

Measurements of the Mechanical Q of Single-Crystal Silicon at Low Temperatures*

**D. F. McGuigan, C. C. Lam, R. Q. Gram, A. W. Hoffman,
and D. H. Douglass**

Department of Physics and Astronomy, University of Rochester, Rochester, New York

and H. W. Gutche

Monsanto Company, St. Peters, Missouri

(Received June 24, 1977)

Measurements of the mechanical quality factor Q in a single crystal of silicon vs. temperature have been made. A value of 2×10^9 has been measured at $T = 3.5$ K.

We have been studying large dielectric and semiconductor single crystals as possible gravitational wave detectors.¹ These detectors are frequently cylinders instrumented to detect vibrations of the first longitudinal mode. One of the important desirable properties is a high mechanical quality factor Q . With an ideal transducer the sensitivity to gravitational waves is proportional to Q . The inverse quality factor Q^{-1} is a direct measure of the dominant mechanism of the attenuation of first sound. Measurements of Q are frequently the best way to gather information concerning the various internal loss mechanisms of a substance. In the region of very high Q (low loss) this sometimes is the only easy method. We report here on measurements on a large single crystal of silicon whose Q values exceed 10^9 .

The crystal was manufactured by Monsanto by the zero-dislocation Czochralski process in the form of a cylinder (diameter 10.6 cm, length 22.9 cm, mass 4.9 kg), with the [111] axis parallel to the cylinder axis. The ends were polished by the University of Rochester Institute of Optics personnel to better than one wavelength of light. The barrel of the crystal, somewhat wavy from the crystal growing process, was left in this state so that its edge deviates from straightness by ~ 1 mm. The crystal is a p -type

*Supported in part by the National Science Foundation.

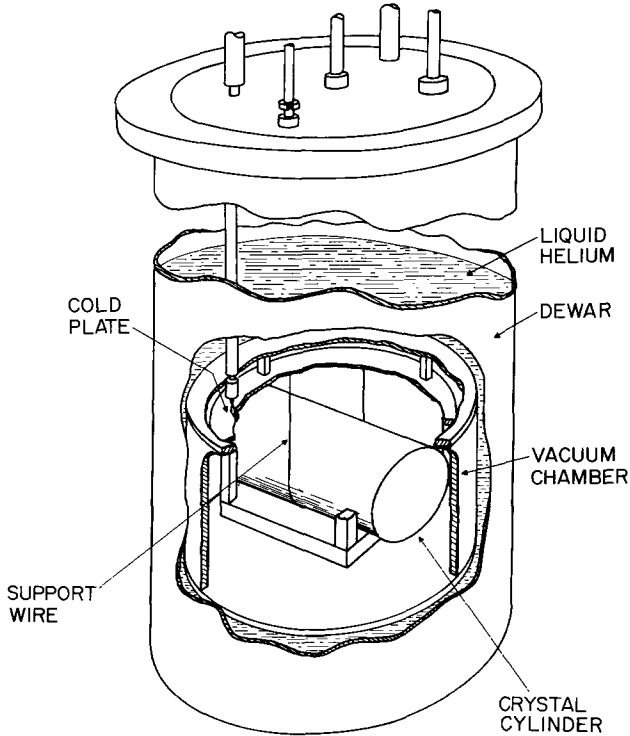


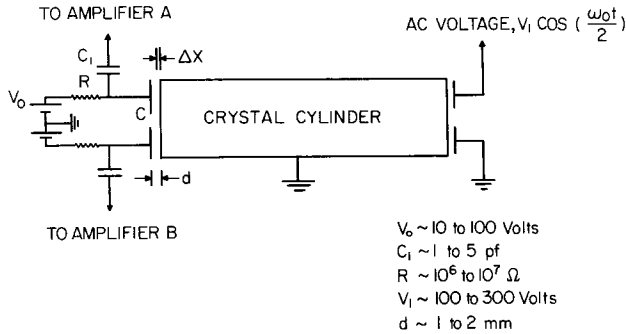
Fig. 1. Schematic of crystal cylinder and liquid helium cryostat.

semiconductor (boron-doped; $2.7\text{--}4.5 \times 10^{15}$ atoms/cm³) with a resistivity of $3\text{--}5 \Omega\text{-cm}$ at room temperature. A crystal grown in this manner also usually contains oxygen (3×10^{18} atoms/cm³) and carbon (8×10^{16} atoms/cm³). The level of other impurities is usually less than 10^{15} atoms/cm³.

