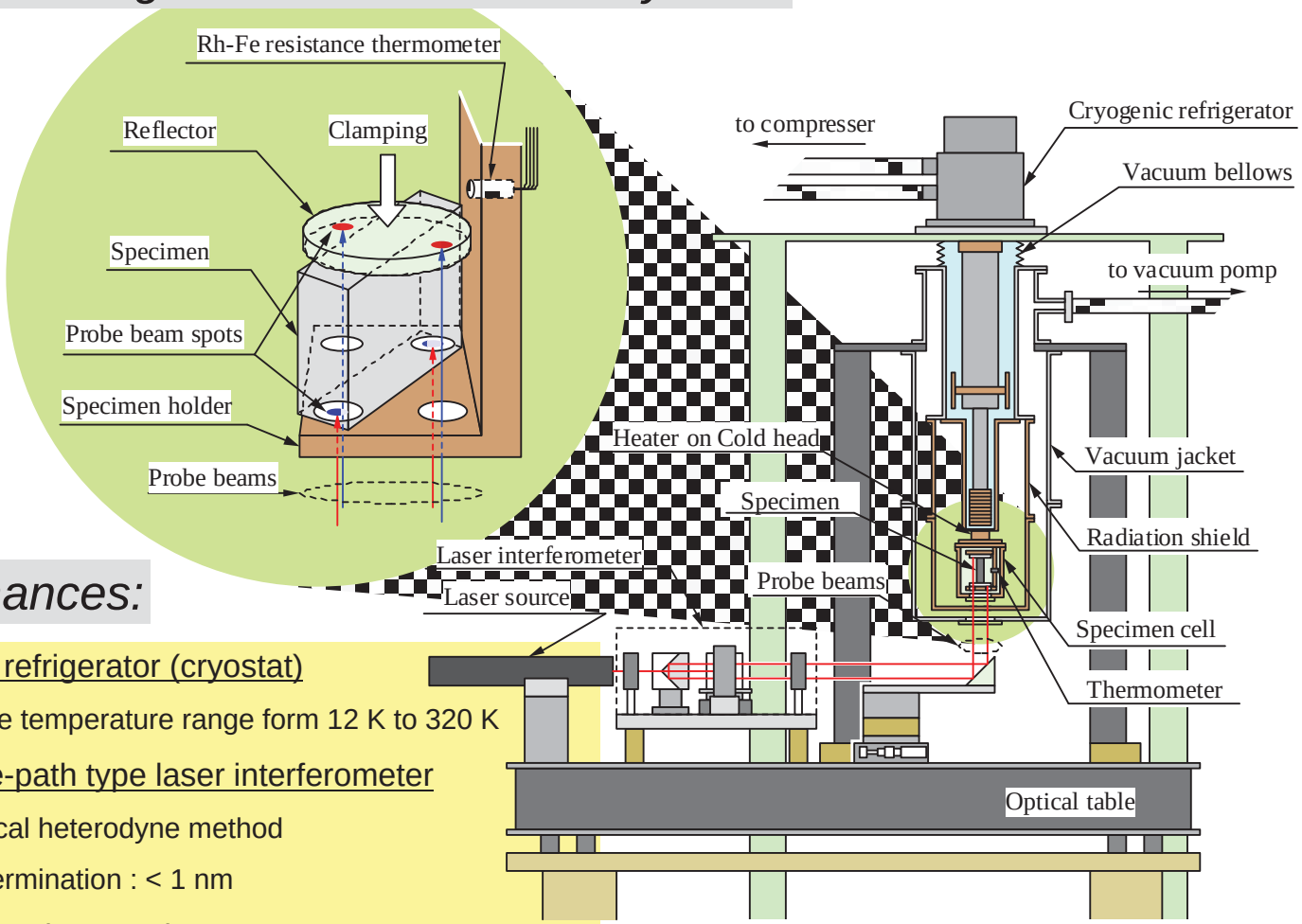
Low-Temperature Interferometric Dilatometer using a Cryogenic refrigerator

Schematic diagram of measurement system



Performances:

○4K-GM refrigerator (cryostat)

- Controllable temperature range form 12 K to 320 K

○ Double-path type laser interferometer

- By an Optical heterodyne method
- Fringe determination : < 1 nm

○ Rh-Fe resistance thermometer + Resistance bridge

- Temperature determination : ～ 6 mK

Measurement results *by this instrument*:

