

Third-Order Elastic Constants of NaF[†]

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(Received 10 November 1971)

The complete set of third-order elastic constants for single crystals of NaF was determined from the hydrostatic-pressure and uniaxial-stress derivatives of natural sound velocities measured with a two-crystal ultrasonic-interference technique. There appeared to be no dislocation motion during the uniaxial measurements since changes in the natural sound velocities with stress were linear, and data recorded before and after a γ irradiation agreed within experimental error. The Cauchy relations were not satisfied revealing that noncentral forces between the ions are appreciable. Calculations of the elastic constants were carried out according to the Born model. The high-temperature Grüneisen constant and thermal-expansion coefficient were determined from the elasticity data and the latter compared with experimental results.

I. INTRODUCTION

The primary concerns of the present work are the measurement and calculation of the complete set of third-order elastic constants for single crystals of NaF. The experimental constants were determined from both the hydrostatic-pressure and uniaxial-stress derivatives of natural sound velocities measured with a two-crystal ultrasonic-interference technique. Results are expressed according to the thermodynamic definition of Brugger.^{1,2}

While applying the uniaxial stresses, dislocation motion can become significant when the external pressure has resolved shear-stress components in the primary-slip systems. If the ultrasonic wave also causes the same shear forces the dislocation may move, and this contributes to the measurement of the third-order moduli.³ However, dislocation motion can be effectively eliminated by damaging the crystal structure slightly using, for example, neutron⁴ or γ ⁵ irradiation or doping.⁶ The γ irradiation creates color centers in alkali halides and these vacancies impede the motion of a dislocation. This method has been used successfully by Swartz⁷ in taking ultrasonic data on NaCl. Another way to at least partially avoid dislocation effects is to stress externally in directions for which there are no resolved shear forces in the primary-slip systems, such as the $\langle 111 \rangle$ direction for alkali halides of the NaCl class.⁸ Additional modes of propagation can be included because there exist elastic waves which have no shear-stress components in the easy slip direction regardless of the external stress. These are the longitudinal mode along $\langle 111 \rangle$ and the C_{44} shear mode along $\langle 011 \rangle$.⁷ But it is well known that the nature of the dislocation-insensitive equations coupling the constants is such that only five linear combinations can be solved for explicitly. Therefore, at least one dislocation-sensitive mode must be measured. For the present crystals it was decided to try both hydrostatic and uniaxial-

stress measurements, γ irradiate with a dose of 3000 R, and repeat all uniaxial modes.

The final elastic constants display a strong deviation from Cauchy conditions revealing that noncentral forces between the ions are appreciable. With this knowledge, it would be interesting to measure the third-order moduli of an alkali halide assumed to have large covalent forces, such as LiF, to compare the pattern of its constants with that of NaF. Certainly the work of investigating the anharmonic properties of the alkali halides is not complete, however, until experimental higher-order elastic constants exist for all of them. Only then will suitable correction terms be developed and applied to the Born model to account for noncentral forces.

II. EXPERIMENTAL PROCEDURE AND RESULTS

Two NaF single crystals were purchased from Harshaw Chemical Company, both having (111) , $(01\bar{1})$, and $(2\bar{1}1)$ faces and $15 \times 16 \times 17$ mm dimensions. The specifications also included $\pm 2^\circ$ orientation, 3–6' parallelism for opposite faces, and a flatness of $2\frac{1}{2}$ –5 wavelengths of sodium light. The orientation was verified to be within these limits by Laue back-reflection photographs. Further polishing was necessary, however, to eliminate ultrasonic interference between the two samples when propagating the same type of wave in the same direction under standard conditions. This was accomplished by fastening the crystals to steel holders with Duco cement and polishing on a Brown & Sharpe black-granite surface plate using a 65- μ alundum abrasive from Norton Company mixed with kerosene. The final differences in path lengths for both samples were less than 0.5×10^{-4} in.

