



ELASTIC MODULUS OF SEPARATED ISOTOPES
OF LITHIUM

Thesis for the Degree of M. S.
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Wayne Marvin Robertson
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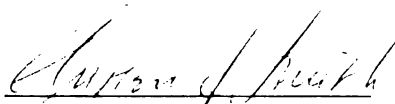
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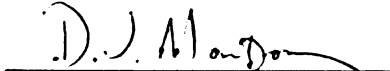
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ABSTRACT

The elastic modulus of the separated isotopes of lithium was measured at room temperature. The modulus was determined from observations of the resonant frequency of transverse vibration of extruded wires supported at one end. Lithium-6 (actually 96.1% Li-6, 3.9% Li-7) was found to have an elastic modulus of $7.93 \pm 0.21 \times 10^{10}$ dynes/cm², and lithium-7 (actually natural lithium, 92.5% Li-7, 7.5% Li-6) an elastic modulus of $7.98 \pm 0.33 \times 10^{10}$ dynes/cm². The values are identical within the limits of precision of the measurements, as would be expected on the basis of the usual theories. Results in the literature for the elastic modulus of lithium range from 4.9×10^{10} to about 9.2×10^{10} dynes/cm². It is believed that the average of the values reported here, 7.96×10^{10} dynes/cm², is within 20% of the absolute value for the modulus of polycrystalline lithium, and represents a more reliable estimate of this value than those reported previously.

INTRODUCTION

The elastic modulus of lithium is of interest because this metal is one of the few for which a theoretical calculation of this property can be made. A primary object of the investigation was to develop a simple technique to measure the elastic modulus. The work grew out of an investigation exploiting isotopic mass as a probe for studies of the solid state¹.

In the course of the development it was found that the values of the elastic modulus of natural lithium occurring in the literature had a wide variance. It seemed worthwhile to re-measure this quantity, as well as to determine it for the separated isotopes of lithium. It appeared possible to develop useful information at the same time that the feasibility of the method was being proved.

The method developed for use at room temperature would be particularly useful if it could be extended easily to higher or lower temperatures. Specifically, it could be used to investigate the martensitic transition² known to occur in natural lithium at about 80°K. The mechanism of the transformation might be elucidated by finding the effect of isotopic mass on the temperature and perhaps the rate of the transition. The detection of the transition is not always easy experimentally, and it is believed that the discontinuity in elastic modulus likely to appear at the transition might constitute a practicable indicator. A simple technique for measuring the elastic modulus at room temperature is necessary before the property can be studied at other temperatures.

THEORY OF THE METHOD

In measuring the elastic moduli of the materials under consideration, problems arise from the high degree of chemical reactivity of lithium and the small quantities of material available. Moreover, if the apparatus is to be suitable for investigations of temperature coefficient of the elastic modulus, differential measurements between isotopes should be made simultaneously on two specimens of different isotopic composition contained in the same chamber. To get to liquid air temperatures, several experimental complications arise. Observation of the resonant frequencies of vibration of a rod was chosen as a promising method, and as one that would be simple to extend to low temperatures.

The method used was the transverse vibration of a wire clamped at one end and free at the other. Thus, the wire behaved as a cantilever beam or bar.

Consider a straight, homogeneous bar of uniform cross-section, symmetrical about a central plane. The differential equation for the motion of the bar perpendicular to this plane is

$$\frac{\partial^4 y}{\partial x^4} = -\frac{\rho A}{EI} \frac{\partial^2 y}{\partial t^2} \quad (1)$$

where y = displacement from equilibrium of the plane of symmetry,

x = distance from clamped end of bar,

ρ = density (mass per unit volume) of the material,

A = cross-sectional area of the bar,

E = elastic modulus (Young's modulus) of the material,

I = area moment of inertia about neutral plane,

t = time.

