Thermal Expansion Coefficient: Gauge block

- Final Report -

Contents

1. Introduction

The metrological equivalence of national measurement standards and of calibration certificates issued by national metrology institutes is established by a set of key comparisons chosen and organized by the Consultative Committees of the CIPM or by the regional metrology organizations in collaboration with the Consultative Committees.

At its meeting in 2002, the Consultative Committee for Length, CCL, decided upon a supplementary comparison on thermal expansion coefficient of gauge block, with the National Metrology Institute of Japan (NMIJ/AIST) as the pilot laboratory.

2. Organizations

The technical protocol was drafted by NMIJ/AIST with the help of contributions from other participants. The protocol document was issued to all participants at the start of the comparison.

2.1. Participants

The list of participants as originally printed in the protocol is given in Table 1.

Contact parson	National Metrology Institute and address	TEL, FAX and e-mail
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Table 1. Participant information

2.2. Schedule

The schedule of the supplementary comparison is given in Table 2

From	To	Organization
-	2004/5/31	NMIJ/AIST
2004/6/9	-	IMGC-CNR
2004/7/13	-	METAS
2004/9/7	-	MIKES
(2004/10/5)	(2004/11/8)	PTB
(2004/12/14)	(2005/1/24)	CENAM
-	2005/2/28	NMIJ/AIST
2005/4/1	2005/5	CMI

Table 2. Comparison schedule

3. Standards: Gauge blocks for comparison

Three ceramics block gauges in length of 20 mm, 50 mm and 100 mm, and a steel gauge block in length of 100 mm were prepared as calibration artifacts. The grade of the gauge blocks was the class K of Japan industrial standard B7506-1997. They were supplied in two wooded boxes containing packing boxes. The picture of gauge blocks in wooded boxes is shown in figure 1(a) and figure 1(b).

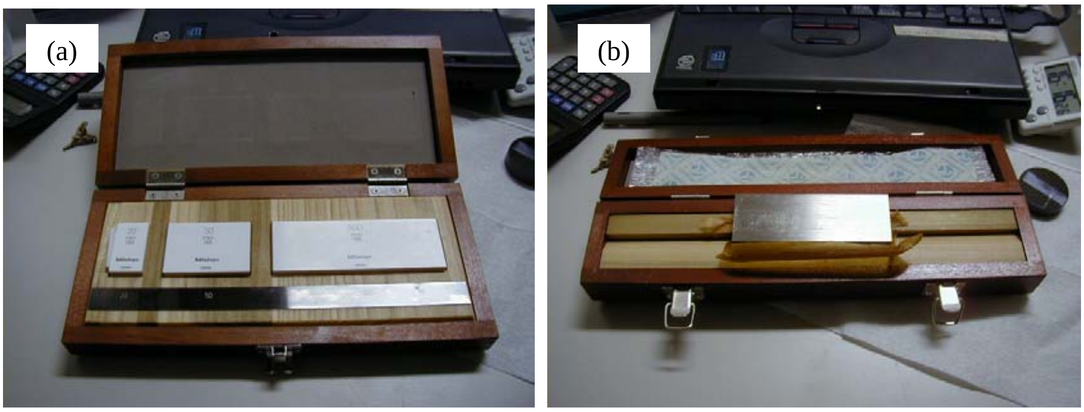

Figure 1. (a): Ceramics gauge blocks (L_0=20 mm, 50 mm and 100 mm), (b): Steel gauge block (L_0=100 mm).

The permissible value of α in the steel gauge block, which is shown the manufacturer's note, is $(10.9\pm1.0)\times10^{-6}$°C^{-1} at 20 °C. Three ceramics gauge blocks were made especially for this comparison. The material of the ceramics gauge block is partially-stabilized zirconia powder, TZ-3Y20AB, supplied by TOSOH Corporation. Table 4 shows the specification of TZ-3Y20AB from manufacturer's technical note.

Partially-stabilized zirconia powder; TZ-3Y20AB (including binder for sint			
	$A_{l2}O_3$	Y_2O_3	ZrO_2 *2
Mass fraction /wt%	20±2.0 *1	3.9±0.3 *1	Balance

*1: nominal values

*2: HfO_2 is included, ZrO_2 : HfO_2 ≅ 98 wt%:2 wt%

Table 3. Specification of the ceramics gauge block (material powder)

The α value of the ceramics gauge blocks is estimated by Turner's equation [1]:

$$\alpha = \frac{\sum_i (\alpha_i F_i k_i / \rho_i)}{F_i k_i / \rho_i},$$

where α_i, F_i, k_i and ρ_i represent ,respectively, thermal expansivity, weight percent, bulk modulus, and density. The subscripts, i, represents each components. The α value of the ceramics gauge blocks for this comparison at 20 ℃ is calculated using the physical constants and parameters, listed in table 4. The calculated α result for mixture using the physical constants and parameters listed in table 4 is $(8.03\pm0.20)\times10^{-6}$℃$^{-1}$ at 20 ℃. The value of $\pm0.20\times10^{-6}$℃$^{-1}$ presents the expanded uncertainty of the estimated α value in the mixture.

		ZrO_2	HfO_2	Al_2O_3	Y_2O_3
LTEC:	α *1	9.6 ± 0.1	3.8 ± 0.2	5.30 ± 0.02	7.3 ± 0.4
Weight propotion:	F *2	73.9 ± 1.3	1.5 ± 0.6	20.6 ± 1.2	4.0 ± 0.2
Bulk modulous:	k *2	184 ± 8	184 ± 8	228 ± 6.0	148 ± 7
Density:	ρ *2	6.00 ± 0.06	9.68 ± 0.06	3.90 ± 0.06	4.92 ± 0.06

*1: refrence [2], [3], [4]
*2: from manifacturer's notes

Table 4. Physical constants and parameters for the ceramics gauge block for the comparison.