First, the second-order constants were measured with the pulse-echo technique. Ten MHz ultrasonic pulses were generated from a Matec TA-56 Ultrasonic Testor and transmitted to the crystals via quartz transducers bonded with Nonaq stopcock grease. The Matec unit also served as

TABLE I. Adjusted second-order adiabatic elastic constants of NaF in units of 10^{11} dyn/cm² from eight measurements on both samples.

C_{11}	C'	C_{44}	B	Experiment
9.63 ± 0.09	3.62 ± 0.05	2.76 ± 0.05	4.82	Present
9.70	3.61	2.82	4.82	Ref. 10
9.70	3.67	2.82	4.81	Ref. 11

an amplifier for the returning pulses which were displayed on a Tektronix 535A oscilloscope equipped with a 1A1 calibrated preamplifier. The time between echoes was obtained by superimposing a second signal with a 0.1- μ sec width and calibrated time delay.

For the longitudinal modes a change in the transit time was observed when two transducers were fastened to a sample on opposite faces. This indicates that the acoustic impedances of the crystal and the transducer were similar so that the ultrasonic pulse was actually travelling in both of them. Measuring the transit time with one and then two transducers allowed a correction on the order of 1.5% to be determined for the time spent in the transducer.

The shear mode along the $\langle 111 \rangle$ direction was excluded from all measurements because a non-exponential-echo pattern was observed. This is due to the fact that along an axis of threefold symmetry the k vector of the wave and the energy-flux vector do not coincide.⁹ In expressing the second-order constants and their temperature and pressure derivatives the constant C' , where $C' = \frac{1}{2}(C_{11} - C_{12})$, is used rather than simply C_{12} . Results for the adjusted second-order constants and bulk modulus calculated from C_{11} and C' appear in Table I along with other recent measurements by Miller and Smith¹⁰ and Vallin *et al.*¹¹ Within experimental error the present results are consistent with those previously reported. The error noted is always the probable error, i. e., 0.6745

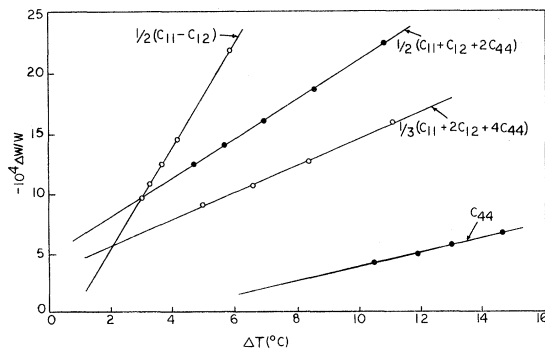


FIG. 1. Typical fractional natural-velocity change $\Delta W/W$ as a function of temperature for four modes of propagation labeled with the appropriate elastic constant.

of the standard deviation, associated with a least-squares fit of the data.

The temperature and pressure derivatives of the elastic moduli were measured with a two-crystal interference technique described in detail elsewhere.^{4,12} It is based on the natural sound velocity W , where $W = 2L_0/t$, L_0 being the natural length of the specimen at room temperature and atmospheric pressure, and t the transit time of an ultrasonic pulse. This is to be distinguished from the true velocity V , where $V = 2L/t$, L being the actual length. The effects of temperature and pressure in the former case are seen only through a change in the transit time since L_0 is a constant. For present purposes it will suffice to merely quote the final results. For the temperature derivatives,

$$\frac{\partial C_{ij}}{\partial T} = (2\beta' - \alpha) C_{ij}, \quad (1)$$

where β' is the temperature coefficient of natural velocity corresponding to C_{ij} and α the linear-thermal-expansion coefficient. The latter was evaluated from the room-temperature volume coefficient of expansion, $0.98 \times 10^{-4}/^\circ\text{C}$, given by Tosi.¹³ Temperature differences were monitored with an iron-constantan thermocouple whose emf was measured using a Leeds & Northrup K-5 potentiometer and standard cell. The imbalance in the potentiometer was displayed on a Leeds & Northrup type W recorder where the full scale reading was adjusted to 10 μ V.