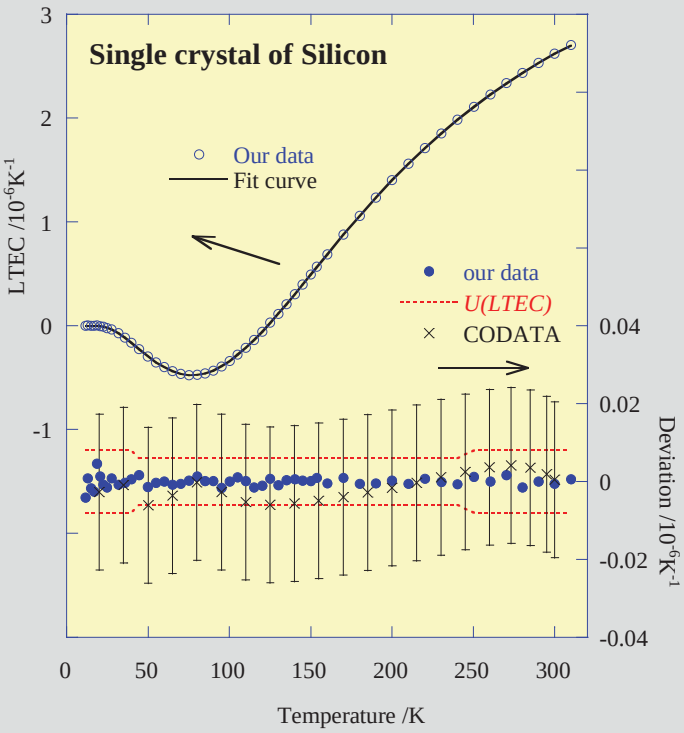


Figure 1. LTEC of FZ silicon (single crystal)

