Precise determination of lattice parameter and thermal expansion coefficient of silicon between 300 and 1500 K

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The lattice parameter of high-purity silicon is measured as a function of temperature between 300 and 1500 K, and the linear thermal expansion coefficient is accurately determined. Precise measurements are made by the high-temperature attachment for Bond's x-ray method to a few parts per million. It is found that the temperature dependence of the linear thermal expansion coefficient $\alpha(t)$ is empirically given by $\alpha(t) = (3.725\{1 - \exp[-5.88 \times 10^{-3}(t - 124)]\} + 5.548 \times 10^{-4}t) \times 10^{-6}$ (K^{-1}), where t is the absolute temperature ranging from 120 to 1500 K. It is shown that the lattice parameter in the above temperature range can be calculated using $\alpha(t)$ and the lattice parameter at 273.2 K (0.5430741 nm). Measured values of the lattice parameter and the thermal expansion coefficient for high-purity float-zoned (100 kΩ cm) and Czochralski-grown (30 Ω cm) single crystals are uniformly distributed within $\pm 1 \times 10^{-5}$ nm and $\pm 2 \times 10^{-7}$ K^{-1} with respect to the values obtained from the above empirical formula.

I. INTRODUCTION

The accurate information of the thermal expansion coefficients and the lattice parameters at high temperatures is required for the semiconductor industry as well as physics. For example, thermal expansion coefficients are used to determine whether the matching between semiconductor elements and substrates is good or not over a certain temperature range. Silicon films on single sapphire crystals are strained under compressive stress, and result in a high density of crystal imperfections in the interface. This is due to the difference in thermal expansion coefficients at high temperatures. Thin silicon-oxide layers on silicon often introduce the strain in the material near the interface. Those strain effects have been generally attributed to a large difference in the lattice parameters or the thermal expansion coefficients between the deposit layer and the substrate at high temperatures.

White[1] has proposed the use of high-purity silicon as a reference standard material for the thermal expansion measurement. This is because silicon is available in extremely high purity. It can be used over a wide temperature range because of its high melting point, and it has an isotropic expansion coefficient resulting from its diamond structure. Since its expansivity is relatively small (e.g., at temperatures higher than 1000 K it is smaller than that of diamond[2]), it is also useful as a reference for measurement of other low expansivity materials.[3]

There are 15 references (Refs. 4–18) about the lattice parameter and/or the thermal expansion of silicon above 800 K. The measured temperature ranges are given in Table I. There exist a few data at 1300–1500 K, which are important for oxidization and for diffusion in silicon device processing. These data are largely divergent and less reliable, as seen in Fig. 4. Slack and Bartram[2] made a thermal expansion coefficient versus temperature curve up to 1583 K based on 19 references. White[19] summarized the data up to 1000 K. However, at temperatures higher than 800 K there are large discrepancies between these two sets of data.

X-ray diffraction measurements using powder specimen are common for the determination of the lattice parameters at high temperatures. However, the eccentricity error, the absorption error, and the zero error are unavoidable in this method. For the accurate temperature measurements, the thermocouples are calibrated from lattice parameters of several substances such as silver or copper for which expansion coefficients at high temperatures are known.[7,9,10] However, for the calibration above 850 K, a suitable standard with an accurately determined expansion coefficient has not been available.

In 1960 Bond made precise x-ray measurements on lattice parameters of single crystals at room temperature.[20] His method allows us to determine lattice parameters and thermal expansion coefficients of various single crystals at high temperatures also. None of the previous work uses the Bond method with single crystals above 800 K. We used a high-temperature furnace for Bond's method and performed a preliminary measurement with silicon single crystals. Details of the experimental technique were discussed previously.[21] Using this method, we made the precise measurement of lattice parameters of silicon single crystals and the accurate determination of linear thermal expansion coefficients between 300 and 1500 K.