4. Measurement instructions and reporting of the results

4.1. Traceability

Length measurements should be traceable to the definition of length (wavelength of light). Temperature measurements should be made using the international Temperature Scale of 1990 (ITS-90).

4.2 Measurand

The measurand in this comparison was the thermal expansion coefficient of the gauge blacks around room temperature. The thermal expansion coefficient is determined from measurements of changing in length and temperature of the gauge block.

From the measurement result of length and temperature, for example, the average linear thermal expansion coefficient, α, can be obtained by the following equation,

$$\alpha(T_{ave}) = \frac{1}{L_0}\cdot\frac{L(T_2)-L(T_1)}{T_2-T_1}; T_{ave} = \frac{T_1+T_2}{2},$$

where $L(T_2)$-$L(T_1)$ and T_2-T_1 (= ΔT) are the length changing and the temperature changing for a gauge black, respectively. The thermal expansion coefficient, $\alpha(T_{ave})$, is the average linear thermal expansion coefficient in temperature range from T_1 to T_2 and L_0 is the length of the gauge black at 20 ℃. The α value at arbitrary temperature in the measurement temperature range can be determined by curve fitting on measurement data.

4.3. Measurement temperature range

The measurement temperature range was from 10 ℃ to 30 ℃. In particular, the temperatures at which thermal expansion coefficient should be determined are 10 ℃, 15 ℃, 20 ℃, 25 ℃ and 30 ℃. The determination of thermal expansion coefficient near these temperatures is preferable.

4.4. Inspection of the artifacts

Before measurement, the artifacts had to be inspected for damage to the measurement surfaces.

4.5. Measurement uncertainty

The uncertainty of measurement should be estimated according to the *ISO Guide to the Expression of Uncertainty in Measurement* [5]. Because for this comparison the measurement equipment and procedure was not fixed, it was not possible to develop a full mathematical model for the measurement uncertainty for all participants.

5. Stability of Artifacts

Two measurements of the gauge blocks were performed by NMIJ, one in June-2003, and in February-2005. No significant change of α value was observed in all gauge blocks. Fig 2.1-4 show the deviation from regression line and error bar denotes uncertainties (k=2). The calibration results show that the α property of the artifacts is stable through the comparison. It is considered that the larger uncertainty of calibration result in February-2005 was caused by the unstable environment condition, not by the artifacts themselves.

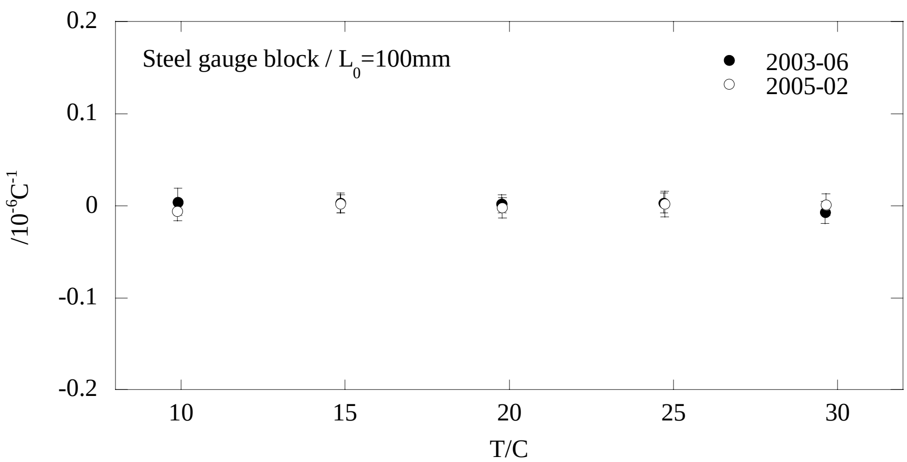

Figure 2.1 Stability of steel gauge block

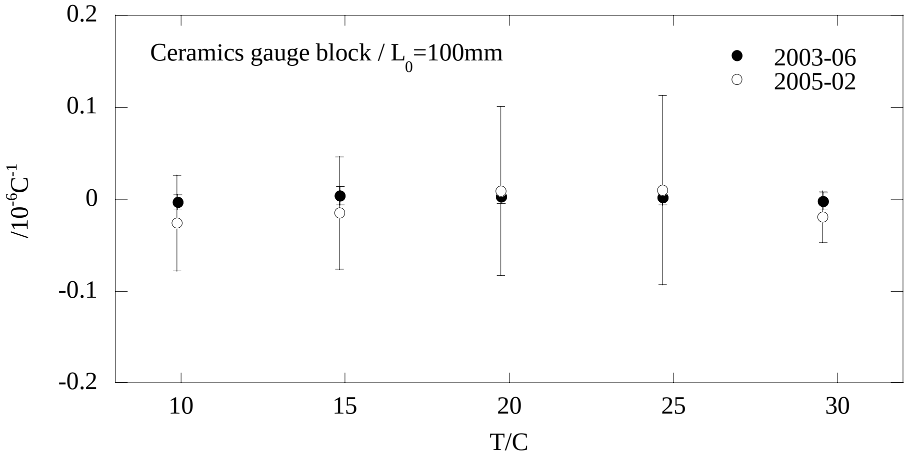

Figure 2.2 Stability of ceramics gauge block (L_0=100 mm)