The flexural rigidity EI is constant along the bar. This equation is derived and solved for several boundary conditions in standard texts.³

For the solution, one writes $y(x, t)$ as a product of a function of x alone, $Y(x)$, and a periodic function of t , $\exp 2\pi i \nu t$, where $i = \sqrt{-1}$, and ν is the frequency of vibration. Thus,

$$y(x, t) = Y(x) e^{2\pi i \nu t}. \quad (2)$$

On substituting equation (2) into (1), it is found that $Y(x)$ must satisfy the equation

$$\frac{\partial^4 Y}{\partial x^4} = 16\pi^4 \mu^4 Y, \text{ where } \mu^4 = \frac{\rho \nu^2 A}{4\pi^2 EI}. \quad (3)$$

The general solution of this equation is

$$\begin{aligned} Y &= c_1 e^{2\pi \mu x} + c_2 e^{-2\pi \mu x} + c_3 e^{2\pi i \mu x} + c_4 e^{-2\pi i \mu x} \\ &= A \cosh(2\pi \mu x) + B \sinh(2\pi \mu x) + C \cos(2\pi \mu x) + D \sin(2\pi \mu x) \end{aligned} \quad (4)$$

For a given problem, the boundary conditions determine the values of A , B , C , D , (or alternatively c_1 , c_2 , c_3 , c_4) and μ that are allowable.

For a bar clamped at one end and free at the other, both the deflection and the slope must be zero at the clamped end, and both the bending moment and the shearing force must be zero at the free end. With a bar of length L having the clamped end at $x = 0$, the boundary conditions are:

$$\begin{aligned}
 Y(x)_{x=0} &= 0 & \left(\frac{\partial^2 Y}{\partial x^2} \right)_{x=L} &= 0 \\
 \left(\frac{\partial Y}{\partial x} \right)_{x=0} &= 0 & \left(\frac{\partial^3 Y}{\partial x^3} \right)_{x=L} &= 0.
 \end{aligned}
 \tag{5}$$

On applying these boundary conditions, it is found that μ must satisfy the transcendental equation

$$\cosh(2\pi\mu L) \cos(2\pi\mu L) + 1 = 0$$

This equation must be solved numerically to get actual values for μL . The lowest three roots are, $(2\pi\mu_1 L) = 1.875$, $(2\pi\mu_2 L) = 4.694$, $(2\pi\mu_3 L) = 7.855$. If these numbers are indicated by the notation s_n , then μ_n corresponding to a given s_n is

$$\mu_n = \frac{s_n}{2\pi L}. \tag{6}$$

From equations (3) and (6),

$$\mu_n^4 = \frac{\rho \nu_n^2 A}{4\pi^2 EI} = \frac{s_n^4}{(2\pi)^4 L^4}. \tag{7}$$

For a wire of given dimensions, this equation gives the natural frequencies of vibration, where ν_n has now been substituted for ν .

Equation (7) may be rearranged to give the elastic modulus E in terms of the dimensions of the bar and the frequency:

$$E = \frac{4\pi^2 \rho A L^4 \nu_n^2}{s_n^4 I}. \tag{8}$$

For a bar of circular cross section, $A = \pi d^2/4$ and $I = \pi d^4/64$. Thus,

$$E = \frac{64\pi^2 \rho L^4 \nu_n^2}{s_n^4 d^2}. \tag{9}$$

Equation (9) applies to a bar of circular section; it also applies to an elliptical bar if the motion is perpendicular to one of the axes of symmetry of the ellipse. In this case d is the principal diameter in the direction of vibration. Numerical values were substituted into this equation for the calculation of E .

For a bar subject to damping forces and excited by some external force of frequency ν , two terms must be added to the differential equation. The first is proportional to $\partial y / \partial t$ and takes damping into account; the second represents the driving force, assumed periodic in time, conveniently written $c_5 \exp 2\pi i \nu t$, where c_5 is some constant. If the damping forces are small the frequency of maximum amplitude of vibration, the resonant frequency, will be the natural frequency for free vibration of the bar, ν_n . Thus, ν_n can be measured by varying ν and noting its value when the amplitude is a maximum.

In the above analysis the various damping effects - air damping, internal friction and energy loss to the support - were considered to be small. If damping is large, the resonant frequency will be lowered and the calculated value of E will be too low. Therefore, in applying equation (9) to the calculation of E , it is necessary to verify that the damping actually is small.

The theory of the effect of air damping has been considered by Stokes⁴. Karrholm and Schroder⁵ applied this theory to the vibration of fibers supported at one end. They concluded that the theory applied to this situation and that air damping was negligible except for very fine fibers. Stauff and Montgomery⁶ reached similar conclusions for a stretched wire. Application of Stokes' theory to these experiments shows

that, because of the large wires used, air damping was completely negligible.