II. EXPERIMENT

A. Specimen preparation

Five (111) wafers of high-purity dislocation-free silicon single crystals were used in this study. Two of them (A and B) were obtained from a float-zoned (FZ) silicon crystal with an intrinsic resistivity of 100 kΩ cm. The other three (C, D, and E) were obtained from a Czochralski-grown (CZ) silicon crystal with a resistivity of 30 Ω cm. The results of photoluminescence analysis[22] of the starting materials are shown in Table II. Oxygen concentration in the CZ crystal measured by infrared absorption spectroscopy[23] is 9.6×10^{17} cm^{-3}. A specimen, approximately 10×25 mm^2 in area, was obtained

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TABLE I. Previous investigations of lattice parameters and thermal expansion coefficients of silicon made above 800 K.

Authors	Year	Maximum temperature (K)	Lattice parameter	Thermal expansion coefficient	Method	Material	Ref.
Roberts	1981	840	NM	M	Polarization interferometer	R	18
Ibach	1969	850	NM	M	interferometric method	Zone-melted R	15
Dolivo-Dobrovol'skaya et al.	1973	870	NM	M	x-ray diffractometry	S	16
Hall	1961	993	M	M	X.P.C.	P	9
Dismukes et al.	1964	1073	M*	M	X.D.	High-purity P	11
Zhdanova et al.	1967	1073	NM	M	D.L.	R	13
Yim et al.	1974	1086	M	M	X.P.C.	P	17
Maissel	1960	1123	NM	M	Straight forward optical method	S(R)	8
Dutta	1962	1151	M	M	X.P.C.	P	10
Shaw et al.	1965	1173	M	M	X.P.C.	P	12
Schulze	1930	1273	NM	M	D.L.	Vacuum melted R.	4
Schlötterer	1968	1273	NM	M	D.L.	...	14
Skinner et al.	1953	1591	M	M	X-ray diffraction	High-purity P	6
Mauer et al.	1957	1612	M	M	X.D.	99.97% P	7
Gonser et al.	1940	1673	NM	M	D.L.	99.84% R	5

M = measured; NM = not measured; ⋯: not described;
X.P.C. = x-ray powder camera (Unicam); X. D. = X-ray diffractometer; D. L. = dilatometer.
S = single crystal; P = powder sample; R = rod.
* Only at room temperature.

from each wafer. The thickness was 0.8 mm for the FZ wafer and 0.3 mm for the CZ wafer. They were deeply etched to remove surface damage completely. The specimen was mounted softly on the specimen holder with a light boron nitride wedge at one end to prevent strain in the specimen.[21]

B. Measurement method

An automatic lattice parameter precision measurement system (Rigaku Co.) equipped with the high-temperature attachment for Bond's method was used to measure lattice parameters of the specimens at high temperatures. This instrument was capable of determining the specimen temperature to an accuracy of $\pm$ 1.6 K below 1000 K and $\pm$ 2.3 K at 1500 K.

Table III shows the lattice parameters of the specimen B measured at 11 fixed temperatures. The x-ray measurement using (444) reflection with CuKα$_1$ [λ_{1973} = 0.15405981 nm $\pm$ 1 ppm (Ref. 24)] was temperature stabilized within $\pm$ 0.5 K. At elevated temperature, measurements were made at 10^{-4} Torr. A diffraction peak was step-scanned in an increment of 5″ and the counting time at each angle point was 20 sec. About 30 data points were obtained in each scan.

We used a computer program which located the peak angle ω_1 or ω_2 of the diffraction curves analytically by least-squares fitting of the curves to the Cauchy distribution function. The Bragg angle Θ_B is given by $\Theta_B = 1/2$ ($180° - |\omega_1 - \omega_2|$). Three repeated measurements of Θ_B were made at each temperature, and the average value was taken. The lattice parameter a_{obs} was calculated from Θ_B. The corrected lattice parameter a_{corr} was obtained by making the refraction correction Δa_{ref}, the axial divergence correction Δa_{div}, and the Lorentz-polarization correction Δa_{Lp}.

The measurement was made at room temperature first, then at elevated temperatures. After the measurements at the highest temperature, the room temperature measurement was carried out again to check the reproducibility. This check is necessary because the specimen properties can be changed by the generation of surface or bulk damage during the high-temperature process. The surface contamination of the measured specimen was analyzed by the electron-probe x-ray microanalyzer (EPMA).

III. RESULTS

Linear thermal expansion of solid is usually measured in terms of the change in length l with respect to the length l_0

TABLE II. Photoluminescence analysis data of impurities in starting silicon materials.