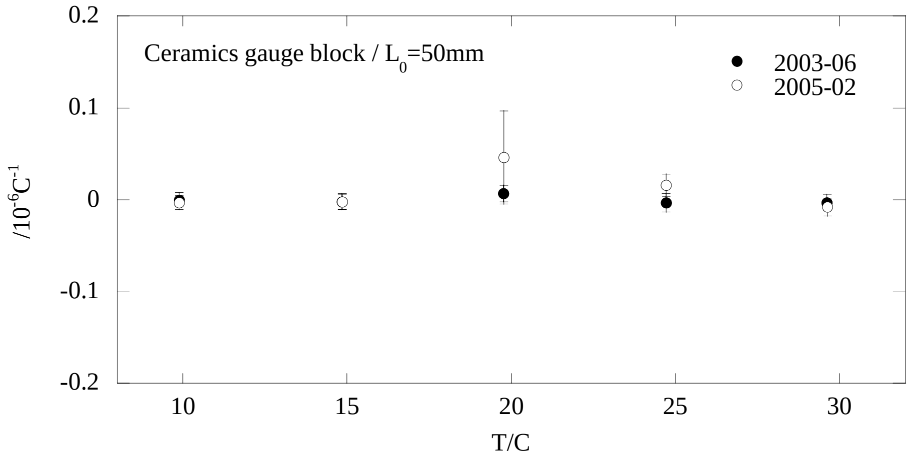

Figure 2.3 Stability of ceramics gauge block (L_0=50 mm)

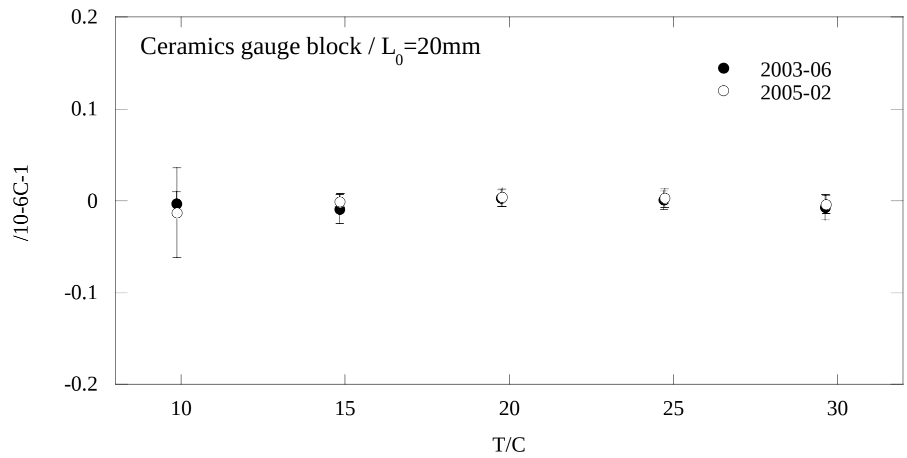

Figure 2.4 Stability of ceramics gauge block (L_0=20 mm)

6. Measurement results

Given measurement results and the measurement uncertainties of α in individual gauge block were listed in Table 2.1-2.4. The determination procedure of α, measurement methods and temperatures at which α was measured were not necessarily corresponding between participants.

Gauge block: L_0=100 mm / Steel				
Organization	T / ℃	α /10^{-6}℃$^{-1}$	$U(\alpha)$; (k=2) /10^{-6}℃$^{-1}$	Remarks
NMIJ/AIST	9.904	10.468	0.015	ΔT=4.957 ℃
	14.857	10.535	0.011	ΔT=4.956 ℃
	19.774	10.602	0.010	ΔT=4.979 ℃
	24.711	10.669	0.011	ΔT=4.943 ℃
	29.620	10.727	0.012	ΔT=4.966 ℃
IMGC-CNR	19.79	10.65	0.02	ΔT= about 4 ℃
METAS	10	10.500	0.036	α value was calculated from seven
	15	10.570	0.030	measerenent data in temperature range
	20	10.640	0.027	form 10 ℃ to 30 ℃
	25	10.709	0.030	
	30	10.779	0.036	
MIKES	15	10.560	0.033	
	20	10.640	0.033	
	25	10.732	0.033	
PTB	10	10.4585	0.0216	α value was calculated from nine
	15	10.5372	0.0039	measerenent data in temperature range
	20	10.5975	0.0030	form 10 ℃to 30 ℃
	25	10.6578	0.0039	
	30	10.7174	0.0215	
CENAM	19.3	10.7	0.1600	ΔT=4.8 ℃
	23.85	10.69	0.1600	ΔT=4.3 ℃
CMI	14.85	10.53	0.064	
	19.91	10.60	0.064	
	24.6	10.67	0.064	

Table 5.1. α data and expand uncertainty for steel gauge block (L_0=100 mm).

Gauge block: L_0=100 mm / Ceramics				
Organization	T / ℃	α /10^{-6}℃$^{-1}$	$U(\alpha)$; (k=2) /10^{-6}℃$^{-1}$	Remarks
NMIJ/AIST	9.900	7.9785	0.0077	ΔT=4.950 ℃
	14.848	8.0503	0.0100	ΔT=4.948 ℃
	19.754	8.1144	0.0078	ΔT=4.960 ℃
	24.675	8.1783	0.0082	ΔT=4.932 ℃
	29.569	8.2388	0.0088	ΔT=4.950 ℃
IMGC-CNR	19.82	8.13	0.02	ΔT= about 4 ℃
METAS	10	8.028	0.036	α value was calculated from six
	15	8.097	0.030	measerenent data in temperature range
	20	8.166	0.027	form 10 ℃ to 30 ℃
	25	8.235	0.030	
	30	8.304	0.036	
MIKES	NA			
PTB	10	7.9786	0.0410	α value was calculated from eight
	15	8.0419	0.0049	measerenent data in temperature range
	20	8.1087	0.0026	form 10 ℃ to 27.5 ℃
	25	8.1755	0.0049	
CENAM	19.3	8.18	0.16	ΔT=4.8 ℃
	23.85	8.19	0.16	ΔT=4.3 ℃
CMI	14.85	8.06	0.095	
	19.91	8.11	0.095	
	24.6	8.19	0.095	

Table 5.2. α data and expand uncertainty for ceramics gauge block (L_0=100 mm).