The effect of internal damping in determination of E by transverse vibration is generally considered to be exceedingly small (Richards⁷, p. 86). Fine⁸ indicates that if damping is small, the curve of amplitude of vibration versus frequency will be very sharp, having its peak at the resonant frequency. In the experiments the actual band width due to damping was not determined, but it was observed that the resonant frequency appeared quite sharp. This would indicate that the effect of internal damping was indeed small and could safely be neglected.

The third possible source of damping was loss of energy at the support, the steel extrusion die. Because the wire was extruded directly and left attached to the die, the wire was clamped quite tightly by the die. The support was very massive compared to the wire sample, so that no appreciable inertial energy was transferred to the support.

SOURCE AND PURITY OF MATERIALS

The lithium-6 used was purchased from Oak Ridge National Laboratory and was purified by redistillation. The material had the following analysis:

Li-6 Lot No. SS5(a) - redistilled 1045 mg

Isotopic analysis (mass number and atomic percent): 6, $96.1 \pm .1$; 7, $3.9 \pm .1$.

Spectrographic Analysis (element and weight percent, precision $\pm 50\%$):

Ag	T	Fe	0.5	Ni	≤ 0.01 T
Al	≤ 0.01 T	K	≤ 0.02 T	Pb	≤ 0.02 T
Ba	≤ 0.02 T	Mg	0.02	Si	≤ 0.05
Ca	0.05	Mn	≤ 0.01	Sn	≤ 0.01
Cr	≤ 0.01 T	Mo	≤ 0.01	Sr	≤ 0.01
Cu	0.01	Na	0.03	V	≤ 0.02

The natural lithium was produced by the Lithium Corporation of America.

It was their low sodium grade, in the form of $3/8$ inch diameter rods with the following specifications:

Na	0.005%
K	0.01
Ca	0.02
N	0.06
Fe	0.001

Preparation of Samples

The samples were stored in mineral oil but nevertheless acquired a coating, probably mostly nitride. This coating could be removed by scraping or cutting and after removal a reasonably clean surface could be retained by coating with oil. The cleaned sample was formed into a small slug that would fit into the extrusion die.

APPARATUS AND PROCEDURE

The electrical apparatus was similar to that used by Stauff⁹. It was essentially a string electrometer as described by Montgomery and Milloway¹⁰. Because the wires used were stiffer than the stretched fibers used by Stauff, it was necessary to modify the apparatus somewhat to get adequate driving force. The electrical circuit diagram is shown schematically in Figure 1.

Brass electrodes diametrically opposed were connected through a step-up transformer to an amplifier fed by a variable-frequency audio oscillator. The oscillator was adjusted until resonance of the fiber was observed. The frequency was read directly from the oscillator dial. The oscillator was calibrated by observing Lissajous figures of the oscillator output against line frequency on a cathode-ray oscilloscope. The calibration was checked periodically, and was found to be quite stable.

The wires were extruded from a small steel die directly into a glass chamber. Because lithium reacts with oxygen and nitrogen in the air, rapidly forming a dense coating of oxide and nitride on the freshly extruded, bare wire, it was necessary to fill the glass chamber with a gas inert with respect to lithium. Carbon dioxide was found to be quite satisfactory. The chamber was evacuated and purged several times with carbon dioxide; then the wire was extruded.