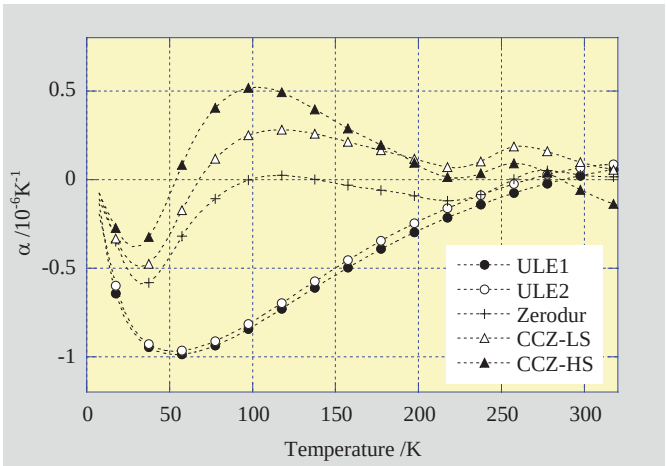


Figure 2. LTEC of some low-LTEC glasses

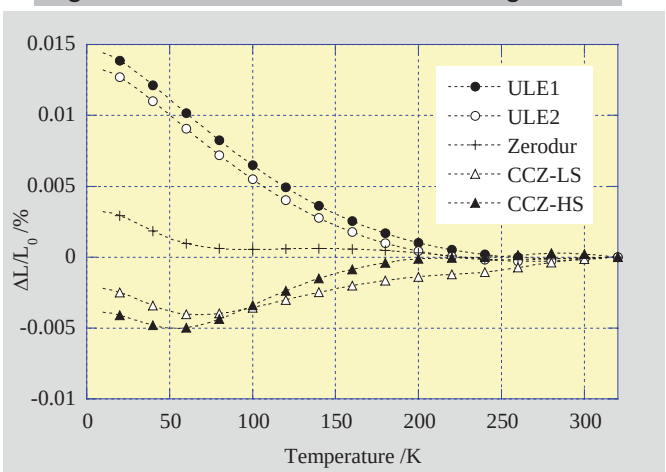
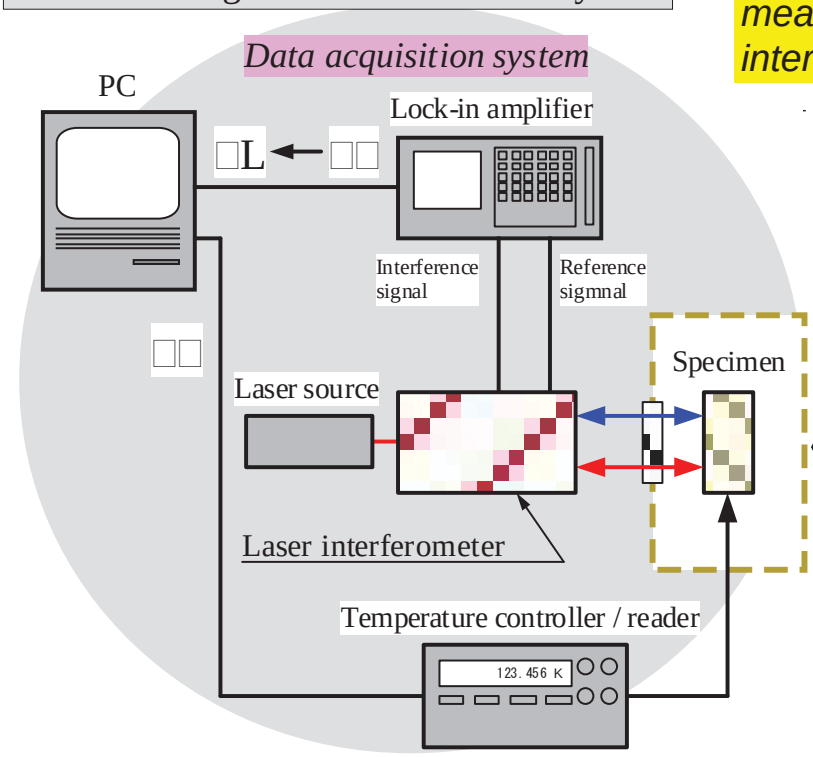


Figure 3. $\Delta L/L_0$ of some low-LTEC glasses

Measurement system for linear thermal expansivity of solids by an absolute measurement method in NMIJ / AIST

Schematic diagram of measurement system

- Our measurement system has achieved CTE measurement with high precision by a laser interferometer.



Temp. control and Temp.measurement

At high temperature range
→ High temperature furnace
Temp. measurement:
Elemental thermocouple (Au-Pt TC) at T <1000℃
Radiation thermometer at T > 1000℃