Gauge block: L_0=50 mm / Ceramics				
Organization	T / ℃	α /10^{-6}℃$^{-1}$	$U(\alpha)$; (k=2) /10^{-6}℃$^{-1}$	Remarks
NMIJ/AIST	9.887	7.9860	0.0084	ΔT =4.964 ℃
	14.850	8.0502	0.0088	ΔT =4.962 ℃
	19.775	8.1244	0.0086	ΔT =4.981 ℃
	24.713	8.1798	0.0095	ΔT =4.944 ℃
	29.621	8.2454	0.0093	ΔT =4.967 ℃
IMGC-CNR	19.8	8.13	0.04	ΔT = about 4 ℃
METAS	10	7.943	0.041	α value was calculated from seven
	15	8.030	0.027	measerenent data in temperature range
	20	8.116	0.020	form 10 ℃ to 30 ℃
	25	8.203	0.027	
	30	8.290	0.041	
MIKES	15	8.047	0.045	
	20	8.098	0.045	
	25	8.198	0.045	
PTB	10	7.9721	0.0147	α value was calculated from eight
	15	8.0375	0.0018	measerenent data in temperature range
	20	8.104	0.0014	form 10 ℃ to 27.5 ℃
	25	8.1705	0.0018	
CENAM	19.3	8.14	0.32	ΔT =4.8 ℃
	24.0	8.21	0.32	ΔT =4.4 ℃
CMI	14.85	8.02	0.122	
	19.91	8.08	0.122	
	24.60	8.14	0.122	

Table 5.3. α data and expand uncertainty for ceramics gauge block (L_0=50 mm).

Gauge block: L_0=20 mm / Ceramics				
Organization	T / ℃	α /10^{-6}℃$^{-1}$	$U(\alpha)$; (k=2) /10^{-6}℃$^{-1}$	Remarks
NMIJ/AIST	9.859	7.9744	0.0128	ΔT =4.975 ℃
	14.834	8.0347	0.0158	ΔT =4.972 ℃
	19.762	8.1123	0.0103	ΔT =4.983 ℃
	24.707	8.1761	0.0104	ΔT =4.955 ℃
	29.623	8.2333	0.0141	ΔT =4.975 ℃
IMGC-CNR	19.8	8.08	0.22	ΔT = about 4 ℃
METAS	10	7.959	0.073	α value was calculated from seven
	15	8.044	0.047	measerenent data in temperature range
	20	8.130	0.035	form 10 ℃ to 30 ℃
	25	8.215	0.047	
	30	8.300	0.073	
MIKES	15	8.05	0.100	
	20	8.09	0.100	
	25	8.24	0.100	
PTB	10	7.9824	0.0221	α value was calculated from eight
	15	8.0483	0.0057	measerenent data in temperature range
	20	8.112	0.0052	form 10 ℃ to 27.5 ℃
	25	8.1642	0.0057	
CENAM	21.9	8.15	0.42	ΔT =10 ℃
CMI	14.85	8	0.301	
	19.91	8.1	0.301	
	24.60	8.25	0.301	

Table 5.4. α data and expand uncertainty for ceramics gauge block (L_0=20 mm).

7. Analysis of the reported results

7.1. Comparison with key reference values

The key reference values of α , $\alpha_{ref,}$ of individual gauge block were determined from the reported data by weighted least square method. The temperature dependence of α is linear sufficiently because the temperature range of measurement is narrow. The weight on the least square method is the uncertainty (k=1) of individual reported result. The calculated values of α_{ref} are listed in table6. The α_{ref} value at 20 ℃ correspond with the permissible value or the value estimated by Turner's equation shown in section 3, within uncertainty. In figure 3.1-4, solid line shows the value of α_{ref} and dot lines show the confidence interval (k=2) of α_{ref}. In figure 3.1-8, Error bars show the expanded uncertainty of individual reported result.

Gauge block: L_0=100 mm / Steel			Gauge block: L_0=100 mm / Ceramics		
T / ℃	α_{ref} /10^{-6}℃$^{-1}$	U_{ref} /10^{-6}℃$^{-1}$	T / ℃	α_{ref} /10^{-6}℃$^{-1}$	U_{ref} /10^{-6}℃$^{-1}$
10	10.474	0.031	10	7.979	0.030
15	10.537	0.021	15	8.045	0.020
20	10.600	0.016	20	8.111	0.016
25	10.663	0.020	25	8.178	0.022
30	10.726	0.030	30	8.244	0.032
Gauge block: L_0=50 mm / Ceramics			**Gauge block: L_0=20 mm / Ceramics**		
T / ℃	α_{ref} /10^{-6}℃$^{-1}$	U_{ref} /10^{-6}℃$^{-1}$	T / ℃	α_{ref} /10^{-6}℃$^{-1}$	U_{ref} /10^{-6}℃$^{-1}$
10	7.972	0.021	10	7.984	0.027
15	8.039	0.014	15	8.047	0.018
20	8.105	0.011	20	8.109	0.014
25	8.171	0.015	25	8.172	0.020
30	8.238	0.022	30	8.235	0.029