Typical curves of the fractional natural-velocity change as a function of temperature difference are plotted in Fig. 1. The adjusted values for the temperature derivatives of the elastic constants and bulk modulus appear in Table II along with other recent measurements. There is some divergence of the present results from the others noted. However, it is felt that the former are quite accurate in view of the interference method and the least-squares-fitting procedure. Perhaps the large discrepancy in $\partial C_{44}/\partial T$ reported by Vallin *et al.*¹¹ is due to a misprint.

The pressure derivatives of the second-order constants as well as linear combinations of the Brugger third-order moduli were determined from $(\rho_0 W^2)_{P=0}'$, where the prime denotes differentiation

TABLE II. Adjusted temperature derivatives of the second-order adiabatic elastic constants of NaF in units of 10^8 dyn/cm²°C from six measurements.

$\frac{\partial C_{11}}{\partial T}$	$\frac{\partial C'}{\partial T}$	$\frac{\partial C_{44}}{\partial T}$	$\frac{\partial B}{\partial T}$	Experiment
-5.84 ± 0.12	-3.08 ± 0.06	-0.452 ± 0.029	-1.73	Present
-6.27	-3.37	-0.592	-1.78	Ref. 10
-6.0	-3.26	-0.85	-1.66	Ref. 11

TABLE III. Stresses and modes of propagation for the present NaF crystals. \vec{M} , \vec{N} , and \vec{U} are directions of stress, propagation, and polarization, respectively.

No.	\vec{M}	\vec{N}	\vec{U}	$(\rho_0 W^2)_{P=0}$
1	Hydro	[001]	[011]	$\frac{1}{2}(C_{11} + C_{12} + 2C_{44})$
2	Hydro	[011]	[100]	C_{44}
3	Hydro	[011]	[0 $\bar{1}$ 1]	$\frac{1}{2}(C_{11} - C_{12})$
4	Hydro	[111]	[111]	$\frac{1}{3}(C_{11} + 2C_{12} + 4C_{44})$
5	[111]	[011]	[011]	$\frac{1}{2}(C_{11} + C_{12} + 2C_{44})$
6	[111]	[011]	[100]	C_{44}
7	[111]	[011]	[0 $\bar{1}$ 1]	$\frac{1}{2}(C_{11} - C_{12})$
8	[2 $\bar{1}$ $\bar{1}$]	[011]	[011]	$\frac{1}{2}(C_{11} + C_{12} + 2C_{44})$
9	[2 $\bar{1}$ $\bar{1}$]	[011]	[100]	C_{44}
10	[2 $\bar{1}$ $\bar{1}$]	[011]	[0 $\bar{1}$ 1]	$\frac{1}{2}(C_{11} - C_{12})$
11	[2 $\bar{1}$ $\bar{1}$]	[111]	[111]	$\frac{1}{3}(C_{11} + 2C_{12} + 4C_{44})$
12	[011]	[111]	[111]	$\frac{1}{3}(C_{11} + 2C_{12} + 4C_{44})$

with respect to stress and $P=0$ signifies evaluation at zero stress. Within the framework of the natural sound velocity,

$$\frac{\partial C_{ij}}{\partial P} = (\rho_0 W^2)'_{P=0} + \frac{1}{3} K C_{ij} \quad , \quad (2)$$

where K is the isothermal compressibility and ρ_0 the natural density. The directions of stress and possible modes of propagation for the present crystals are listed in Table III. The linear combinations of third-order constants and their coefficients whose sum is equal to $(\rho_0 W^2)'_{P=0}$ for each of the 12 modes have been listed elsewhere.^{7,12} The hydrostatic pressure was applied with nitrogen gas and measured with a Heise gauge. The uniaxial stresses were applied with a modified Simmons Silver King hydraulic jack and read on an Ashcroft pressure gauge calibrated with a dead-weight tester.