The lithium wire had a tendency to curl as it was extruded. To

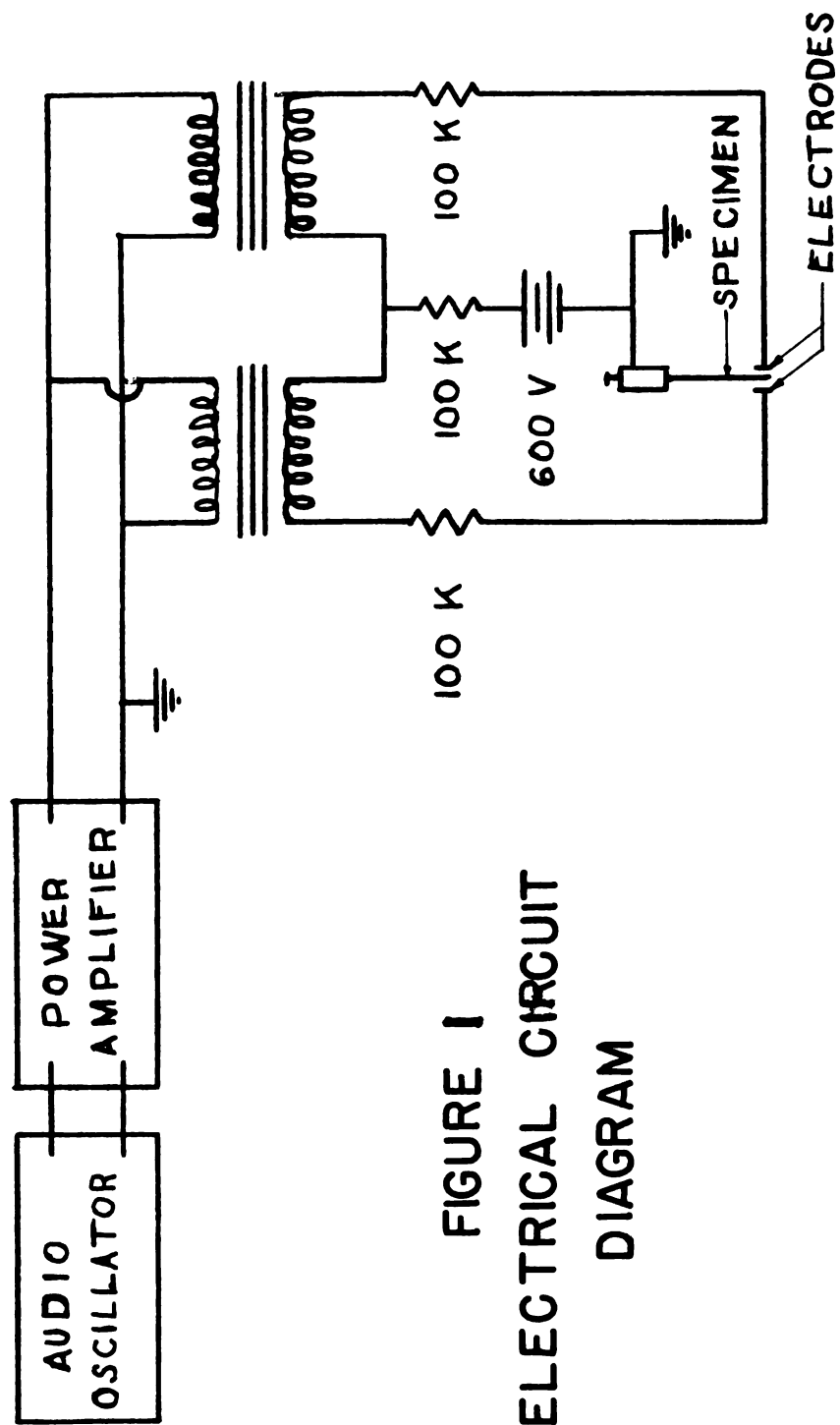


FIGURE 1
ELECTRICAL CIRCUIT
DIAGRAM

prevent this a small glass tube was brought up to the extrusion die through the bottom of the chamber and the wire was extruded directly into the small glass tube. This gave wires that had only a moderate variation from perfect straightness. After extrusion the wire was allowed to stand in the small glass tube for ten to fifteen minutes. If it was not allowed to stand the wire would continue to creep for a few minutes and would move from the center of the chamber. After the wire had been allowed to stand the small glass tube was pulled down out of the way for determination of the resonant frequency. A schematic diagram of the chamber with a wire extruded is shown in Figure 2.

Several observations were made of a single wire by changing its length. A short wire first was extruded, the length, diameter and resonant frequency of it were measured, and then more wire was extruded.

Wire length was measured with a cathetometer microscope. At least two separate length measurements were made of each wire to be sure that the measurement was accurate. Wire lengths were varied from about 3 cm to 10.5 cm.

Diameter measurements were made with a scale in the eyepiece of the cathetometer microscope. The scale was calibrated by observing several wires of known diameter over the whole range of diameters used. The diameter was measured at two to five different positions along the wire. The diameter was measured at 0° , 45° , 90° and 135° as the wire was turned at each position; this gave eight to twenty different readings of the diameter. The readings were averaged so that the average maximum diameter and the average minimum diameter were known for each length of wire. Wire diameters varied from about .064 cm to .097 cm.