Table 6. Key reference values, α_{ref}, of individual gauge block

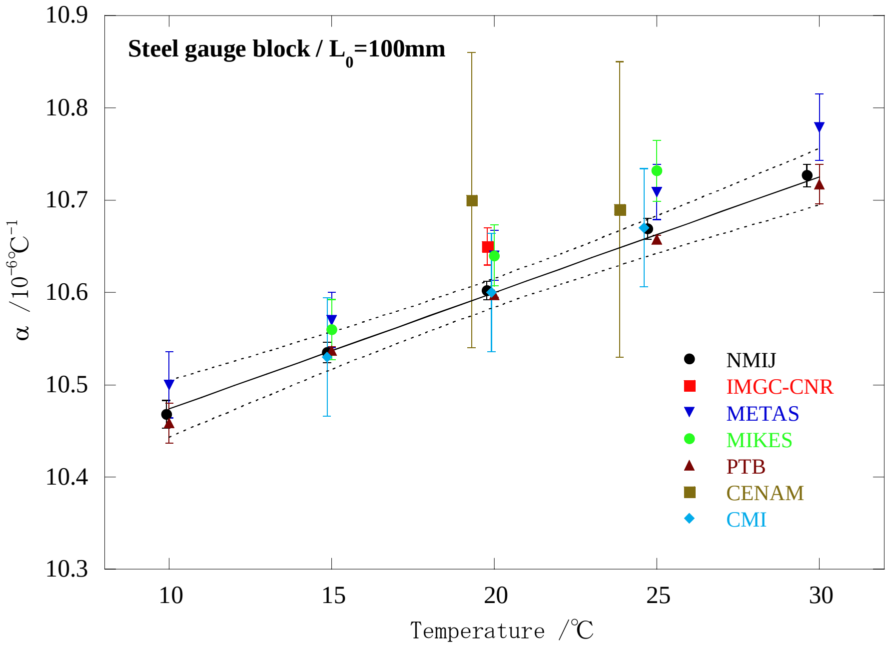

Figure 3.1. Results of α for the steel gauge block (L_0=100 mm).

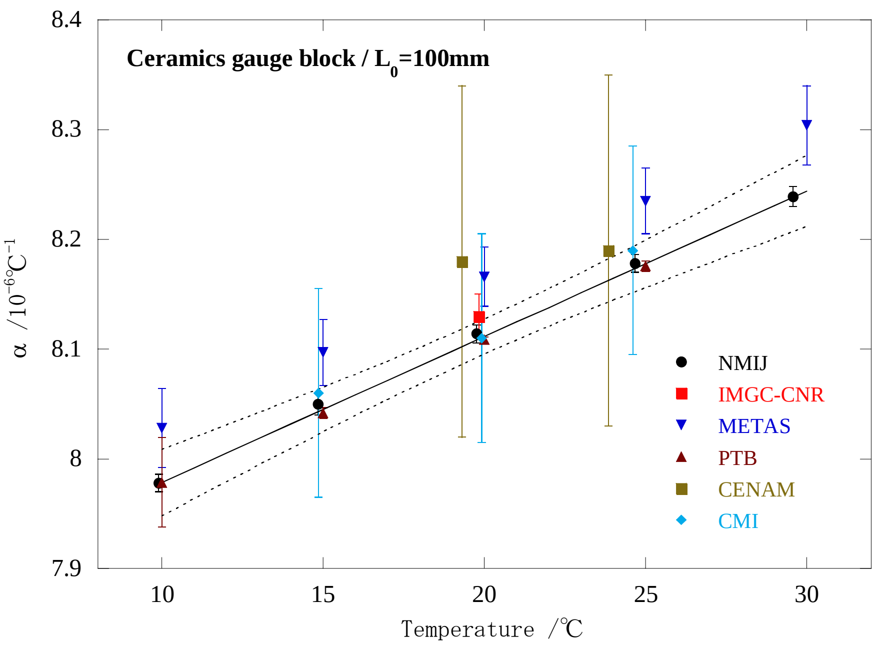

Figure 3.2. Results of α for the ceramics gauge block (L_0=100 mm).

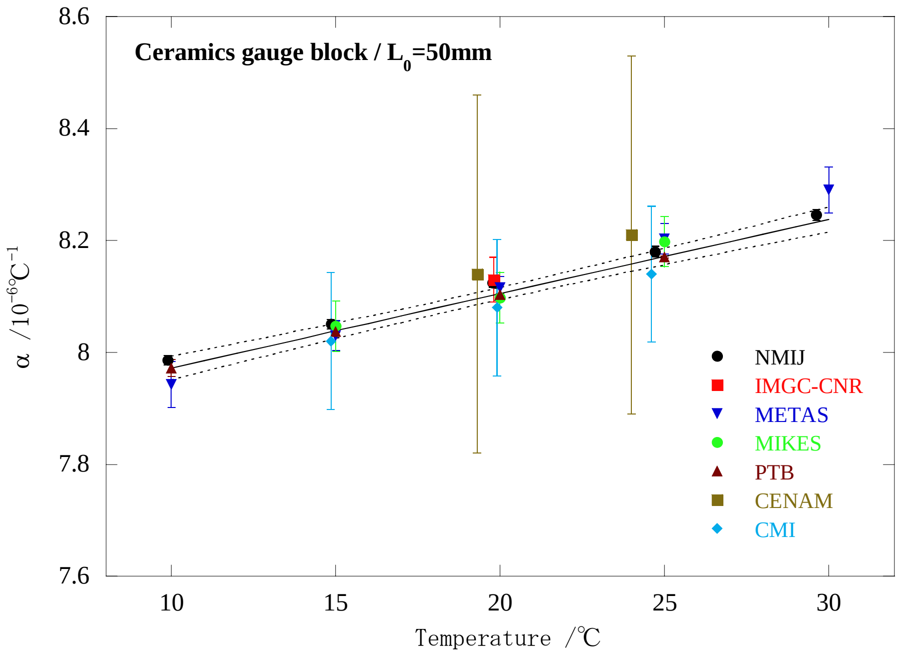

Figure 3.3. Results of α for the ceramics gauge block (L_0=50 mm).