The crystal was suspended by a 0.25-mm-diameter tungsten wire around its center as Fig. 1 indicates. Figure 2 shows schematically the electrical circuit for exciting the crystal into resonance and the detection of the subsequent motion. This scheme is essentially that of a capacitance microphone. In this circuit the displacement ΔX of the face of the crystal causes a change in the capacitance C , which produces a change in the output signal ΔV at the resonator frequency ω_0 ,

$$\Delta V = V_A - V_B \quad (1)$$

The voltages V_A and V_B are put into the differential input of a lock-in amplifier and $V_A - V_B$ is demodulated with a local oscillator (a Rockland



SCHMATIC OF Q MEASURING CIRCUIT

Fig. 2. Schematic of electrical circuit. A voltage at one-half the cylinder frequency excites the cylinder into resonance. The amplitude of vibration ΔX is determined by measuring the output $V_A - V_B$ of the balanced capacitance microphone circuit. This measurement is always made with the driving circuit off or disconnected. Displacements of $\Delta X \approx 5 \times 10^{-11}$ cm can easily be measured.

5100 frequency synthesizer locked to a high-stability quartz oscillator) at a frequency close to ω_0 . If $RC_1 \gg 2\pi/\omega_0$ (where $2\pi/\omega_0$ is the period of oscillation of the crystal), the relationship between ΔX of the crystal and the signal ΔV at ω_0 is

$$\Delta V \approx (V_0/C)(\partial C/\partial X)\Delta X \tag{2}$$

The value of $(1/C)(\partial C/\partial X)$ was approximately $10\text{--}100 \text{ cm}^{-1}$. The precise value depends on the electrical conductivity of the crystal. For most of the data reported here the faces had a 1000-\AA aluminum film evaporated onto the end. In later runs this was not done. (At room temperature silicon is a poor conductor; at low temperatures it approaches an insulator.) For the voltages used in this experiment, $V_0 \approx 10\text{--}200 \text{ V}$, $\Delta V \approx 10^{-7}\text{--}10^{-3} \text{ V}$, the range of displacements measured was $10^{-11}\text{--}10^{-6} \text{ cm}$. The Q values, which were always measured in a vacuum (approximately 10^{-5} Torr), were obtained by observing the exponential decay of the amplitude ΔV and applying the relation

$$Q = \frac{\omega_0 t}{2 \ln [\Delta V(t)/\Delta V(0)]} \tag{3}$$

where t is the time.

After placing the crystal in a liquid helium cryostat, shown also in Fig. 1, the temperature of the crystal was varied in the range $1.3\text{--}300 \text{ K}$. The temperature was determined by placing standard cryogenic thermometers

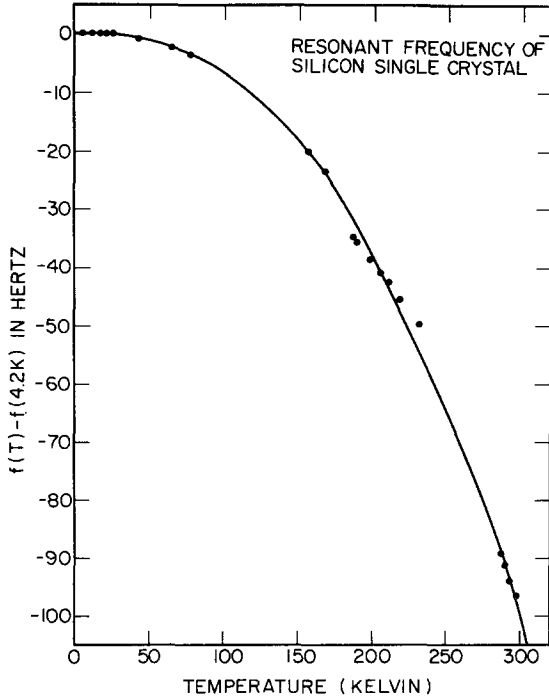


Fig. 3. Resonant frequency of longitudinal vibration vs. temperature of the silicon crystal. $1.3 < T < 300$ K. Here $f(4.2) = 19553.46542$ Hz. A smooth curve has been drawn through the experimental points.