Material	Resistivity (Ω cm)	Impurity concentration (cm^{-3})	
		Boron	Phosphorus
FZ-Si (A and B)	100 000	4.12×10^{12}	3.72×10^{12}
CZ-Si (C, D and E)	30	3.40×10^{14}	3.50×10^{12}

TABLE III. Result of measurements of lattice parameter and linear thermal expansion coefficient of FZ single-crystal silicon (sample B). Three repeated measurements of the parameter were made at each temperature using (444) reflection of a (111) wafer with $CuK\alpha_1$. λ_{1973} of $CuK\alpha_1 = 0.15405\ 81$ nm $\pm$ 1 ppm (Ref. 24). Δt is the accuracy of temperature determination.

t (K)	Δt (K)	Θ_B (degree)	a_{obs} (nm)	Δa_{ref} $\times 10^{-6}$ (nm)	Δ_{div} $+ \Delta a_{Lp}$ $\times 10^{-6}$ (nm)	a_{corr} (nm)	standard deviation $\times 10^{-7}$ (nm)	$\alpha(t)$(exper) Temp. (K)	$\alpha(t)$ ($\times 10^{-6}$ K^{-1})
298.5	0.6	79.3114	0.5431018	4.2	1.3	0.5431074	2		
		79.3113	0.5431019						
		79.3111	0.5431022					336.0	2.69
373.5	0.7	79.2500	0.5432119	4.2	1.3	0.5432171	3		
		79.2502	0.5432116						
		79.2503	0.5432114					433.3	3.39
493.1	1.1	79.1285	0.5434320	4.2	1.2	0.5434375	1		
		79.1284	0.5434322						
		79.1284	0.5434321					546.2	3.74
599.2	1.0	79.0107	0.5436478	4.2	1.2	0.5436533	2		
		79.0108	0.5436477						
		79.0106	0.5436480					674.6	3.98
749.9	1.5	78.8352	0.5439738	4.2	1.2	0.5439789	4		
		78.8356	0.5439731						
		78.8355	0.5439734					853.2	4.12
956.5	1.1	78.5914	0.5444361	4.2	1.2	0.5444415	0		
		78.5914	0.5444361						
		78.5914	0.5444361					1059.5	4.27
1162.5	1.0	78.3450	0.5449140	4.2	1.2	0.5449192	2		
		78.3451	0.5449138						
		78.3452	0.5449135					1236.9	4.43
1311.3	1.0	78.1641	0.5452719	4.2	1.2	0.5452772	2		
		78.1641	0.5452718						
		78.1643	0.5452715					1364.3	4.44
1417.3	1.1	78.0364	0.5455279	4.2	1.3	0.5455326	7		
		78.0369	0.5455269						
		78.0370	0.5455267					1465.3	4.76
1513.2	1.2	77.9146	0.5457751	4.2	1.3	0.5457805	5		
		77.9144	0.5457754						
		77.9148	0.5457745						
371.7	0.7	79.2559	0.5432013	4.2	1.4	0.5432068	1		
		79.2559	0.5432014						
		79.2560	0.5432012						

at some temperature t_0. Usually t_0 is 273.2 K. The linear thermal expansion coefficient $\alpha(t)$ is given by $\alpha(t) = 1/l_0(dl/dt)$. In practice, $\alpha(t)$ is calculated by

$$\alpha(t) = \frac{1}{l_0}\frac{(l_1 - l_2)}{(t_1 - t_2)}, \tag{1}$$

where l_1 and l_2 are lengths at temperatures t_1 and t_2, and $t = (t_1 + t_2)/2$ K. In general there is a difference between the macroscopic length change with temperature when measured optically or mechanically and the lattice parameter change measured by x rays. However, this difference is believed to be small enough for the diamondlike crystal such as silicon.[2] In cubic crystals $\alpha(t)$ is a scalar, independent of crystal direction.