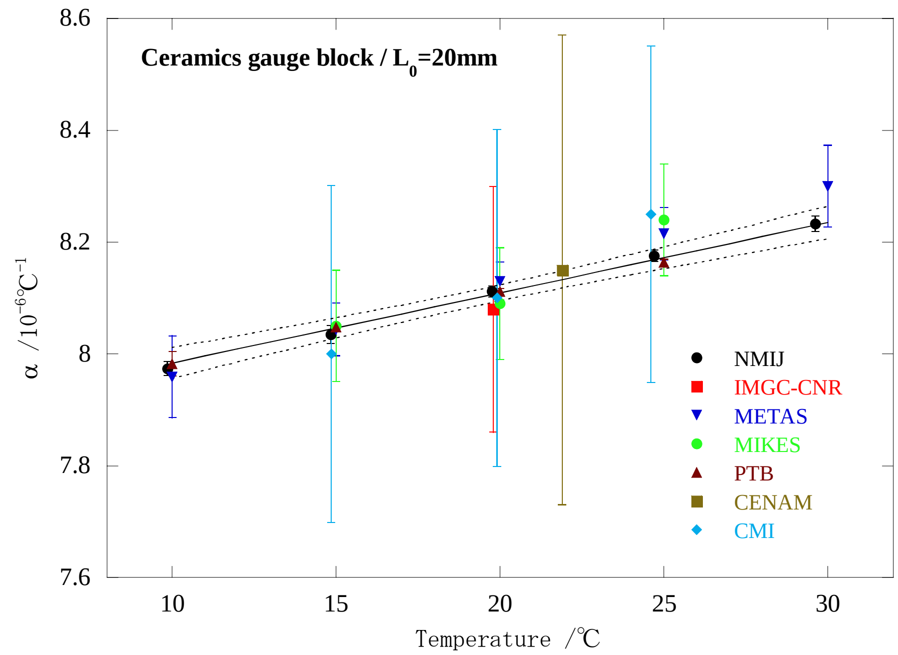

Figure 3.4. Results of α for the ceramics gauge block (L_0=20 mm).

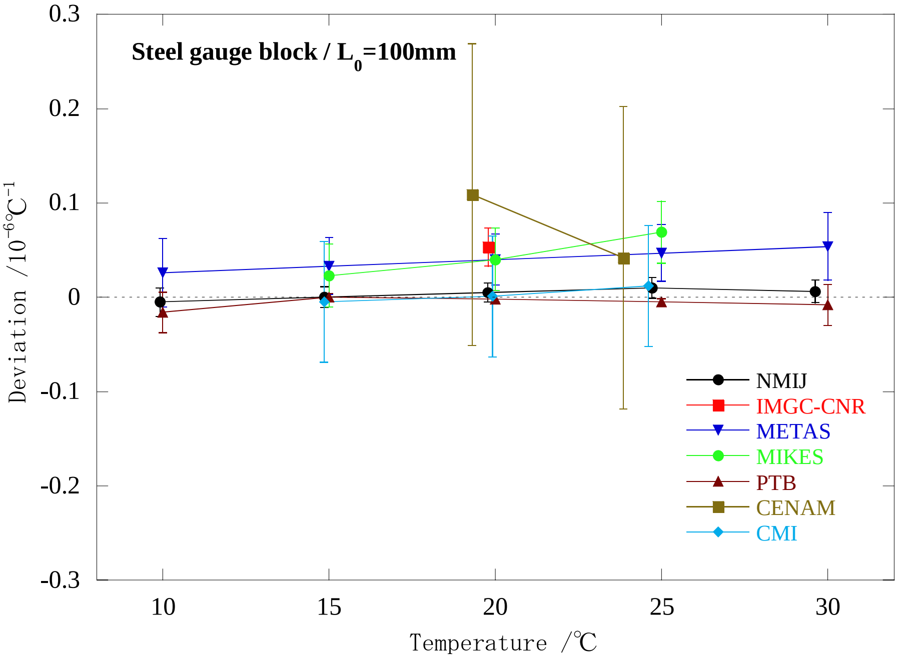

Figure 3.5. Deviation of α from key reference value for the steel gauge block (L_0=100 mm).

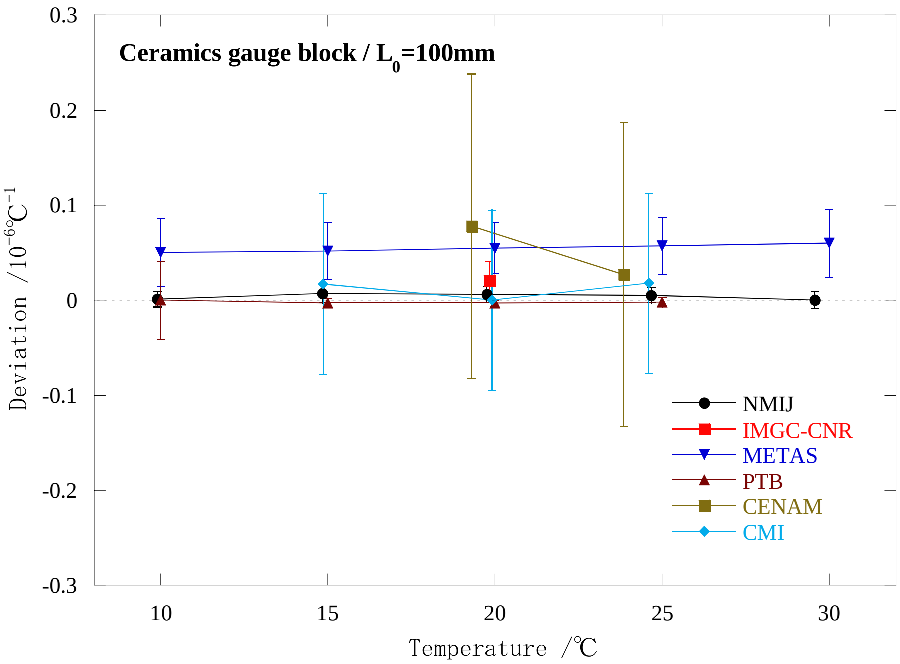

Figure 3.6. Deviation of α from key reference value for the ceramics gauge block (L_0=100

mm).

