that, over the past 18 years, the uniformity of surfaces has not been significantly changed. Especially indicative are the values of

standard deviations for *Rz* parameter which is particularly sensitive to surface damage and consequent changes in profile geometry.

3. Conclusion

The paper examines the metrological characteristics of the first roughness standards that were produced from silicon back in 1986. For the purpose of this research two Croatian national roughness standards were analysed. The analysis relies on the roughness parameters measurement results (*Ra*, *Rz* and *RSm*) given in 8 calibration certificates, for the period from 1994 to 2012.

The statistical consistency of this comparison was analysed by the Birge ratio test, in order to compare the observed spread of the results with the spread expected from the individual reported uncertainties. Weighted mean and its difference from the reported results were calculated, along with corresponding *En* values (k = 2) for each roughness parameter. Calibration results obtained during last 18 years showed an overall good comparability within the declared levels of measurement uncertainties.

Declared values of estimated standard deviations for measured roughness parameters indicate that over the past 18 years the uniformity of surfaces was not altered i.e. measurement surfaces suffered no significant damage that would impair the metrological characteristics of these standards.

The analysis of metrological characteristics on two roughness standards, after 27 years of continuous use, indicates the outstanding quality of the first roughness reference standards made from silicon.

References:

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