From $\alpha(t)$ the total fractional length change between t_0 and t can be determined as

$$\frac{(l - l_0)}{l_0} = \int_{t_0}^{t} \alpha(t)dt. \tag{2}$$

Results of measurements for the FZ specimen B are shown in Table III. Accuracy of specimen temperature Δt was obtained from the comparison with a standard PR$_{13}$ thermocouple.[21] Experimental values of linear thermal expansion coefficient $\alpha(t)$ (exper) were determined by Eq. (1). Results of the FZ specimen A and the CZ specimens C and D are shown in Figs. 2 and 5.

After the high-temperature measurements it was found that the specimen surface was deposited with a gray thin layer of some material. Boron was detected by EPMA analysis. We consider that this thin layer is due to the deposition of boron from a boron nitride pipe which covered the specimen holder.[21] The lattice parameters measured at room temperature before and after the high-temperature measurements agree within the error of one part in 10^6 as shown in Fig. 2 (specimen D), which indicates that the thin layer gives no appreciable influence on the measurement.

The lattice parameter of the CZ specimen E was measured at room temperature first, and then held at 1068 K for 55 h. After that, it was measured again at room temperature. The good agreement between the two indicates that the lat-
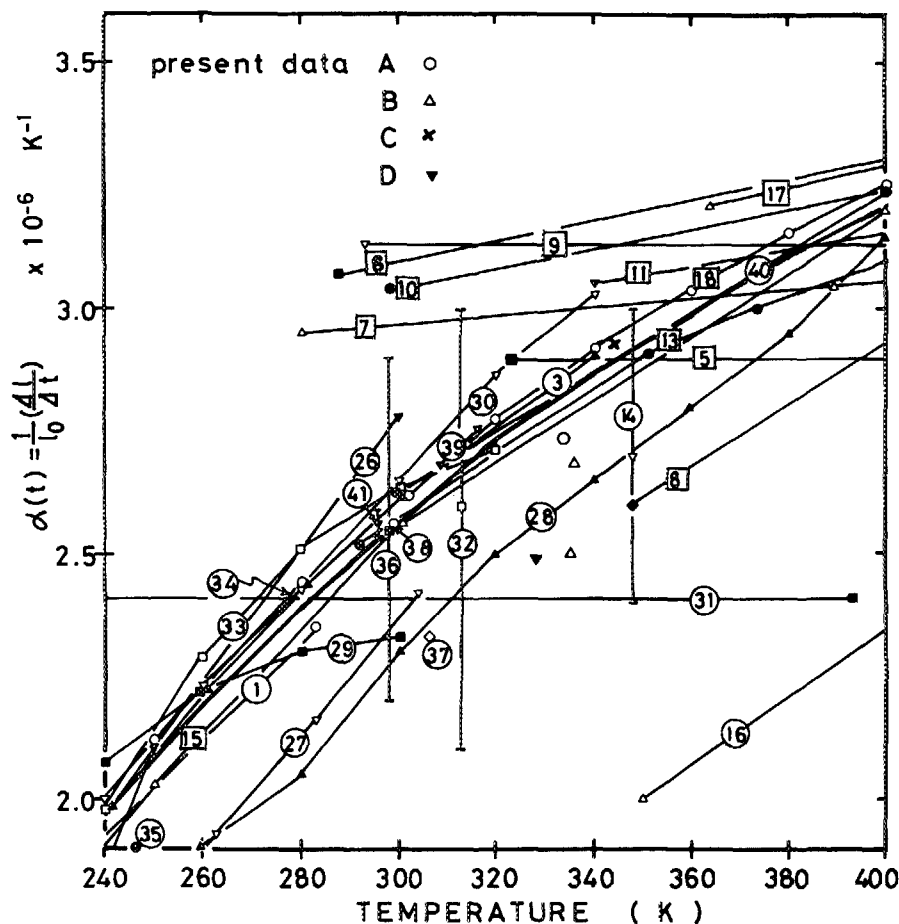
FIG. 1. Linear thermal expansion coefficients of silicon near room temperature taken from 31 references (Numbers in the figure are reference numbers) and five present measurements. The bold solid curve shows the empirical equation (5). □ shows data from references in Table I.

tice parameter was not influenced by the generation of Si-O complex in the CZ crystal[25] (see Fig. 2).