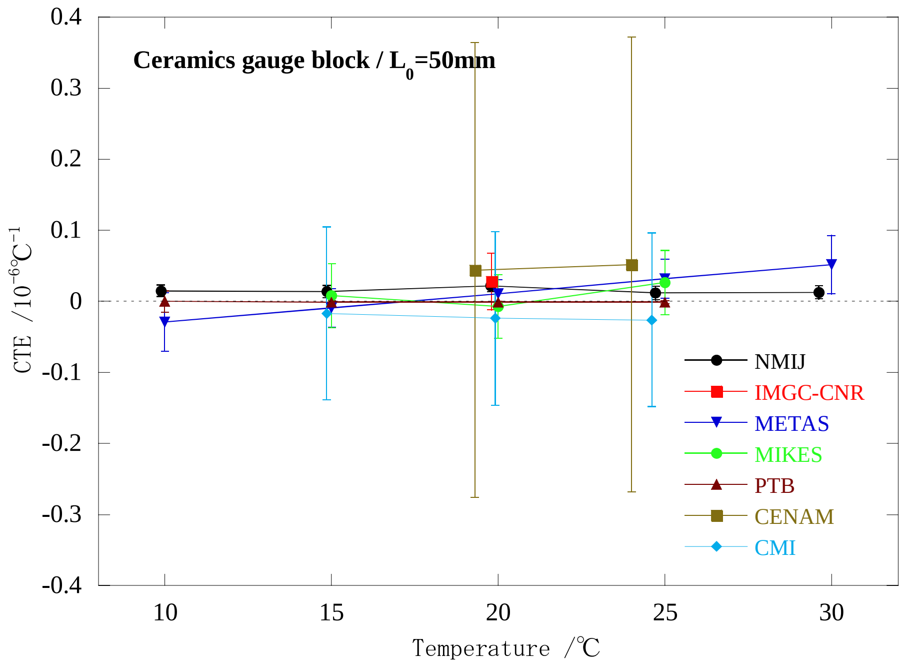

Figure 3.7. Deviation of α from key reference value for the ceramics gauge block (L_0=50 mm).

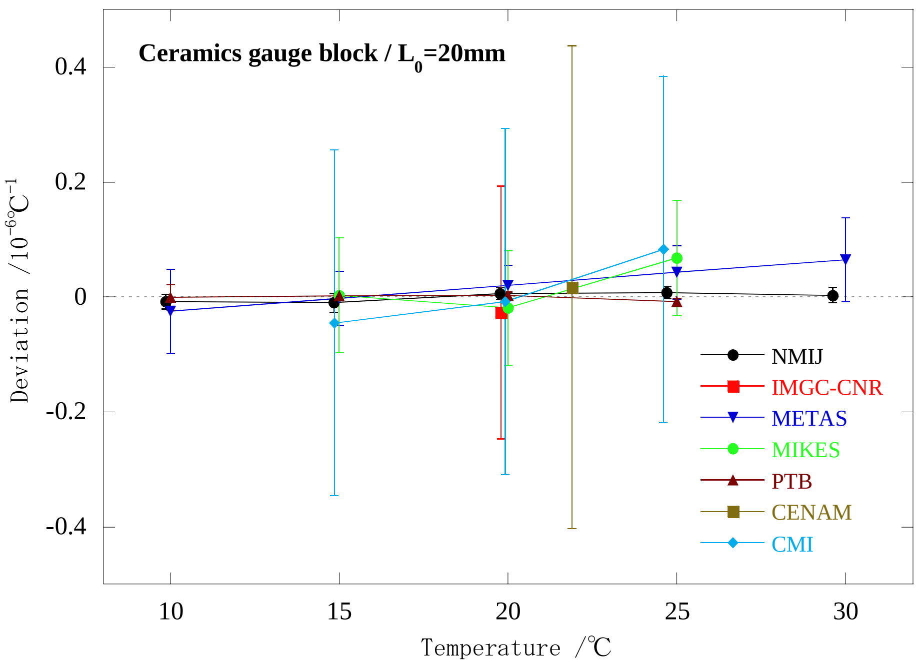

Figure 3.8. Deviation of α from key reference value for the ceramics gauge block (L_0=20 mm).

7.2. E_n value

The E_n value is defined as a following equation.

$$E_n = \frac{\alpha_{lab} - \alpha_{ref}}{\sqrt{U^2_{\alpha lab} + U^2_{\alpha ref}}}$$

where α_{lab} and α_{ref} present individual data value of a participant and the key reference value of the data, respectively. U_{lab}, U_{ref} and $\Delta\alpha$ present expanded uncertainty (k=2) of a participant and the key reference value of the data, deviation of α from its key reference value, respectively. Measurement results and the E_n value in individual gauge block listed in table 7.1-4.

Gauge block: L_0=100 mm / Steel				
Organization	T / ℃	α /10^{-6}℃$^{-1}$	$\Delta\alpha$ /10^{-6}℃$^{-1}$	E_n
NMIJ/AIST	9.904	10.468	-0.005	-0.133
	14.857	10.535	0.000	0.003
	19.774	10.602	0.005	0.243
	24.711	10.669	0.010	0.447
	29.620	10.727	0.006	0.190
IMGC-CNR	19.79	10.65	0.053	2.077
METAS	10	10.500	0.026	0.543
	15	10.570	0.033	0.900
	20	10.640	0.040	1.273
	25	10.709	0.047	1.290
	30	10.779	0.054	1.141
MIKES	15	10.560	0.023	0.592
	20	10.640	0.040	1.099
	25	10.732	0.069	1.787
PTB	10	10.4585	-0.016	-0.417
	15	10.5372	0.000	0.010
	20	10.5975	-0.002	-0.147
	25	10.6578	-0.005	-0.236
	30	10.7174	-0.008	-0.219
CENAM	19.3	10.7	0.109	0.678
	23.85	10.69	0.042	0.259
CMI	14.85	10.530	-0.005	-0.076
	19.91	10.600	0.001	0.020
	24.6	10.670	0.012	0.184

Table 7.1. E_n value of the steel gauge block (L_0=100 mm)