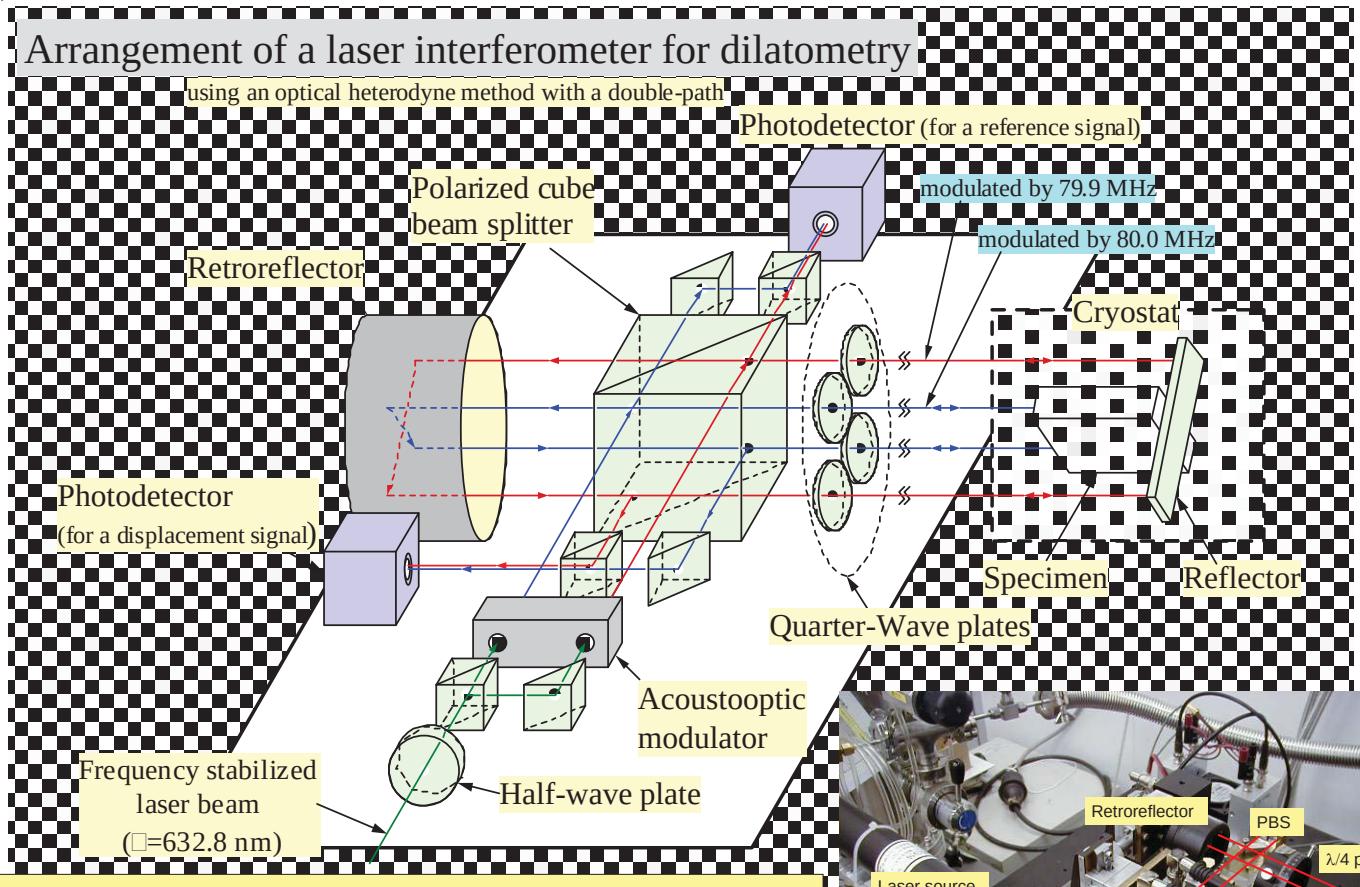
At room temperature range
→ Thermal bath by using thermo-electric device
Temp. measurement:
Pt resisntance thermometer @ r. t. ±20 ℃

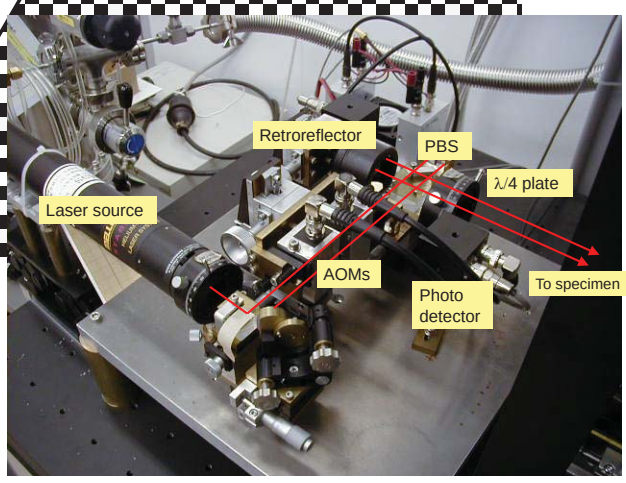
At cryogenic temperature
→ Cryostat by using cryogenic refrigerator
Temp. measurement:
Rh-Fe (or Pt) resistance thermometer
at 20 K < T < 320 K

Key technique: Displacement measurement by a laser interferometer

Arrangement of a laser interferometer for dilatometry

using an optical heterodyne method with a double-path





Features:

- **Optical heterodyne method with high resolution**
- **Acousto-optic modulators to generate probe beams**
- **Auto-cancellation of the effect of specimen tilt** on displacement measurement

Development of New Reference Material for Thermal Expansion Measurements at Cryogenic Temperature

We developed a new certified reference material (CRM; NMIJ CRM 5803) for thermal expansion measurements in solid materials at cryogenic temperature. This CRM can be used to calibrate a pushrod dilatometer and a thermo-mechanical analyzer, to estimate the measurement uncertainty, and to confirm the validity of measurement methods. It was made from a single crystal of silicon and can be available in the temperature range from 20 K to 300 K.

