JULY 15, 1933 PHYSICAL REVIEW VOLUME 44

The Temperature Dependence of Young's Modulus for Nickel

JERROLD ZACHARIAS, *Department of Physics, Columbia University*

(Received April 10, 1933)

A new method is described for the measurement of Young's modulus and its variation with temperature below 400°C. The method is applied to single crystals of pure nickel and to hard drawn, polycrystalline, commercial nickel between 30°C and 400°C. The variation of Young's modulus with temperature in these substances depends on the previous thermal history of the sample. Between 30° and 200° Young's modulus for *annealed* specimens decreases about 13 percent. This is followed by an increase to the Curie point of about 6 percent and above the Curie point by a linear decrease. For *hard drawn* specimens and specimens *quenched* at 1100° the minimum is wholly absent. Young's modulus decreases continuously to the Curie point, where the temperature coefficient changes abruptly.

THE object of this research is the measurement of Young's modulus for various specimens of nickel as a function of temperature between 30°C and 400°C. The specimens are rods of circular cross section, 5 mm to 7 mm in diameter and 3 cm to 8 cm long. The observations here reported yield the fundamental frequencies of free longitudinal vibration of the rods. The values of these quantities are related to the corresponding values of Young's modulus by the formula:[1]

$$G_1=\rho_1 f_n^2(4L_1^2+2\pi^2\sigma^2r^2) \qquad (1)$$

where G_1 = Young's modulus for the direction of the cylinder axis, f_n = fundamental frequency of free longitudinal vibration, ρ_1 = density of the material, L_1 = length of the rod, σ = Poisson's ratio, and r_1 = radius of the rod.

THEORY

1. The vibrating system

The specimen forms one part of a composite piezoelectric oscillator of the sort described by Quimby.[2] A quartz rod of square cross section equal in area to that of the specimen is so cut from a crystal that the optic axis is perpendicular to one pair of opposite sides and an electric axis to the other. The latter pair is coated with gold leaf. One end of the nickel rod is thinly copper plated and covered with a molten mixture of copper oxide and boric oxide. The rods are then cemented together in a vacuum, under pressure, at a temperature of 600°C.

The oscillator is supported by fine wires and a sinusoidal potential difference is established between the coatings of gold leaf. The electric field in the quartz is accompanied by a piezoelectric stress proportional thereto. In consequence of this harmonically varying stress, a stationary state of forced longitudinal vibration is established in the composite rod.

The present method demands an expression relating the time derivative of the space average strain in the quartz to the frequency of the exciting voltage.

It is assumed that the equations which describe the propagation of the disturbance in the quartz and nickel are, respectively:[2]

$$\partial^2u/\partial t^2=V_2^2\partial^2u/\partial x^2, \qquad (2)$$

and

$$\partial^2u/\partial t^2=V_1^2\partial^2u/\partial x^2+Q_1\partial^3u/\partial t\partial x^2, \qquad (3)$$

where u is the particle displacement in the direction of x (Fig. 1), and V_1, V_2 and Q_1 are independent of u, x and t. The form of these equations implies: (1), Hooke's law; (2), absence of internal friction in the quartz; and (3), pure longitudinal vibration.

If the potential difference, E, is of the form,

$$E=E_0e^{int},$$

then the piezoelectric stress, Π, is of the form,[3]

---

[1] Rayleigh, *Theory of Sound*, 2nd edition, vol. I, p. 252.

[2] S. L. Quimby, Phys. Rev. **25**, 558 (1925).

[3] W. G. Cady, Phys. Rev. **19**, 1 (1922).

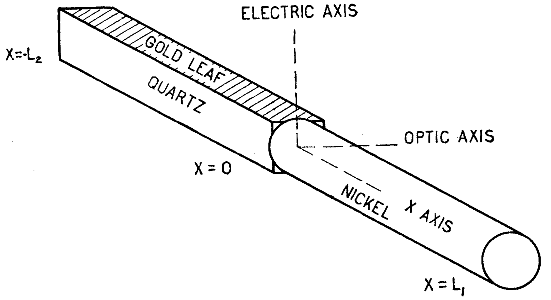

FIG. 1.