IV. DISCUSSION

A. Data at 298.2 K

It is necessary to obtain the linear thermal expansion coefficient at 298.2 K for the determination of the lattice parameter at the same temperature. Figure 1 shows the linear thermal expansion coefficients near room temperature calculated by Eq. (1) together with data from 31 references.[1,3,5-11,13-18,26-41] Among these, 14 sets of data from Refs. 1, 3, 13, 15, 18, 30, 32–34, 36, and 38–41 were judged to be reliable, because they differ by no more than $\pm 10^{-7}$ K^{-1}. From these 14 sets of data, we thus estimate the linear thermal expansion coefficient at 298.2 K, $\alpha_{298.2}$, to be $(2.59 \pm 0.05) \times 10^{-6} K^{-1}$. The data from references listed in Table I, except those from Refs. 13–15 and 18, are scattered as shown in Fig. 1, and are judged to be less reliable.

Figure 2 shows our experimental results on lattice parameters of the high-purity FZ (A and B) and CZ (C, D, and E) crystals. The solid line in Fig. 2 shows the temperature dependence of the lattice parameter $a(t)$ using $\alpha_{298.2}$ $= 2.6 \times 10^{-6} K^{-1}$. These data are within $\pm 6 \times 10^{-7}$ nm from the solid line. The lattice parameter at 298.2 K is found to be $a_{298.2} = 0.5431073 \pm 6 \times 10^{-7}$ nm.

Figure 3 shows the lattice parameters at 298.2 K from 34 references[6,7,9-12,17,20,24,32,35,36,41-62] together with our value. These values at 298.2 K were deduced by using values of
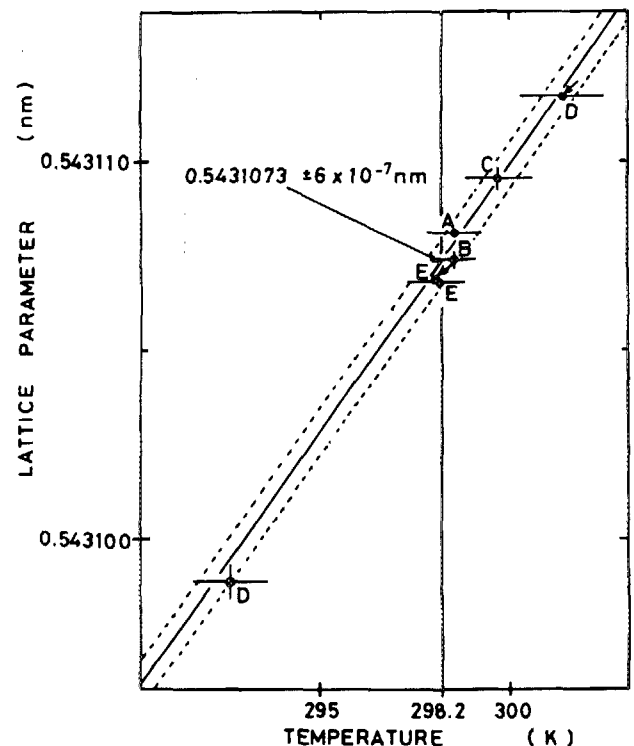