Photo 1 CRM; NMIJ CRM 5803-a

Procedure of making CRM:

1) Preparation of a silicon ingot
2) Sampling of specimens for calibration
3) CTE Calibration by the laser-interferometoric dilatometer
4) Evaluation of the uncertainty in the calibration values
5) Evaluation of homogeneity and long-term stability on CTE value
6) Determination of the certified value and its uncertainty

1), 2) The material of CRM is the single crystal of silicon which was made by a floating zone method. The purity of the silicon ingot is more than 99.99998 %. Some specimens were sampled from the ingot to calibrate CTE value.

Photo 2. FZ-silicon ingot (ϕ5 inchx130 mm)

3) The specimens were measured by using a laser-interferometric dilatometer for cryogenic temperature. The CTE values of the specimens were measured by absolute measurement method using a laser interferometer.

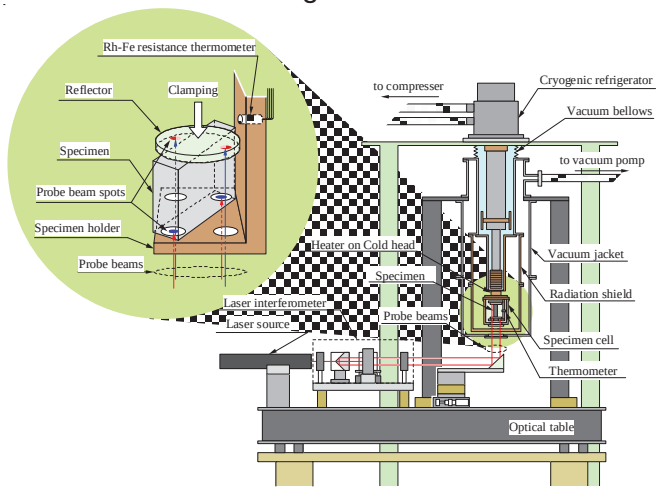
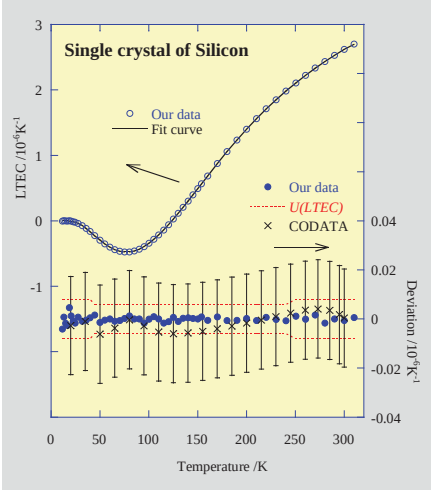


Figure 1. Schematic diagram of Laser-interferometric dilatometer

4) The measurement uncertainty of each obtained value was evaluated respectively. The uniformity of thermal expansivity in the ingot was also estimated by analysis of variance.

Conditions:	L_0 /m = 0.0200685	→→	α /10^{-6}K^{-1} = 1.4124		
	T_{AVE} /K = 200.0174		Data file name: 070716p		
	ΔT /K = 10.0087		Step No.: 40		
Factor of uncertainty (x_i)	Detail	Uncertainty $u(x_i)$		Unit	$u_i(\alpha)$	Type
Fringe detamination (ϕ_1 and ϕ_2)	Resolution of phase detection	$u(\phi_1)$ = 1.0E-05		rad	1.3E-12	B
		$u(\phi_2)$ = 1.0E-05			1.3E-12	
	Reproducebility of phase change	$u(\phi_1)$ = 2.3E-02			2.9E-09	A
		$u(\phi_2)$ = 2.3E-02			2.9E-09	
Optical arraiment (ε)	Inclination of optical axis	$u(\varepsilon)$ = 3.3E-07		1	4.7E-13	B
Wave length of laser souce (λ_1 and λ_2)	Stability of wave length	$u(\lambda_1)$ = 1.6E-15		m	2.5E-10	B
		$u(\lambda_2)$ = 1.6E-15			2.5E-10	
Refrective index (n_1 and n_2)	Change of refractive index of themal exchange gas	$u(n_1)$ = 2.8E-09		1	2.8E-10	A
		$u(n_2)$ = 2.8E-09			2.8E-10	
Determination of L_0 (L_0)	Resolution of linear gauge	$u(L_0)$ =	2.5E-06	m	1.7E-10	B
	Calibration of liniar gauge		8.1E-08		5.7E-12	B
	Temperature of specimen at measurement		7.8E-08		5.5E-12	B
Determination of temperature (ΔT, δT and T_D)	Detection of temperature	$u(\Delta T)$ =	2.5E-03	K	3.5E-10	B
	Correction of temperature difference between specimen and termometer	$u(\delta T)$ =	9.4E-03		1.3E-09	B
	Stability of temperature control	$u(\Delta T)$ =	6.4E-05		9.1E-12	A
	Calibration of thremometer	$u(T_D)$ =	1.0E-02		1.7E-10	A
		Combined standard uncertainty/K^{-1}: $u_c(\alpha)$=			4.3E-09	
Date: 01/17/07		Expanded uncertainty/K^{-1} (Converage factor; k =2): U =$ku_c(\alpha)$=			8.6E-09	
		Relative expanded uncertainty: (U /α)=			0.61%	