$\Pi = \Pi_0 e^{int}$. The expressions

$$u_2 = e^{int}\{A_2 e^{ikx} + A_2' e^{-ikx}\}, \tag{4}$$

and

$$u_1 = e^{int}\{A_1 e^{(\alpha+i\beta)x} + A_1' e^{-(\alpha+i\beta)x}\} \tag{5}$$

are solutions of Eqs. (2) and (3) respectively provided

$$k = n/V_2, \qquad \beta = n(1-3\lambda^2/8)/V_1,$$
$$\alpha = \beta\lambda(1-\lambda^2/4)/2, \qquad \lambda = nQ_1/V_1^2,$$

and λ^4 is small compared with unity.

The constants A_1, A_1', A_2, A_2' are evaluated with the aid of equations expressing continuity of total force and velocity at the boundaries. Thus at $x = -L_2$

$$G_2(\partial u/\partial x)_2 + \Pi = 0$$

at $x = 0$

$$q_2\{G_2(\partial u/\partial x)_2 + \Pi\} = q_1\{G_1(\partial u/\partial x)_1 + \rho_1 Q_1(\partial^2 u/\partial x \partial t)_1\},$$

and

$$(\partial u/\partial t)_2 = (\partial u/\partial t)_1,$$

and at $x = L_1$

$$G_1(\partial u/\partial x)_1 + \rho_1 Q_1(\partial^2 u/\partial x \partial t)_1 = 0.$$

In these expressions the subscripts 1 and 2 refer to conditions in the nickel and the quartz, respectively, and q_1 and q_2 denote the cross-sectional areas of the rods. Accordingly the displacement in the quartz is given by Eq. (4) where

$$\begin{aligned} A_2 &= \Pi_0\{iM(1-e^{iy})(e^{z+iw}+e^{-(z+iw)}) - (iN-P)(e^{z+iw}-e^{-(z+iw)})\}/\Delta, \\ A_2' &= \Pi_0\{iM(1-e^{-iy})(e^{z+iw}+e^{-(z+iw)}) + (iN-P)(e^{z+iw}-e^{-(z+iw)})\}/\Delta, \end{aligned} \tag{6}$$

and

$$\Delta = 4iM\{M \sin y \cosh(z+iw) - (iN-P)\sinh(z+iw)\},$$

$$P = nf_n m_1 \lambda(1-\lambda^2/8),$$

$$N = 2nf_n m_1(1+\lambda^2/2),$$

$$M = 2nf_q m_2,$$

$$z = \alpha L_1 = \pi f \lambda(1-5\lambda^2/8)/2f_n,$$

$$w = \beta L_1 = \pi f(1-3\lambda^2/8)/f_n,$$

$$y = kL_2 = \pi f/f_q,$$

$$f = n/2\pi,$$

f_q = fundamental frequency of free longitudinal vibration of quartz rod alone, m_1 = mass of nickel rod, m_2 = mass of quartz rod.

If $\dot{s}$ denote the time derivative of the average strain in the quartz, then,

$$\dot{s} = \frac{\partial}{\partial t}\int_{x=0}^{x=-L_2} \frac{\partial u}{\partial x} dx. \tag{7}$$

2. The electric current

The observed quantity in the present experimental method is the variation with frequency of the amplitude of the current which flows between the electrodes on the quartz when the amplitude of the applied potential difference is kept constant. Van Dyke[4] has shown that this current is related to $\dot{s}$ by the formula

$$I = \dot{E}C - \epsilon A\dot{s}, \tag{8}$$

where I = current between the electrodes on the quartz, C = the electrostatic capacity between the electrodes, ϵ = the appropriate piezoelectric constant, and A = a geometrical constant.