FIG. 2. Lattice parameters of silicon measured near room temperature. A and B are for FZ, 100 kΩ cm Si; C, D, and E are for CZ, 30 Ω cm Si. Two data points with an arrow are the data taken after high temperature measurements. The solid line shows the temperature dependence of the lattice parameter obtained by Eq. (1) and $\alpha_{298.2} = 2.6 \times 10^{-6} K^{-1}$. The dotted lines are drawn to include all data points between them.
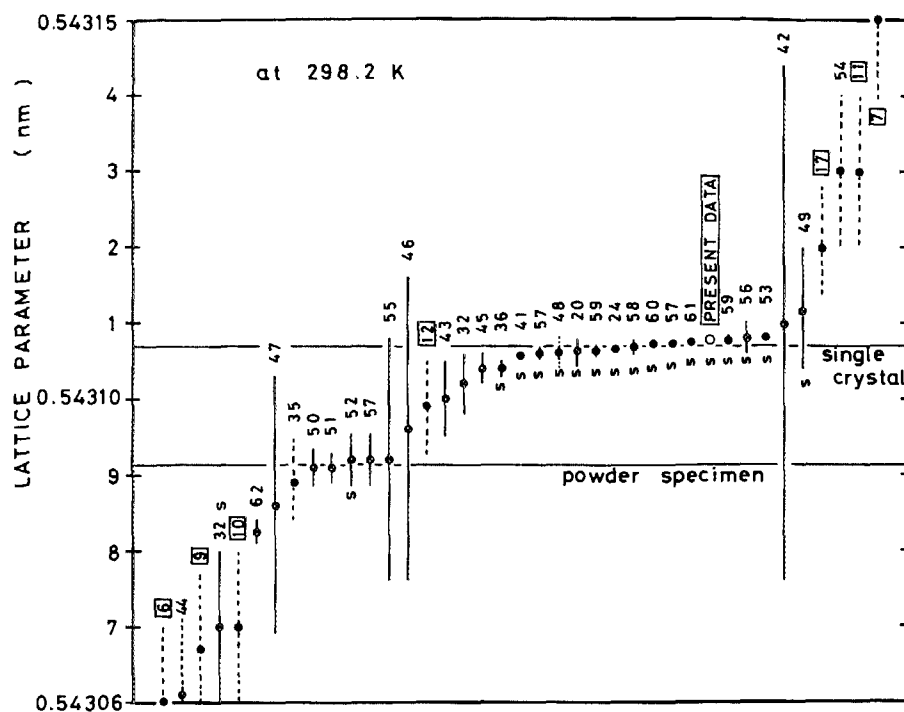
FIG. 3. Distribution of other author's values (with reference numbers) of the lattice parameter of silicon. The error limit is indicated by vertical bars. These values at 298.2 K were deduced by us using $\alpha_{298.2} = 2.6 \times 10^{-6}\,\mathrm{K}^{-1}$ and $\lambda_{1973} = 0.15405981$ nm. The letter S denotes the value of a single crystal. The other data are for powder specimens. The horizontal lines show the reliable values of single and powder specimens. □ shows the data from references in Table I.

$\alpha_{298.2}$ and λ_{1973} as described above. We observe that there is a systematic difference of lattice parameters, about 1.5×10^{-5} nm, between powder and single-crystal samples. This difference was also pointed out by Hubbard et al.[59] Present values agree with other author's data on single crystal. Data at 298.2 K obtained from references listed in Table I exhibit large dispersion compared with the value of 0.5430915 nm for powder specimens shown in Fig. 3.

B. Data at high temperatures

In Fig. 4, the calculated linear thermal expansion coefficients were plotted together with data from references listed in Table I and Lyon et al.'s data.[3] Our data are coincident with a smooth curve by Slack and Bartram[2] which was made by the best fitting of data from 19 references. To fit our data, we assume the following empirical formula:

$$\alpha(t) = (C_1\{1 - \exp[-C_2(t - C_3)]\} + C_4 t) \times 10^{-6}\ (\mathrm{K}^{-1}). \tag{3}$$

The lattice parameter $a(t)$ at t K is given by

$$a(t) = a_0\left[\int_{273.2}^{t} \alpha(t)dt + 1\right]\ (\mathrm{nm}), \tag{4}$$

where a_0 is the lattice parameter at 273.2 K.[2] This expression was fitted to our data in Fig. 4 by the least-squares technique. The result is

$$\alpha(t) = (3.725\{1 - \exp[-5.88 \times 10^{-3}(t - 124)]\}$$
$$+ 5.548 \times 10^{-4} t) \times 10^{-6}\ (\mathrm{K}^{-1}). \tag{5}$$

The solid line in Fig. 4 shows the curve by this formula. The lattice parameter at any temperature between 298.2 and 1500 K can be calculated from Eq. (4) using $a_0 = 0.5430741$ nm.