Table. Error budget sheet of LTEC measurement



4)- 6) The certified value were determined by a weighted least square method as functions of temperature and it is in good agreement with the recommended value of CODATA. The uncertainty of the certified value is also much smaller than that of previous reference materials.

Certified value and uncertainty

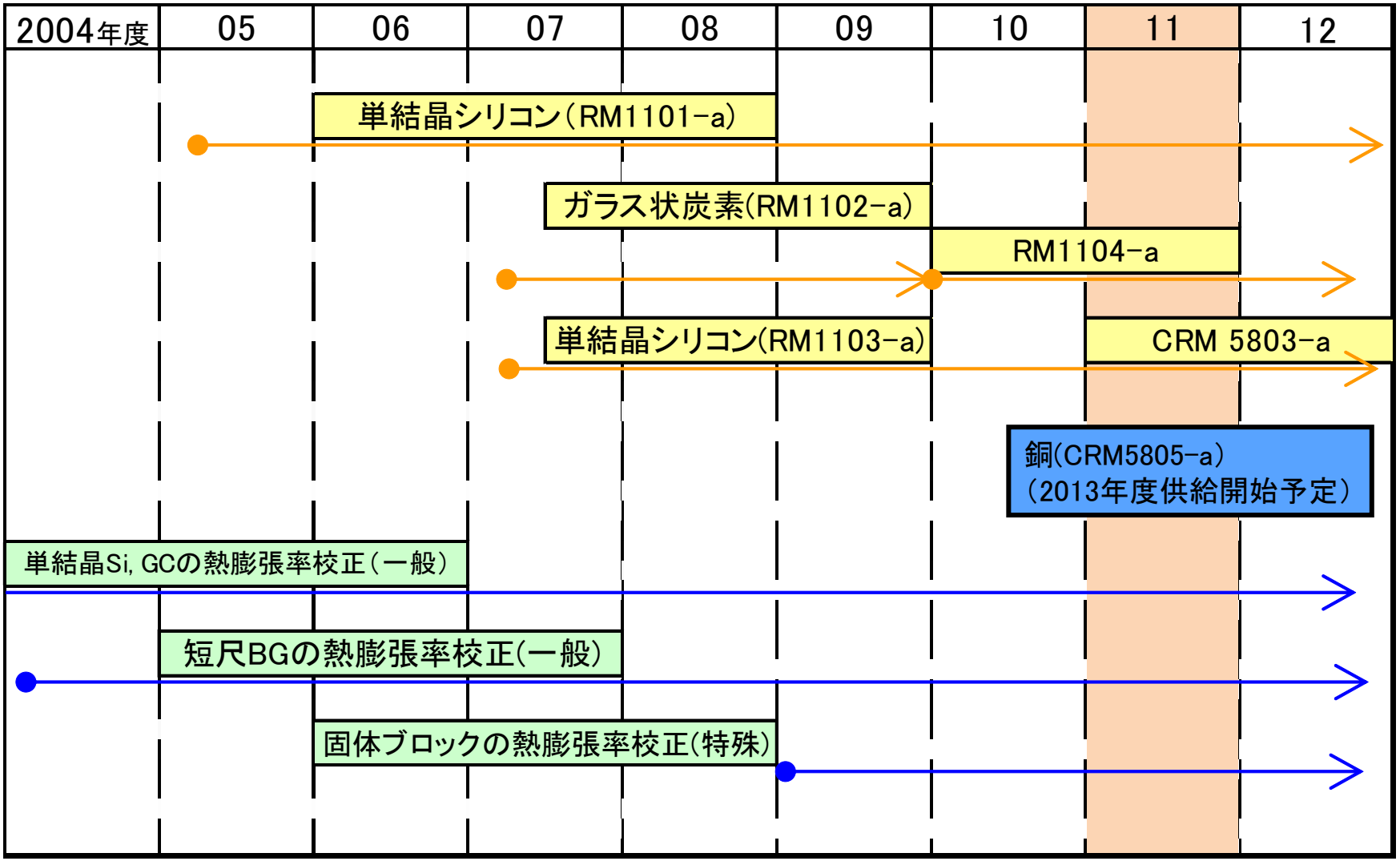
T (K)	α(10^{-6}K^{-1})	U(10^{-6}K^{-1})
20	-0.0038	0.0080
50	-0.2947	0.0053
100	-0.3420	0.0050
150	0.4907	0.0050
200	1.4007	0.0050
250	2.1039	0.0050
300	2.6190	0.0050