It follows from Eqs. (4), (6), (7) and (8) that over the range of frequencies for which $\tan^4 y$, $\tan^4 w$, $\lambda^2 \tan^2 y$ and $\lambda^2 \tan^2 w$ are small compared with unity, the current I may be expressed in the form:

$$I = E_0 e^{int}\{inC + 1/(R+iX)\}, \tag{9}$$

---

[4] Van Dyke, Proc. I.R.E. **16**, 742 (1928).

where

$$R=(aL_2/4A\epsilon^2q_2)(1-\tan^2 y/2-\tan^2 w)(\pi f_n m_1\lambda),$$

$$X=(aL_2/2A\epsilon^2q_2)(1-\tan^2 y/2)$$
$$\times\{(m_2f_q\tan y+m_1f_n\tan w+m_1f_n\lambda^2\Lambda/4)\},$$

$$\Lambda=w-\pi w\tan w-\pi^2m_1f_n\tan y/4m_2f_q,$$

and a = distance between electrodes.

The equation,

$$X=0 \tag{10}$$

defines a set of "resonance frequencies." Let one of these be denoted by f_0. Then as the frequency is increased through f_0 the amplitude of I passes through a maximum and a minimum. The frequency, f_0, lies between the frequencies which correspond to these maximum and minimum values of I.

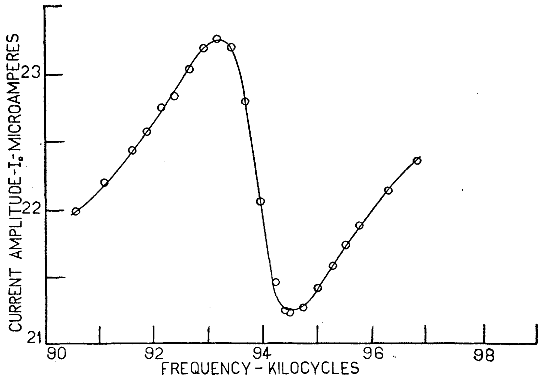

FIG. 2. Typical frequency-current curve.

A typical frequency-current curve is shown in Fig. 2. The points indicate observed values of the current amplitude, denoted by I_0; the line is a graph of this quantity as given by Eq. (9). If $f_q=f_0\pm 4000$ cycles, then the frequency, f_0, is related to the frequencies and currents corresponding to the maximum and minimum of this curve as follows: Let I_0', I_0'', f', f'', denote the current amplitudes and frequencies corresponding to the maximum and minimum respectively. Then

$$f_0=f'+(f''-f')I_0'/(I_0'+I_0''). \tag{11}$$

Furthermore, from Eq. (9)

$$\lambda^2=4(1-m_2/m_1)^2(f''-f_0)(f_0-f')/f_n^2. \tag{12}$$

λ and f_n are calculated with the aid of Eqs. (10), (11), and (12) together with a value of f_q obtained from observations on an oscillator which consists of the quartz rod alone. In fact, f_0 is given by Eq. (11) and λ by Eq. (12) with an approximate value of f_n. The values of f_0, f_q, and λ so obtained are substituted in Eq. (10) and this is solved for f_n.

APPARATUS AND EXPERIMENTAL METHODS

1. Electrical

To complete the calculations described in the preceding section it is not necessary to know the value of the quantity I_0; the ratio I_0/E_0 suffices. Fig. 3 indicates the arrangement used to supply the alternating potential difference, and to measure its frequency and the quantity I_0/E_0. The potential difference across the quartz is supplied through an amplifier by a vacuum tube oscillator. Its frequency is measured by comparison with the output of a piezoelectric clock.[5] The method of comparison is described elsewhere.[6] I_0/E_0 is measured as follows: r_1 (Fig. 3) is a resistance whose magnitude is negligible in comparison with the impedance of the quartz oscillator. The magnitude of the parallel resistance r_2 is adjusted with the aid of a vacuum tube voltmeter, V, until the potential difference across r_2 is equal to that across r_1. Then