Gauge block: L_0=100 mm / Ceramics				
Organization	T / ℃	α /10^{-6}℃$^{-1}$	$\Delta\alpha$ /10^{-6}℃$^{-1}$	E_n
NMIJ/AIST	9.900	7.978	0.001	0.041
	14.848	8.050	0.007	0.326
	19.754	8.114	0.006	0.354
	24.675	8.178	0.005	0.211
	29.569	8.239	0.000	0.010
IMGC-CNR	19.82	8.13	0.021	0.821
METAS	10	8.028	0.050	1.054
	15	8.097	0.052	1.442
	20	8.166	0.055	1.739
	25	8.235	0.057	1.542
	30	8.304	0.060	1.231
MIKES	NA			
PTB	10	7.9786	0.000	0.002
	15	8.0419	-0.003	-0.146
	20	8.1087	-0.003	-0.164
	25	8.1755	-0.002	-0.103
CENAM	19.3	8.18	0.078	0.485
	23.85	8.19	0.027	0.170
CMI	14.85	8.06	0.017	0.176
	19.91	8.11	0.000	-0.002
	24.6	8.19	0.018	0.180

Table 7.2. E_n value of the ceramics gauge block (L_0=100 mm)

Organization	T / ℃	α /10^{-6}℃$^{-1}$	$\Delta\alpha$ /10^{-6}℃$^{-1}$	E_n
NMIJ/AIST	9.887	7.9860	0.015	0.676
	14.850	8.0502	0.014	0.825
	19.775	8.1244	0.022	1.638
	24.713	8.1798	0.012	0.712
	29.621	8.2454	0.013	0.539
IMGC-CNR	19.8	8.13	0.028	0.669
METAS	10	7.943	-0.029	-0.636
	15	8.030	-0.009	-0.294
	20	8.116	0.011	0.507
	25	8.203	0.032	1.038
	30	8.290	0.052	1.122
MIKES	15	8.047	0.008	0.179
	20	8.098	-0.007	-0.150
	25	8.198	0.027	0.563
PTB	10	7.9721	0.000	-0.002
	15	8.0375	-0.001	-0.076
	20	8.1040	-0.001	-0.089
	25	8.1705	-0.001	-0.058
CENAM	19.3	8.14	0.044	0.138
	24.0	8.21	0.052	0.162
CMI	14.85	8.02	-0.017	-0.135
	19.91	8.08	-0.024	-0.194
	24.6	8.14	-0.026	-0.212

Table 7.3. E_n value of the ceramics gauge block (L_0=50 mm)

Gauge block: L_0=20 mm / Ceramics				
Organization	T / ℃	α /10^{-6}℃$^{-1}$	$\Delta\alpha$ /10^{-6}℃$^{-1}$	E_n
NMIJ/AIST	9.859	7.974	-0.008	-0.253
	14.834	8.035	-0.010	-0.407
	19.762	8.112	0.006	0.332
	24.707	8.176	0.008	0.350
	29.623	8.233	0.003	0.097
IMGC-CNR	19.79	8.08	-0.027	-0.121
METAS	10	7.959	-0.025	-0.315
	15	8.044	-0.002	-0.043
	20	8.130	0.020	0.535
	25	8.215	0.043	0.837
	30	8.300	0.065	0.826
MIKES	15	8.05	0.003	0.033
	20	8.09	-0.019	-0.192
	25	8.24	0.068	0.666
PTB	10	7.9824	-0.001	-0.041
	15	8.0483	0.002	0.088
	20	8.1120	0.003	0.171
	25	8.1642	-0.008	-0.392
CENAM	21.9	8.15	0.017	0.040
CMI	14.85	8.00	-0.045	-0.148
	19.91	8.10	-0.008	-0.027
	24.6	8.25	0.083	0.275

Table 7.4. E_n value of the ceramics gauge block (L_0=20 mm)

8. Conclusions

The difference of reported uncertainty between participants was so large. It seemed that the cause of the difference is not only the difference of measurement capacity but also the measurement condition, for example ΔT. Therefore, the determination of suitable key reference values was difficult, because given temperatures at which thermal expansion coefficient were measured did not correspond each other. In this report the weighted least square method was utilized, however there might be how to decide a better reference value.

Most reported results were corresponded each other within their measurement uncertainty. However, the some given results for the steel gauge block had the systematic deviation of $0.03\times 10^{-6}℃^{-1}$～$0.07\times 10^{-6}℃^{-1}$ from the key reference value. The deviation of α is corresponding to the uncertainty of the determination of temperature, $U(\Delta T)$, of 14 mK～24 mK, when ΔT = 5 ℃. On the other hand, the corresponding uncertainty of the length determination , $U(\Delta L)$, is 15 nm～25 nm. The estimated $U(\Delta L)$ value is too large to think to be cause of the deviation from α_{ref} . As the result, it is considered that the deviation in given results by some participants were mainly caused by the uncertainty of temperature measurement of gauge blocks.

9. Reference

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10. Appendix

METAS has submitted an additional report on the deviation of his result from reference value later. The additional report is as follows;

* METAS does not operate the equipment for thermal expansion measurements on a regular basis, because there is almost no customer request, and it does not provide any regular CTE measurement service under its quality system. METAS does not claim CMCs in this field.

* METAS takes the observed deviations from the reference values seriously and will investigate the problems before the instrument is put into operation next time. From the past EUROMET comparison, where METAS was pilot laboratory and achieved good results, we still have samples with well known CTE values, which can be used for checking.

* The length measurements are done by interferometry in vacuum. It is therefore very unlikely to be the source of the deviations.

* Since for short gauge blocks the results were much better than for long gauge blocks, the calibration of the temperature sensors is also unlikely to be the error source.
* The largest problem is the temperature gradient on the gauge blocks, which is much more important for long than for short samples. In particular, this gradient is not linear. The problem is then to determine the average temperature of the gauge block. This has been done by taking the arithmetic mean from three uniformly distributed temperature sensors fixed on the gauge blocks. For a non-linear temperature distribution this does not give the average temperature. A simulation has shown that this effect would account for up to the half of the observed deviations.
* Another problem might be the temperature equilibrium after temperature changes. Further investigations will be needed in order to estimate the minimum required stabilization time at each step.