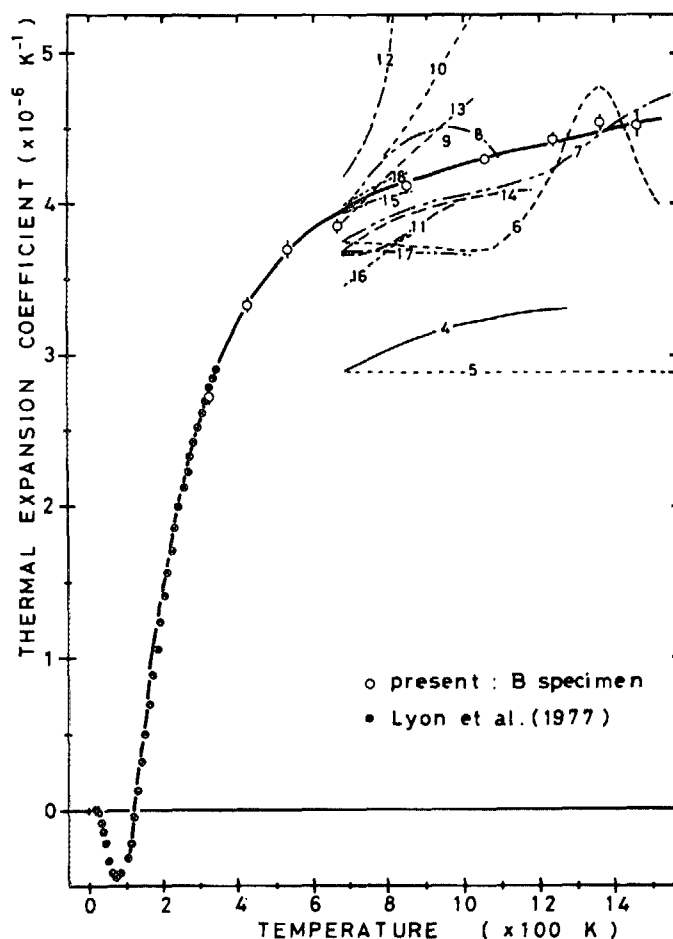Figure 5(a) shows the difference between the experi-

FIG. 4. Linear thermal expansion coefficient vs temperature. Present results (O) are shown together with data (with reference numbers) from literatures in Table I and data by Lyon et al. (Ref. 3). These were recalculated by Eq. (1) using λ_{1973}. The solid curve is for the empirical Eq. (5).

(a)

$C_1 = 3.725$
$C_2 = 5.88 \times 10^{-3}$
$C_3 = 124$
$C_4 = 5.548 \times 10^{-4}$
$a_0 = 0.5430741$

specimen A ○
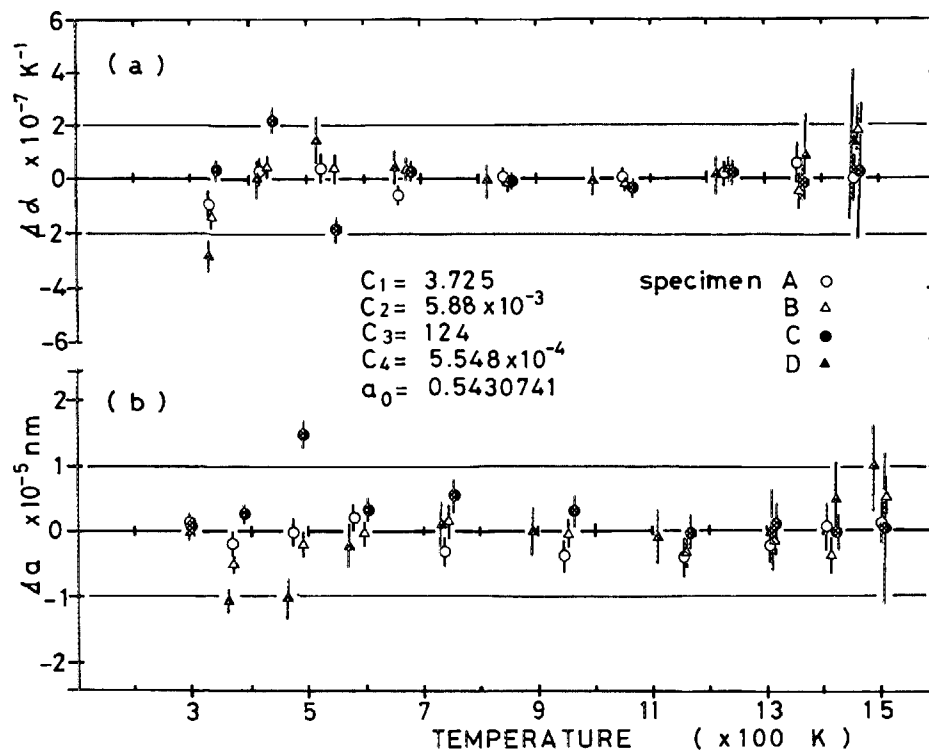B △
C ●
D ▲

(b)