$$I_0/E_0=r_2/Rr_1. \tag{13}$$

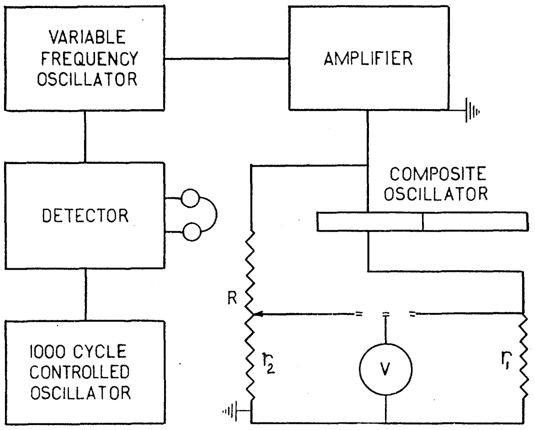

FIG. 3. Circuit diagram.

---

[5] Hull and Clapp, Proc. I.R.E. **17**, 267 (1929).

[6] S. L. Quimby, Phys. Rev. **39**, 345 (1932).

2. Magnetic and thermal

The composite oscillator lies in a fused silica boat on two fine wires located at nodes of vibration. The boat is situated at the center of a horizontal electric furnace about which is wound a solenoid. An astatic magnetometer is mounted above the solenoid and over the specimen. Prior to each observation the specimen is demagnetized with a diminishing alternating current in the solenoid.

The temperature of the specimen is measured with a Chromel P—Alumel thermocouple together with a Leeds and Northrup Type K potentiometer. The junction of the couple is placed about a millimeter from the free end of the specimen.

Experimental Results

The nickel from which the specimens are prepared is of two kinds, first, commercial nickel, initially in the form of hard drawn rods supplied by the International Nickel Company, and second, Mond nickel purified by the method of Fink and Rohrman.[7] The chemical analysis of the commercial nickel is as follows, where the numbers show the percent of impurity present: C, 0.11; Cu, 0.13; Fe, 0.41; Mn, 0.02; Si, 0.09; S, 0.005; Co, 0.3–0.4. The purified nickel is initially spectroscopically pure except for traces of copper and silver. The material is melted in an evacuated molybdenum resistance furnace[8] and single crystals are grown from the melt. Spectroscopic and chemical analyses of such specimens reveal contamination by copper (less than 0.003 percent) and by molybdenum (0.11 percent) and the absence of the following possible impurities: Al, C, Cr, Co, Fe, Mn, Si, Ag.

Observations are reported upon specimens with the following designations and thermal histories:

A. Commercial nickel, heated to 700°C and cooled slowly;

B. Commercial nickel, annealed at 900°C for two hours and cooled slowly;

C. Commercial nickel, annealed at 1100°C for two hours and cooled slowly;

D. A single crystal of purified nickel, cooled slowly from 1450°C;

E. A single crystal of purified nickel, reheated to 1100°C and quenched in water;

F. Commercial nickel, annealed at 1100°C for two hours, cooled slowly, reheated to 1100°C and quenched in water.

1. Young's modulus

The curves of Fig. 4 exhibit the temperature dependence of Young's modulus for specimens A