※*The certified value is expressed as a function of T on the certificate.*

熱膨張率標準供給項目 詳細(2012. 1現在)

供給形態	名称	適用範囲等	不確かさ等
標準物質※	標準物質名	適用温度範囲	参照値の不確かさ/頒布形状
	単結晶シリコン (NMIJ RM 1101-a)	293 K - 1000 K	相対拡張不確かさ: < 1.2 % 頒布形状:4.5角×L60 mm^3, 9.0角×L60 mm^3
	ガラス状炭素 (NMIJ RM 1102-a)	293 K - 1100 K	相対拡張不確かさ: < 3.3 % 頒布形状:6.0角×L10 mm^3, 6.0角×L20 mm^3
	ガラス状炭素 (NMIJ RM 1104-a)	293 K - 約1600 K	相対拡張不確かさ: 0.91 %～1.7 % 頒布形状:6.0角×L10 mm^3
	単結晶シリコン (NMIJ CRM 5803-a)	20 K -300 K	拡張不確かさ: 6.0×10^{-9} K^{-1} ～ 8.0×10^{-9} K^{-1} 頒布形状:10.0角×L30 mm^3, 10.0角×L60 mm^3
依頼試験	試験名	校正温度範囲**他**	校正・測定能力(k =2)
	・単結晶シリコン もしくはガラス状炭素の 熱膨張率校正	・25×25×t6の単結晶シリコン もしくはガラス状炭素試験片 ・校正温度範囲は293 K-1000 K	2.0×10^{-8} K^{-1}
	・固体ブロックの 熱膨張率校正(特殊)	・20×20×t8の固体ブロック ・校正温度範囲は15 K-320 K	5.8×10^{-9} K^{-1}～4.2×10^{-7} K^{-1}
	・短尺ブロックゲージの 熱膨張率校正	・呼び長20 mm以上かつ100 mm以下のブロックゲージもしくはこれと同等寸法精度形状の固体ブロック ・校正温度範囲は5 ℃ - 35 ℃	$\left[\frac{A\times\sqrt{\alpha^2+B^2}}{\Delta T}+C\cdot\alpha\right]\times10^{-9}\,℃^{-1}$; $A=4.3+\left(\frac{5.9}{L_0-23}\right)^2, B=0.38+\frac{39}{L_0}, C=0.020+\left(\frac{6.3}{L_0}\right)^2$ 尚、L_0<40の場合はA=2.0とする。また、入力量の単位はαが$10^{-6}K^{-1}$, L_0がmm, ΔTがKとする。

今後の整備予定


2004年度
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単結晶シリコン（RM1101−a）
ガラス状炭素(RM1102−a)
RM1104−a
単結晶シリコン(RM1103−a)
CRM 5803−a
銅(CRM5805−a)
（2013年度供給開始予定）
単結晶Si, GCの熱膨張率校正（一般）
短尺BGの熱膨張率校正(一般)
固体ブロックの熱膨張率校正(特殊)