In order to fully assess the effects of any dislocation motion during the uniaxial-stress measurements, the two NaF crystals were γ irradiated with a dose of 3000 R after taking data on all 12 modes listed in Table III. Then the uniaxial modes were repeated. The irradiation was done at room

temperature with a ^{60}Co source at the U. S. Army Natick Laboratories, Natick, Mass. The results were difficult to interpret because no attenuation changes were observed during the uniaxial stresses either before or after irradiation, and all the curves of natural-velocity change versus stress appear to be linear. The two sets of $(\rho_0 W^2)'_{P=0}$ were not inconsistent with each other since most of the differences were well within experimental error. Apparently stress levels were low enough so that the slip systems were not appreciably activated and dislocation contributions to $(\rho_0 W^2)'_{P=0}$ were minimal. As yet unexplained, however, was the slight decrease in attenuation for modes Nos. 10 and 12 as the stress was applied. In these cases the amplitude of the echoes increased with increasing stress. There was also uncertainty in the slope of the velocity-change-vs-pressure curve for No. 5 before irradiation since only two points could be measured.

Eliminating modes Nos. 5, 10, and 12 from the before-irradiation uniaxial-stress data and only the latter two from the after-irradiation measurements, a least-squares analysis was performed for (i) hydrostatic-pressure plus before-irradiation uniaxial-stress data, (ii) hydrostatic-pressure plus after-irradiation uniaxial-stress data, and (iii) hydrostatic-pressure plus all uniaxial-stress data. In view of the error associated with the fits there were no significant differences among the three sets of third-order constants. Those reported are from the hydrostatic-pressure and before-irradiation uniaxial-stress data since their over-all consistency was best. The possibility of dislocation contributions to these constants was further diminished by weighting first the hydrostatic-pressure data and then the before-irradiation uniaxial-stress data by a factor of 10. Only slight changes occurred in both the constants and their errors indicating mutual consistency between the two types of stress measurements and a uniform distribution of error.

Typical data appear in Table IV where the coefficients of the third-order constants and the ex-

TABLE IV. Typical data for third-order elastic constants of NaF in units of 10^{12} dyn/cm². The numbers to the left refer to Table III. One reads across a line, for example, $0.7235C_{144} + 1.4470C_{155} = -1.65 \pm 0.11$.

No.	C_{111}	C_{112}	C_{123}	C_{144}	C_{155}	C_{456}	Experiment
1	0.3617	1.4470	0.3617	0.7235	1.4470	0	-10.55 ± 0.54
2	0	0	0	0.7235	1.4470	0	-1.65 ± 0.11
3	0.3617	0	-0.3617	0	0	0	-6.37 ± 0.20
4	0.2412	1.4470	0.4823	0.9648	1.9293	0	-7.35 ± 0.62
6	0	0	0	0.2412	0.4823	-1.2060	-0.441 ± 0.031
7	0.1206	0	-0.1206	0	0	0	-2.092 ± 0.077
8	0.0054	0.3671	0.3510	0.7020	-1.1845	0	1.64 ± 0.11
9	0	0	0	0.0107	0.7127	-0.6030	-0.839 ± 0.053
11	0.0804	0.4823	0.1608	-0.4824	-0.9649	-1.6080	-1.145 ± 0.067

TABLE V. Adjusted third-order elastic constants and third-order combinations for NaF in units of 10^{12} dyn/cm².

$C_{111} = -14.8 \pm 0.8$	$C_{111} + 2C_{112} = -20.1 \pm 0.3$
$C_{112} = -2.7 \pm 0.4$	$C_{111} - C_{123} = -17.6 \pm 0.4$
$C_{123} = 2.8 \pm 0.6$	$C_{144} = 0.46 \pm 0.07$
$C_{144} = 0.46 \pm 0.06$	$C_{155} = -1.14 \pm 0.03$
$C_{155} = -1.14 \pm 0.03$	$C_{456} = 0.00 \pm 0.01$
$C_{456} = 0.00 \pm 0.01$	

perimental quantities are those obtained by substituting into the Brugger equations. In general, the hydrostatic modes have a much larger value for the experimental quantity than the uniaxial modes. Since the least-squares process minimizes the sum of the squares of the deviations of the last column, they should all be of the same order of magnitude in order to be weighted equally. Therefore, the original data were normalized so that the experimental quantity was between 1.5 and 2.5×10^{12} dyn/cm². The values of the third-order moduli determined from a total of 30 normalized equations appear in Table V. Figures 2 and 3 show typical curves of the fractional natural-velocity change as a function of hydrostatic pressure and uniaxial stress, respectively.