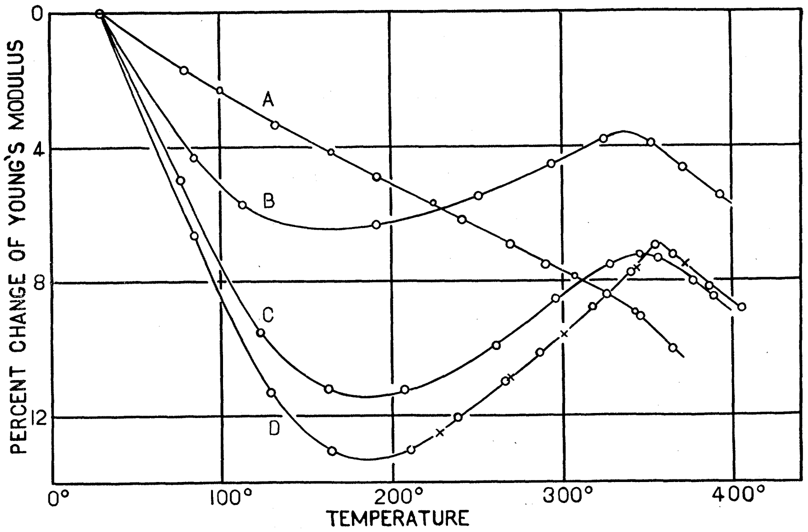

Fig. 4. Temperature dependence of Young's modulus for specimens *A* to *D*.

---

[7] Fink and Rohrman, Trans. Electrochem. Soc. **57**, 325 (1930); **58**, 403 (1930); **59**, 359 (1931).

[8] A description of the apparatus used in the production of the crystals is given in Phys. Rev. **39**, 345 (1932).

to D inclusive. The ordinates of these curves are $100(G_{30}-G_t)/G_{30}$ percent, where G_t denotes the value of Young's modulus at t°C. The circles represent observations taken between room temperature and 400°C and the crosses observations taken between 400°C and room temperature. Numerical values for the single crystal (curve D) are given in Table I. The observed values of Young's modulus at 30°C for the remaining specimens are given in Table II.

Table I. *Values of Young's modulus and its variation with temperature for a single crystal of nickel.*

T(°C)	$G_t\times10^{-11}$ dynes/cm²	$G_{30}-G_t/G_{30}$ $\times100$ percent	T(°C)	$G_t\times10^{-11}$ dynes/cm²	$G_{30}-G_t/G_{30}$ $\times100$ percent
33°	20.71	0.24	354°	19.33	6.91
84°	19.39	6.60	365°	19.26	7.21
129°	18.42	11.28	386°	19.04	8.27
164°	18.06	13.01	405°	18.94	8.79
211°	18.06	13.00	371°	19.00	7.48
238°	18.26	12.04	342°	19.17	7.68
267°	18.48	10.98	301°	18.78	9.56
287°	18.65	10.16	268°	18.50	10.90
317°	18.94	8.76	226°	18.18	12.44
340°	19.16	7.73	30°	20.76	0.00

Table II. *Values of Young's modulus for other specimens of nickel.*

Designation	Length at 30°C cm	Diameter cm	G_{30} dynes/cm²
A	8.118	0.48	21.41
B	6.666	0.48	19.86
C	7.820	0.48	20.77
D*	2.860	0.67	20.76
E	2.598	0.51	21.38
F	2.800	0.48	23.01

* The direction cosines of the cylinder axis with respect to the tetragonal axes of the crystal are 0.071; 0.68; 0.73.

These results indicate that the nature of the temperature dependence of Young's modulus below the Curie point is largely determined by the previous thermal history of the nickel at temperatures considerably above the Curie point. It appears that whether the nickel be pure or impure, single- or polycrystalline, the internal state characterized by a maximum at the Curie point and a minimum below it is produced by annealing at temperatures above 600°C. The difference between these maximum and minimum values of the modulus increases as the annealing temperature is raised. Further evidence in support of these conclusions is afforded by the following experiment: An annealed rod of impure polycrystalline nickel and a single crystal of the pure nickel were quenched at 1100°C. The behavior of both these specimens, as shown in Fig. 5, is substantially that of the hard drawn rod A.*

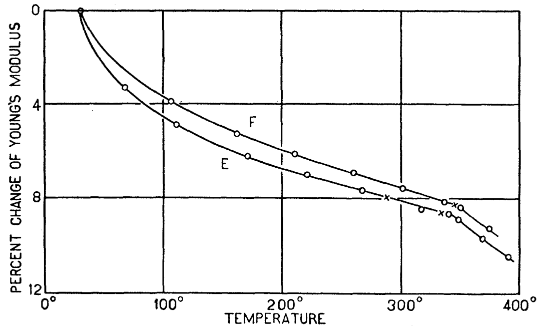

Fig. 5. Temperature dependence of Young's modulus for specimens E and F.