Resonance was observed through the cathetometer microscope. A bright

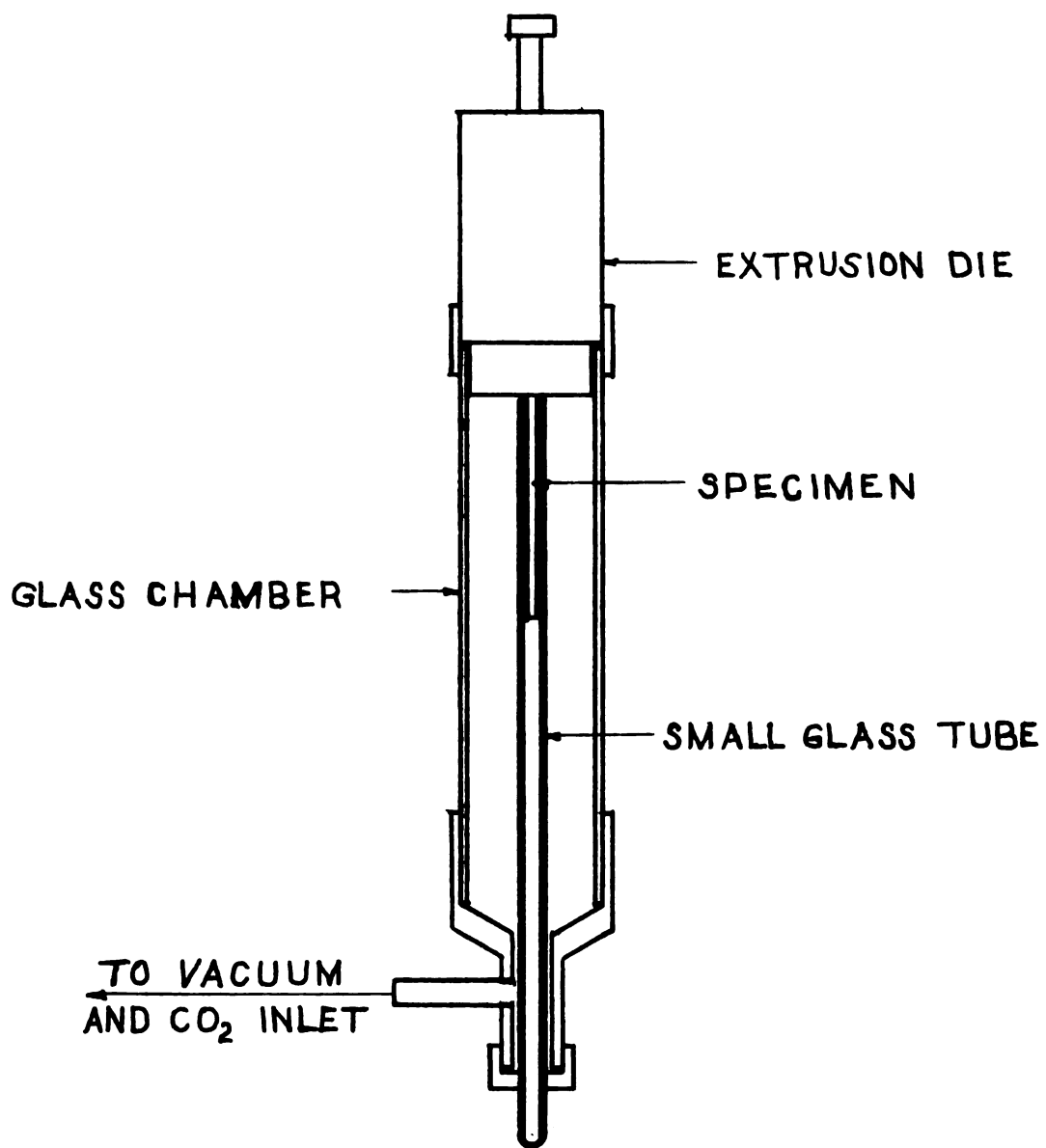


FIGURE 2
CONTROLLED ATMOSPHERE CHAMBER

light was placed in front of the wire so that the wire could be observed by reflected light. The microscope was adjusted so that bright spots would appear on the wire. Resonance could be observed easily by noting the broadening of a given spot as the frequency was varied through the resonant frequency. The magnitude of vibration was usually considerably less than half the diameter of the wire.