Of the nine usable modes of Table III, it should be noted that only No. 8 is dislocation sensitive. Therefore, it is this equation alone which allows the constants C_{111} , C_{112} , and C_{123} to be solved for

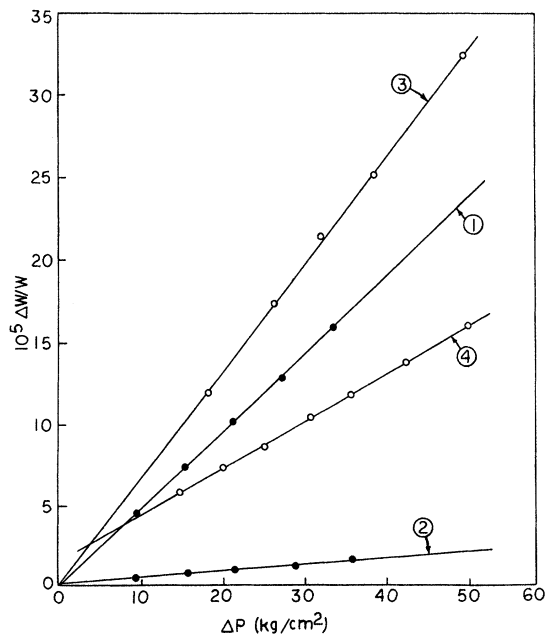


FIG. 2. Typical fractional natural-velocity change $\Delta W/W$ as a function of hydrostatic compression for the first four modes of Table III.

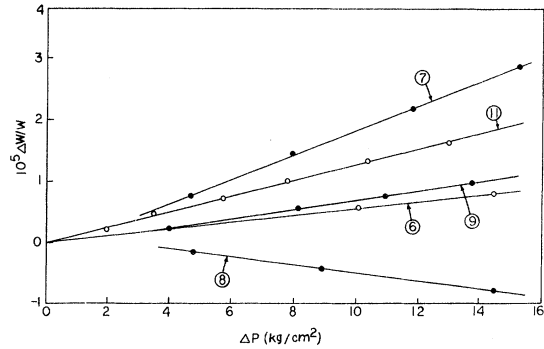


FIG. 3. Typical fractional natural-velocity change as a function of uniaxial stress for the modes listed in Table III.

individually. As a final test for possible dislocation contributions to the third-order moduli, the combinations $C_{111} + 2C_{112}$, $C_{111} - C_{123}$, C_{144} , C_{155} , and C_{456} were determined from 27 normalized dislocation-insensitive equations. These constants also appear in Table V. Comparison of the five adjusted combinations with those calculated from the six third-order moduli demonstrates that mode No. 8 contains no significant dislocation effects.

A self-consistent set of pressure derivatives was evaluated by substituting the adjusted third-order constants back into the Brugger equations in order to determine $(\rho_0 W^2)'_{P=0}$ needed in Eq. (2). The results along with other recent measurements are presented in Table VI. Except for $\partial C_{44}/\partial P$, the differences between the present pressure derivatives and those of Miller and Smith¹² are within the combined experimental error. The latter claim a maximum (unspecified) error of 5%. The large deviation of $\partial C_{44}/\partial P$ is not unexpected because of its very small magnitude.

III. ELASTICITY CALCULATIONS

The calculation of elastic constants as well as cohesive energy for ionic crystals is usually done according to the Born model. The Born model assumes that there are long-range electrostatic interactions which occur in pairs between point-like ions and depend only on their separation dis-

TABLE VI. Self-consistent pressure derivatives of the second-order adiabatic elastic constants of NaF determined from the adjusted third-order elastic moduli. The error is the experimental error calculated from the original data.