(i.e., Lake Shore germanium resistance thermometers) in contact with the crystal. Since the mechanical loading of the thermometers degraded the Q , the temperature dependence of Q was measured by the following procedure. In a run with the thermometers bonded to the crystal the frequency of the crystal was measured as a function of temperature (see Figs. 3 and 4). The thermometers were then removed and in subsequent runs to measure the Q , the temperature was determined from the measured frequency. Figure 3 shows the measured frequency vs. temperature relationship, which is quite reproducible, over the range 1.4–300 K, and Fig. 4 shows the low-temperature portion (1.4–28 K) of the same data. By this method one can see the ease with which the crystal has become its own thermometer. At temperatures below 8 K, where our frequency sensitivity is poor, we assumed that the crystal was at the same temperature as a thermometer placed on the support structure after waiting a suitable time.

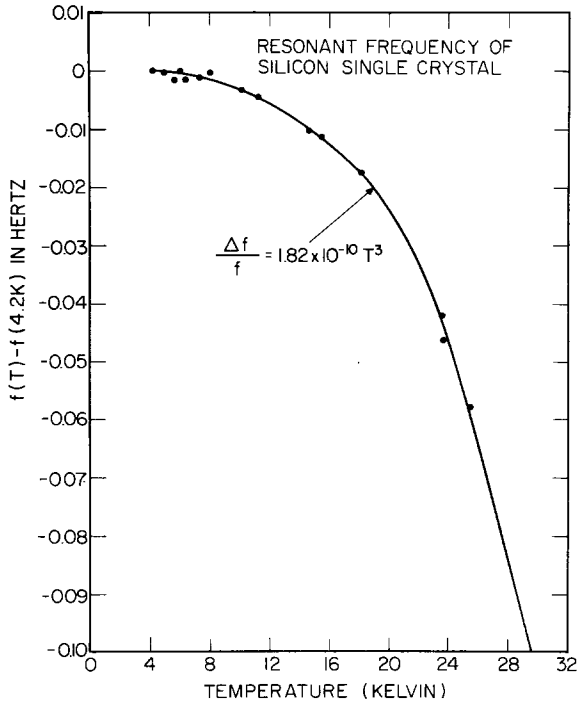


Fig. 4. Resonant frequency of longitudinal vibration of the silicon crystal. $1.3 < T < 30$ K. Here $f(4.2) = 19553.46542$ Hz. It is determined that the curve $\Delta f/f = 1.8 \times 10^{-10} T^3$ fits the data quite well.

This is a good assumption because of the high thermal conductivity of the wires.

Figure 5 shows an amplitude vs. time curve taken at 3.5 K. (All curves are taken in "free decay," i.e., with driving circuit off or disconnected.) This is exponential with a $Q \approx 2.0 \times 10^9$. The Q values determined in this way at various temperatures are shown in Fig. 6. We will now discuss the above results.

The relationship between the resonant frequency f of the first longitudinal mode of vibration and the properties of the crystal is

$$f = \eta v_s / 2l \quad (4)$$

where v_s is the velocity of sound, l is the length of the crystal, and η is a dimensionless parameter² which depends weakly on the ratio of diameter to length and Poisson's ratio. For this crystal η is ~ 0.98 . At room temperature, we measured for the first longitudinal mode of vibration

VIBRATION OF SILICON CYLINDER

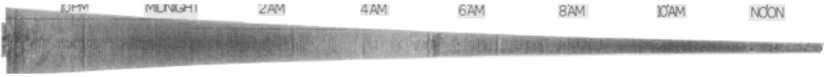


CHART RECORDING OF DISPLACEMENT
 ↑
 TIME →

$\tau_{1/e} \approx 3.1 \times 10^4 \text{ sec} \approx 8.4 \text{ hrs}$
 $Q \approx 2 \times 10^9$

Fig. 5. Amplitude of vibration in first longitudinal mode of vibration vs. time. The value of $Q \approx 2 \times 10^9$ corresponding to a relaxation time $1/e$ of $3.1 \times 10^4 \text{ sec} = 8.6 \text{ h}$ was measured. The oscillation in amplitude is caused by a slight frequency difference (which is deliberate) between the local oscillator and the cylinder.