The wire was turned to five different positions for a given length and the resonant frequency measured. In almost all cases two different frequencies were noted; these corresponded to the maximum diameter and the minimum diameter of the elliptical cross-section.

The fundamental mode of vibration was observed in all the wires. The first overtone was observed in some of the wires but, because the frequency was higher and the amplitude of vibration much smaller, the overtone was much harder to observe. No calculations were made using the first overtone, but in each case the frequency of the overtone was exactly that expected from the frequency of the fundamental.

RESULTS

Twelve different wires of lithium were vibrated, seven of natural lithium, and five of lithium-6. Each wire was vibrated for one to five different lengths. For each measured length the two slightly different fundamental frequencies and the maximum and minimum diameters were measured. From these data the elastic modulus was calculated for each length. The densities used in the calculations were those given by Snyder and Montgomery¹¹: Lithium-6, $0.460 \pm .002$ g/cc, natural lithium, $0.531 \pm .003$ g/cc. The several values of E determined for each length of a given wire were averaged to give the value of E for that wire. These data are presented in Table I for natural lithium and in Table II for lithium-6.

The average values of E for the wires of natural lithium and for the wires of lithium-6 were averaged to give a value of E for natural lithium and for lithium-6. The values of E for all wires and the average values for the two types of lithium are given in Table III. It is seen that within the experimental error, the elastic moduli of natural lithium and of lithium-6 are the same.

TABLE I - ELASTIC MODULUS OF NATURAL LITHIUM

Wire No.	ν_1 (c/s)	d(cm)	L(cm)	E dynes/cm ²
1	257	.0693	3.694	6.94
	280	.0728	3.694	7.46
	102	.0640	5.817	7.81
	112	.0737	5.817	7.16
				<u>7.34</u>
2	319	.0782	3.670	8.19
	324	.0791	3.670	8.21
	179	.0764	4.880	8.46
	183	.0791	4.880	8.27
	111	.0773	5.960	7.07
	121	.0800	5.960	7.78
	69	.0773	7.714	7.65
	71	.0791	7.714	7.73
	39.5	.0773	10.190	7.62
	41.5	.0782	10.190	8.24
				<u>7.92</u>
3	273	.0791	3.929	7.67
	278	.0800	3.929	7.75
	139	.0791	5.520	7.81
	143	.0800	5.520	8.08
	86	.0764	7.011	8.35
	90	.0791	7.011	8.49
	57	.0773	8.566	7.94
	60	.0800	8.566	8.22
	45.5	.0773	9.568	7.84
	47.5	.0791	9.568	8.22
				<u>8.03</u>
4	271	.0897	3.924	5.86
	293	.0923	3.924	6.45
	175	.0906	5.392	8.57
	179	.0923	5.392	8.65
	103	.0897	7.135	9.27
	105	.0923	7.135	9.11
	52	.0897	9.870	8.65
	53	.0923	9.870	8.46
				<u>8.13</u>

TABLE I (continued)

5	384	.0897	3.600	8.38
	389	.0942	3.600	<u>7.81</u>
				8.10
6	324	.0914	3.949	8.30
	334	.0958	3.949	7.97
	175	.0924	5.346	7.99
	183	.0968	5.346	7.99
	101	.0933	7.039	7.75
	105	.0951	7.039	8.02
	67	.0915	8.550	7.78
	71	.0959	8.550	7.92
	49	.0924	9.998	7.62
	52	.0959	9.998	<u>7.97</u>
				7.93
7	319	.0914	4.025	8.65
	324	.0952	4.025	<u>8.22</u>
				8.43