TEMPERATURE ($\times 100$ K)

FIG. 5. Deviations of measured linear thermal expansion coefficients and lattice parameters from the calculated values by the empirical Eqs. (4) and (5). (a) Deviation of the linear thermal expansion coefficient; (b) deviation of the lattice parameter. The error bars show the standard deviation of the thermal expansion coefficient and the lattice parameter measured at each temperature.

mental values determined by Eq. (1) and the calculated values from the empirical formula (5). Figure 5(b) shows the differences between the measurements and the calculations by Eqs. (4) and (5). $\Delta a(t)$ of the FZ specimens (A and B) and the CZ specimens (C and D) are distributed randomly within $\pm 2 \times 10^{-7} K^{-1}$. The experimental values of $a(t)$ agree with the calculations within $\pm 1 \times 10^{-5}$ nm. No difference is observed between FZ and CZ crystals.

TABLE IV. Comparison of reported data and calculated values of $\alpha(t)$ and $a(t)$ from empirical Eqs. (4) and (5).

Temp. (K)	$\alpha(t)(\times 10^{-6} K^{-1})$ Reported data	Calculated value	$a(t)$ (nm) Calculated value
100	− 0.339	− 0.51	0.5429670
120	− 0.057	− 0.02	9643
140	0.306	0.41	9664
160	0.689	0.80	9730
180	1.061	1.14	9835
200	1.406	1.45	9977
220	1.715	1.73	0.5430150
240	1.986	1.97	0352
260	2.223	2.19	0578
280	2.432	2.39	0823
298.2		2.55	1072
300	2.616	2.57	1092
320	2.772	2.73	1383
340	2.909	2.87	1688
360	3.041	2.99	2004
380	3.153	3.11	2336
400	3.253	3.21	2677
500	3.614	3.59	4537
600	3.842	3.83	6557
700	4.016	3.99	8685
800	4.151	4.10	0.5440882
900		4.19	3130
1000		4.26	5423
1100		4.32	7754
1200		4.38	0.5450121
1300		4.44	2514
1400		4.50	4943
1500		4.56	7403

Reported data at 100–340 K: Lyon et al. (1977) (Ref. 3); data at 360–800 K: Roberts (1981) (Ref. 18).

$\alpha(t)$ obtained by Eq. (5) is coincident within $\pm 1 \times 10^{-7}$ K^{-1} above about 120 K with that by Lyon *et al.*[3] (see Fig. 4 and Table IV). Equation (4) is applicable between 120 and 1500 K.

The major cause of the experimental error comes from the temperature measurements. Since the measurement error of the lattice parameter at a temperature is less than $\pm 7 \times 10^{-7}$ nm, the accuracy will become higher by improving the temperature determination.

V. CONCLUSIONS

(1) The lattice parameters of FZ and CZ silicon single crystals were accurately measured in the temperature range between 300 and 1500 K. We determined the thermal expansion coefficient of crystal silicon to be $(2.59 \pm 0.05) \times 10^{-6}$ K^{-1} at 298.2 K.

(2) We obtained empirical formulas for $\alpha(t)$ and $a(t)$, which are applicable between 120 and 1500 K.

(3) No difference in $\alpha(t)$ and $a(t)$ was found between pure FZ and CZ crystals. In the measurements we found no influence by the generation of Si-O complex in CZ specimen.

(4) The present result makes it possible to use silicon as a thermal expansion reference standard from 120 to 1500 K with an uncertainty of less than $\pm 2 \times 10^{-7}$ K^{-1}. We can further expand it to the temperature range from 14 to 120 K using the data by Lyon *et al.*[3]

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