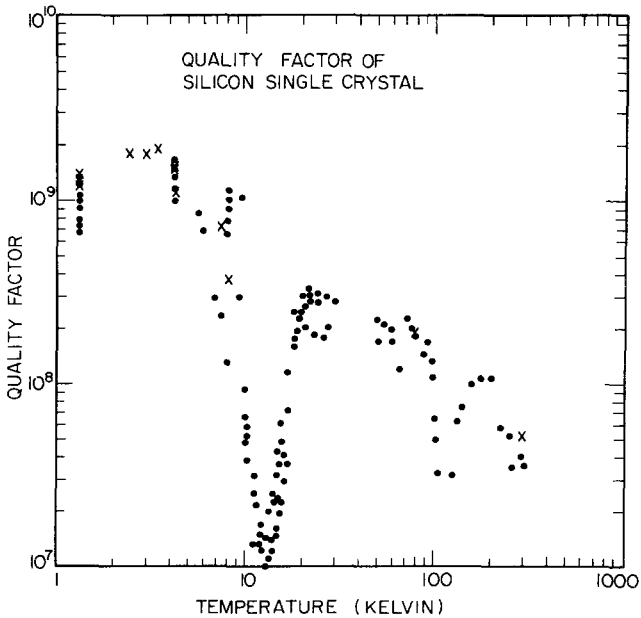


Fig. 6. Quality factor of silicon vs. temperature. The solid dots (data points) were obtained during several runs with aluminum films evaporated on the faces. The data points indicated by \times were obtained in a later run after chemical polishing; no metal film was evaporated onto the face.

$f = 19,456$ Hz. Putting this into Eq. (4), one obtains $v_s = 9.08 \times 10^5$ cm/sec. For a cubic crystal the velocity of longitudinal sound in the 111 direction is $v_s(111) = [(C_{11} + 2C_{12} + 4C_{44})/3\rho]^{1/2}$, where the C_{ij} are the elastic constants and ρ is the density. Using $\rho = 2.33$ g cm $^{-3}$ and the values of McSkimen³ ($C_{11} = 16.57$, $C_{12} = 6.39$, $C_{44} = 7.96 \times 10^{-11}$ dyn cm $^{-2}$), we obtain $v_s = 9.35 \times 10^5$ cm/sec, which is about 3% higher than was measured. From Eq. (4) the change of f with temperature can be expressed as

$$\frac{1}{f} \frac{\Delta f}{\Delta T} = \frac{1}{v_s} \frac{\Delta v_s}{\Delta T} - \frac{1}{l} \frac{\Delta l}{\Delta T} \quad (5)$$

The second term is recognized as the negative of the thermal expansion coefficient α_T . Thus

$$\frac{1}{f} \frac{\Delta f}{\Delta T} = \frac{1}{v_s} \frac{\Delta v_s}{\Delta T} - \alpha_T \quad (6)$$

At room temperature the measured $(1/f) \Delta f/\Delta T$ is -4.0×10^{-5} K $^{-1}$. The value of α_{273} is 2.3×10^{-6} K $^{-1}$.⁴ Thus the change in frequency due to the change in length is only about 5%. One calculates using Eq. (6) that $(1/v_s) \Delta v_s/\Delta T = -4.2 \times 10^{-5}$ K $^{-1}$. This can be compared with the value $\sim -3 \times 10^{-5}$ K $^{-1}$ for silicon reported by McSkimen.³ At low temperatures, $T < 25$ K, we have found that the data can be fit very well by the function $[f(T) - f(0)]/f(0) = -1.8 \times 10^{-10} T^3$, although there is no reason for expecting such a simple power law relationship. This leads to

$$\left. \frac{1}{f} \frac{\Delta f}{\Delta T} \right|_{T < 25\text{K}} = -5.5 \times 10^{-10} T^2 \quad (7)$$

The thermal expansion coefficient of silicon at low temperatures has been measured by Sparks and Swenson.⁵ Below 16 K they find $\alpha_T \approx 8.9 \times 10^{-12} T^3$. One sees that below $T \approx 16$ K, in addition to having a different power law, the expansion coefficient has a small effect (less than 20%) on the observed change of frequency with temperature.

Although the temperature vs. frequency results are interesting, the remarkable results are the measurements of quality factor Q . The quality factor vs. temperature measurements are shown in Fig. 6. The solid points are measurements consisting of a series of runs (at least three cooldowns) with the crystal faces mechanically polished and with 1000-Å aluminum evaporated on them.* The \times symbols correspond to values in a later run

*In the measurements with aluminum films evaporated onto the faces of the crystal we occasionally observed slight nonexponential decays for very long relaxation times (high Q 's) at low temperatures. We were able to associate this with a residual voltage on the plates of the crystal that changed with time. This is probably due to the fact that the silicon is an insulator at low temperatures and that the charge state of the aluminum films is not well defined or controlled. These effects were not observed in the later experiments, where the sample had no metal film on the faces.

for which a chemical polish of all of the surfaces of the crystal was undertaken (and with no metal film evaporated onto the faces).