2. Internal friction

The coefficient of internal friction in solids is best defined by the relation

$$W=\xi S_0^2, \tag{14}$$

where W = energy loss per cm³ per cycle, ξ = coefficient of internal friction, and S_0 = amplitude of elastic stress. The relation (14) was first suggested by Kimball and Lovell.[9] The quantity ξ is related to the logarithmic decrement of free longitudinal vibration in a rod by the formula $\delta=\xi G$, and to the quantity λ by the formula $\pi\lambda=\xi G$.

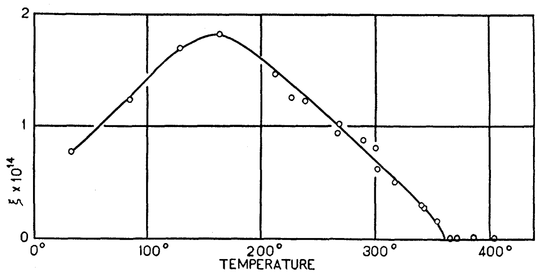

Fig. 6. Variation of internal friction with temperature in a single crystal specimen.

---

* These results are in qualitative agreement with measurements of the torsion modulus of nickel by W. Möbius, Phys. Zeits. **33**, 411 (1932).

[9] Kimball and Lovell, Phys. Rev. **30**, 954 (1927).

The present experimental method does not yield precise values of ξ, particularly when that quantity is small. However, the observed variation of ξ with temperature in the single crystal specimen is so striking that the results seem worth reporting at this time. They are shown in Fig. 6. A more complete study of this phenomenon is now in progress.

Discussion of Method

Since the present method for measuring Young's modulus has not yet come into general use, it is appropriate here to present the results of experiments designed to establish its validity.

The resonance curves, of which that shown in Fig. 2 is typical, show that Eq. (9) correctly describes the electrical behavior of the composite oscillator.

The quantity f_q is the frequency of free longitudinal vibration of the quartz rod alone and is measured in the manner described by Cady.[10] It follows that the only quantities appearing in Eq. (10) which are not independently measured are f_n and λ. The values of the quantity f_n calculated with this equation should be characteristic of the rod itself. This conclusion is verified by the results of the following experiments. (1), Different quartz rods affixed to the same specimen yield the same values of f_n. (2), Values of f_n are calculated from observations on a nickel rod 28 cm long, at all the resonance frequencies of the system between 20,000 and 120,000 cycles per second. The results are given in Table III. Only those values of f_n which are calculated from resonance frequencies near $0.5f_q$ and $1.5f_q$ deviate appreciably from the average. These deviations may be ascribed in part to the layer of adhesive and in part to the fact that the areas of the quartz and nickel rods are not matched at the interface. It appears that both these effects are negligible over a wide range of resonance frequencies lying above and below f_q. This conclusion, as far as it concerns the adhesive, is confirmed by a theory** in which the adhesive is treated as one part of a tripartite oscillator. A theoretical accuracy is gained by utilizing in the calculation of f_n a value of f_0 which lies near to f_q. (3), Table IV shows the percent change of Young's modulus with temperature for specimen A as calculated from observations taken at three different harmonic resonance frequencies. While, as has been stated, the observations at 90 kilocycles are the most precise, the result constitutes a further confirmation of the method. (4), The small circles of curve A (Fig. 4) represent data taken on the same rod after resticking to the same quartz rod.