$\frac{\partial C_{11}}{\partial P}$	$\frac{\partial C'}{\partial P}$	$\frac{\partial C_{44}}{\partial P}$	$\frac{\partial B}{\partial P}$	Experiment
12.7 ± 0.8	5.10 ± 0.20	0.279 ± 0.024	5.89	Present
11.6	4.79	0.205	5.18	Ref. 10

TABLE VII. Repulsive parameters for NaF short-range lattice energy according to the Born model. Method 1 employed the constants C_{11} and C_{111} , while method 2 made use of the compressibility and equilibrium condition on the lattice energy. A is in units of 10^{-8} erg/molecule, and b is in units of 10^{-8} cm.

A	b	Method
0.357	0.240	1
0.102	0.292	2

tance. Owing to studies of core-electron wave functions, the short-range repulsive energy of two ions separated by a distance r is approximated by an exponential term of the form $Ae^{-r/b}$, where the parameters A and b are evaluated from crystal data. Ghate¹⁴ has employed this model to calculate the third-order elastic constants for all alkali halides at 0°K and then derived the temperature derivatives so that room-temperature values could be obtained. The short-range interactions were extended to next-nearest neighbors and the two parameters fixed by 0°K crystal data. The present calculations use Ghate's static expressions for the elastic constants, but introduce a temperature dependence by allowing A and b to be determined by room-temperature data.

Two different methods were employed to evaluate the repulsive parameters. The first consisted of using the experimental repulsive contributions to the constants C_{11} and C_{111} . These particular constants were chosen because they are primarily dependent on nearest-neighbor central forces and thus most consistent with the Born model. The second method involved the compressibility and equilibrium condition on the lattice energy. This more common technique was used by Tosi¹³ for calculating the cohesive energies of the alkali halides and by Ghate. Quantities needed to evaluate the present repulsive parameters include the room-temperature and atmospheric-pressure value of the nearest-neighbor distance $r = 3.317 \times 10^{-8}$ cm,¹³ the isothermal compressibility determined from the present elasticity data $K^T = 0.217 \times 10^{-11}$ cm²/dyn and the electronic charge $e = 4.803 \times 10^{-10}$ esu. The results for both cases appear in Table VII.

From Table VII, one notes that the change in b due to the two methods is much less than the change in A . Obviously the latter is more sensitive to the actual two conditions used to fix them. The large differences in A and b can be attributed to the neglect of the temperature derivatives of the elastic constants and bulk modulus. Ghate's calculations show that the constant C_{111} may change by approximately 10% in the range 0°K to room temperature, depending on the value of b . Vallin *et al.*'s data demonstrate that C_{11} does vary by about 13% over

the same range.

The two sets of repulsive parameters were substituted into the expressions for the elastic constants and cohesive energy. The best over-all agreement occurred when A and b were determined from the two elastic constants. Then, in addition to C_{11} and C_{111} being exact, C_{12} , C_{44} , and C_{155} had noticeably smaller deviations. These calculations are presented in Table VIII. As shown in this table, the repulsive contributions to C_{11} and C_{111} are mostly those of the nearest neighbors. On the other hand, both repulsive terms in the cohesive energy contribute very little, that of the next-nearest neighbors being approximately 1% of the total. C_{12} , C_{44} , C_{112} , and C_{155} contain second-neighbor short-range terms smaller than the electrostatic ones. Finally, C_{123} , C_{144} , and C_{456} depend solely on the long-range forces. The long-range Cauchy condition, $C_{123} = C_{144} = C_{456}$, is hardly satisfied, but the short-range conditions, $C_{12} = C_{44}$ and $C_{112} = C_{155}$, are obeyed better.