The data in Fig. 6 show a number of prominent features. The first is that there are two strong minima at 13 and 115 K. These are probably associated with impurities and mechanical imperfections in the crystal. Absorption in silicon in the temperature region of 115 K has been seen by Starodubtsev *et al.*,⁶ who interpret the effect as a Bordoni-type relaxation peak associated with the energy of a kink on a dislocation. This same group showed that the magnitude of the effect depended on plastic deformation and irradiation from γ -rays and even reported two distinct absorptions in *p*-type silicon at 91 and 124 K.⁷ Other investigators did not confirm these findings.^{8,9} We will not comment at this time on the nature of this absorption except to point out that this crystal is "dislocation-free," which means that the dislocation density with good probability does not exceed 10 cm^{-2} (visual inspection of the etched surface reveals etch pits totalling approximately ten). The internal friction dip at 13 K has not to our knowledge been observed before. In searching for phenomenon corresponding to this energy, one notices that some of the level differences of the B impurity atom are close to this value¹⁰ and one might wish to try to invent a mechanism connecting the absorption of sound with the impurity energy states. It is perhaps not a coincidence that Mason and Bateman¹¹ in their studies of ultrasonic pulses in boron-doped Si in the 100-MHz band find at low temperatures an absorption relaxation time with a corresponding activation energy of 13 K. One sees from the data that there may be another internal friction dip at temperatures below 1 K, but we attempt no interpretation at this time. We note the interesting result that Q values as large as $\sim 2.0 \times 10^9$ have been observed at $T \approx 3\text{--}4 \text{ K}$.

These results suggest that in this crystal the Q value at low temperatures is determined by internal processes. If one had a crystal free from impurities and imperfections, one might expect significantly higher Q 's. We note that the Q values reported here are comparable in magnitude to the best values reported in other pure materials: 4.2×10^9 in quartz¹² and 5×10^9 in sapphire.¹³ The question of how large Q can be in a pure crystal is an interesting one. The ultimate loss mechanism is due to phonon-phonon scattering and an estimate of the magnitude leads to very large Q values at low temperatures. Before reaching this limit one will probably encounter the case of a pure crystal with rough surfaces. Savedoff^{14,*} has given an argument to suggest that for this case $Q \approx (\pi L/a)^2$, where L is the size of the crystal and a is a measure of the surface roughness. Choosing $L \approx 30 \text{ cm}$, $a \approx 10^{-7} \text{ cm}$, one estimates that $Q \approx 10^{18}$ is feasible, which is

*A preliminary discussion of the model is given in Ref. 1.

seen to be eight or nine orders of magnitude higher than what we have presently achieved.

ACKNOWLEDGMENT

We wish to thank Gregory Johnson for helpful discussions.

REFERENCES

1. D. H. Douglass, Gravitational Wave Experiments, to be published in the *Proceedings of the Academia Nazionale dei Lincei International Symposium on Experimental Gravitation, Pavia, Italy* (September 1976).
2. D. Bancroft, *Phys. Rev.* **59**, 588 (1941).
3. J. H. McSkimen, *J. Appl. Phys.* **24**, 988 (1953).
4. D. F. Gibbons, *Phys. Rev.* **112**, 136 (1958).
5. P. W. Sparks and C. A. Swenson, *Phys. Rev.* **163**, 779 (1967).
6. S. V. Starodubtsev *et al.*, *Fiz. Tverd. Tela* **8** (6), 1924 (1966) [*Sov. Phys.—Solid State* **8**, 1521 (1966)].
7. I. P. Khiznichenko *et al.*, *Phys. Stat. Sol.* **21**, 805 (1967).
8. B. M. Mecs and A. S. Nowick, *Appl. Phys. Lett.* **8**, 75 (1966).
9. H. J. Möller and J. Buchholtz, *Phys. Stat. Sol. (a)* **20**, 545 (1973).
10. K. Colbow, *Can. J. Phys.* **41**, 1801 (1963).
11. W. Mason and T. Bateman, *Phys. Rev.* **134**, A1387 (1964).
12. A. G. Smagin, *Cryogenics* **15**, 483 (1975).
13. V. B. Braginsky, in *Proceedings of the Academia Nazionale dei Lincei International Symposium on Experimental Gravitation, Pavia, Italy* (September 1976); see also *Sov. Phys.—Crystallogr.* **19**, 549 (1974).
14. M. Savedoff, private communication.