Table III.

f_0 cycles/sec.	f_n cycles/sec.	$\bar{f}_n - f_n$ cycles/sec.
17,643	9294	− 1
25,884	9287	− 8
32,931	9273*	−22
39,706	9294	− 1
47,622	9290	− 5
56,280	9296	+ 1
65,137	9297	+ 2
74,027	9298	+ 3
82,810	9298	+ 3
91,230	9295	0
98,671	9292	− 3
105,134	9283*	−12
$f_q = 68087$	$\bar{f}_n = 9295$	

* Not included in average.

The absence in these test experiments of any evidence of systematic error indicates that the present method yields in f_n a number which is characteristic of the specimen rod alone. A very detailed study of the elastic behavior of the composite oscillator† shows that the relation of this quantity to Young's modulus for the material is, in fact, given by Eq. (1).

Precision of Measurements

1. Temperature

The longitudinal temperature gradient in the furnace is less than 0.2° per cm. The junction of the thermocouple is within 2 mm of the free end of the specimen. Calibrations of the couple against the melting points of tin, lead and zinc before and after the observations agree to 1° at 327.3°C and to 1.5° at 419.5°C.

---

[10] W. G. Cady, Proc. I.R.E. **10**, 83 (1922).

** This theory shows that the "sandwich" type of oscillator described by Quimby (6) is the worst that can be designed for the present purpose.

† This will appear as part of a paper shortly to be published by Lewis Balamuth.

TABLE IV.

Temp. °C	f_q cycles /sec.	f_0 cycles /sec.	$G_{30}-G_t/G_{30}$ $\times 100$ percent	f_0 cycles /sec.	$G_{30}-G_t/G_{30}$ $\times 100$ percent	f_0 cycles /sec.	$G_{30}-G_t/G_{30}$ $\times 100$ percent
30	87,650	63,950	0.00	90,368	0.00	115,830	0.00
79	87,640	63,540	1.65	89,690	1.69	115,060	1.69
132	87,610	63,145	3.34	89,043	3.34	114,337	3.28
192	87,540	62,750	4.94	88,440	4.84	113,620	4.84
241	87,440	62,450	6.15	87,930	6.15	113,034	6.17
290	87,280	62,100	7.52	87,410	7.40	112,420	7.40
325	87,150	61,810	8.63	86,990	8.41	111,900	8.41
345	87,060	61,630	9.25	86,756	8.99	111,550	9.09
369	86,930	61,334	10.44	86,390	9.88	111,038	10.08

2. Density and length

The densities of the specimens are determined at room temperature to 0.1 percent by the method of hydrostatic weighing. The length at 30°C is measured to 0.1 percent and the change of length with temperature to 400°C is measured to one percent of its value. Within this limit of accuracy the thermal expansions of single and polycrystalline specimens are the same. It is therefore assumed that the crystals expand equally in all directions, and hence that the fractional change in density is three times the fractional change in length.

3. Magnetization

The astatic magnetometer is sufficiently sensitive to show that the intensity of magnetization of the smallest specimen is less than 1.4 e.m.u. Magnetizations of this magnitude produce no detectable effect on the phenomenon.

4. Frequency

The frequency of the vacuum tube oscillator can be determined to one part in 100,000. The uncertainty in the frequency measurements arises from the flatness of the maximum and minimum of the resonance curve (Fig. 2). The consequent uncertainty in Young's modulus is not greater than 0.2 percent.

The major portion of the present work was done while the author held the Tyndall Fellowship and the Barnard Fellowship at Columbia University. In addition he wishes to acknowledge his indebtedness to Dr. Colin G. Fink and Dr. F. A. Rohrman who donated the pure nickel from which the crystals were grown; to Dr. Andrew Dingwall who made the spectroscopic analyses of the specimens; and to Dr. S. L. Quimby who suggested the problem and who followed the progress of its solution with helpful counsel and encouragement.