IV. GRÜNEISEN PARAMETER CALCULATION

In the process of evaluating the equation of state of a solid in the quasiharmonic approximation one defines the mode Grüneisen parameters γ_i as $\partial \ln \omega_i / \partial \ln V$, where ω_i is the frequency of the i th mode and V the volume.¹⁵ Generalizing this expression so that the volume dependence of ω_i is only a particular example of a total strain dependence, Brugger has defined a Grüneisen tensor as¹⁶

$$\gamma_i^{\alpha\beta} = \frac{1}{\omega_i} \left. \frac{\partial \omega_i}{\partial \eta_{\alpha\beta}} \right|_{T,P=0}, \quad (3)$$

where $\eta_{\alpha\beta}$ are the Lagrangian strains,¹⁷ T the temperature, and $P = 0$ signifies evaluation at zero stress. The elements of $\gamma_i^{\alpha\beta}$ are related to linear combinations of second- and third-order elastic constants with components of unit vectors in the direc-

TABLE VIII. Elastic-constant and cohesive-energy calculations for NaF according to the Born model. Units are 10^{12} dyn/cm². Repulsive parameters were determined from C_{11} and C_{111} . Coulomb, nearest-neighbor (NN), and next-nearest-neighbor (NNN) contributions are listed. Cohesive energy taken from Ref. 13.

	C_{11}	C_{12}	C_{44}	Cohesive energy		
Coulomb	-1.023	0.278	0.278	-0.699		
NN	1.918	0	0	0.056		
NNN	0.068	0.034	0.034	0.002		
Calc.	0.963	0.312	0.312	-0.641		
Expt.	0.963	0.240	0.276	-0.595		
	C_{111}	C_{112}	C_{123}	C_{144}	C_{155}	C_{456}
Coulomb	8.215	-0.967	0.543	0.543	-0.967	0.543
NN	-22.501	0	0	0	0	0
NNN	-0.540	-0.270	0	0	0.270	0
Calc.	-14.82	-1.237	0.543	0.543	-1.237	0.543
Expt.	-14.82	-2.61	2.77	0.466	-1.14	±0.01

TABLE IX. Room-temperature Grüneisen-parameter calculations and corresponding volume-thermal-expansion coefficients determined from elasticity data of NaF. Units of β are $10^{-4}/^{\circ}\text{C}$.

γ	Elasticity data	β
1.55	Present	1.01
1.40	Ref. 10	0.94
	Direct experiment from Ref. 13	0.98

tions of propagation and polarization as coefficients. For cubic symmetry, the Grüneisen parameter is equal to $\frac{1}{3}$ the trace of the tensor.

For the case of high temperatures when all modes are fully excited, the average Grüneisen parameter was evaluated by spacing 734 points uniformly over $\frac{1}{4}$ of the Debye sphere in k space in a manner similar to that described by Hiki, Thomas, and Granato.¹⁸ For the sake of comparison, the pressure derivatives of Miller and Smith were used to evaluate each γ_i according to the definition for cubic crystals involving the second-order constants, their pressure derivatives, and the isothermal compressibility. Using an RCA Spectra 70 computer, the three elastic constants and polarization vectors for a given direction were determined from the secular equation after the method suggested by

Quimby and Sutton.¹⁹ The results appear in Table IX.

As a test of the quasiharmonic approximation, the room-temperature volume-thermal-expansion coefficient β was evaluated from the average Grüneisen parameter γ by means of the thermodynamic relation

$$\beta = \rho_0 c_v \gamma / B, \quad (4)$$

where c_v is the specific heat and B the isothermal bulk modulus. The former was determined from the heat capacity given by Tosi.¹³ In Table IX, the calculated values of β are compared with the experimental one. The agreement of theory and experiment is within the limits normally expected for this type of calculation.

ACKNOWLEDGMENTS

The author wishes to express his appreciation for the many instructive hours concerning both the experimental procedure and interpretation of the data spent with Dr. Karl Swartz. The suggestions and criticisms of Dr. Thomas Keil are also gratefully acknowledged. Thanks are extended to the Materials Engineering section of the Mechanical Engineering Department of W. P. I. for the loan of the hydraulic press and to the U. S. Army Natick Laboratories, Natick, Mass., for irradiating the two NaF crystals.

[†]Based on a thesis submitted in partial fulfillment of the requirements for the Ph. D. degree at Worcester Polytechnic Institute (W. P. I.), 1971.

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