TABLE II - ELASTIC MODULUS OF LITHIUM-6

Wire No.	ν_1 (c/s)	d (cm)	L (cm)	E dynes/cm ²
8	319	.0782	3.821	8.32
	324	.0825	3.821	7.73
				<u>8.03</u>
9	435	.0781	3.265	8.17
	440	.0808	3.265	7.82
	192	.0781	4.898	8.17
	198	.0817	4.898	7.99
	130	.0764	6.009	8.81
	133	.0799	6.009	8.46
	96	.0781	6.924	8.15
	99	.0808	6.924	8.15
				<u>8.22</u>
10	218	.0791	4.563	7.73
	227	.0835	4.563	7.52
	124	.0773	5.995	7.89
	130	.0817	5.995	7.68
	79	.0782	7.528	7.73
	83	.0826	7.528	7.59
				<u>7.68</u>
11	339	.0782	3.753	8.74
	339	.0817	3.753	8.03
	158	.0791	5.414	8.06
	160	.0808	5.414	7.87
	89	.0782	7.184	8.13
	91	.0808	7.184	7.99
	57	.0764	8.851	8.10
	59	.0808	8.851	7.70
	43.5	.0773	10.020	7.49
	45	.0808	10.020	7.31
				<u>7.94</u>
12	329	.0773	3.720	8.10
	339	.0799	3.720	8.03
	224	.0773	4.430	7.59
	229	.0799	4.430	7.42
				<u>7.79</u>

TABLE III - ELASTIC MODULUS OF NATURAL LITHIUM AND LITHIUM-6
AT ROOM TEMPERATURE

Material	Wire No.	$E \times 10^{-10}$ (dynes/cm ²)
Natural Lithium	1	7.34
	2	7.92
	3	8.03
	4	8.13
	5	8.10
	6	7.93
	7	8.43
Average of Values for Natural Lithium		7.98 ± 0.33
Lithium-6	8	8.03
	9	8.22
	10	7.68
	11	7.94
	12	7.79
Average of Values for Lithium-6		7.93 ± 0.21
Average E for natural lithium and lithium-6		7.96

ACCURACY OF MEASUREMENTS

The accuracy of the density values used was about $\pm 0.4\%$. The length measurements were estimated to be accurate within $\pm 0.1\%$, the principal uncertainty being the measurement at the point of support. It could not be determined if the wire was supported exactly at the edge of the die, or if it was supported slightly back of the edge.

The frequency readings were estimated to be accurate within $\pm 0.1\%$. The diameter measurements were estimated to be accurate within $\pm 1\%$.

Considering these variations, the calculated values of E are estimated to be accurate within about 3%.

Considering the internal consistency of the measurements, the calculated values are considered to be accurate within 5%. In view of the uncertainties in the method itself, the inaccuracy in the absolute values does not exceed 20%.

DISCUSSION

Measurement of elastic modulus of lithium by forced vibration of extruded wire was found to be feasible. Extrusion directly into a controlled atmosphere chamber eliminated problems arising from the high chemical reactivity of lithium. With minor modification of the apparatus the measurements could be extended to higher or lower temperatures. The other alkali metals could also be investigated easily with the same apparatus.

The values of the elastic modulus for natural lithium (92.5% Li-7) and for lithium-6 (96.1% Li-6) at room temperature were found to be identical within the experimental error. It is natural to conclude that the values for the isotopes of lithium are identical.

This result is to be expected. Mott and Jones¹² give a method for calculating the elastic modulus of a material based on the force field of the atoms and the atomic volume of the atoms. The force fields of lithium-6 and lithium-7 are identical. The lattice constants (and therefore the atomic volumes) of the two isotopes were shown by Covington and Montgomery¹³ to differ by just a few parts in ten thousand at room temperature. Therefore, within the experimental error, the elastic modulus would not be expected to vary between lithium-6, lithium-7, and natural lithium (92.5% Li-7) at room temperature.

The force field of the atoms of the isotope would remain identical as the temperature varied. However, the difference in atomic volume would

be expected to become larger as the temperature approaches absolute zero. Therefore measurements of the elastic modulus at low temperatures would be likely to show a difference between the isotopes.

The measurements give the elastic modulus for natural lithium as $7.98 \pm 0.33 \times 10^{10}$ dynes/cm², and for lithium-6, $7.93 \pm 0.21 \times 10^{10}$ dynes/cm². The average of both sets of measurements is 7.96×10^{10} dynes/cm².

Bridgman¹⁴, in measuring the elastic modulus of polycrystalline natural lithium in bending, found a value of 4.9×10^{10} dynes/cm².

Bender¹⁵ measured the modulus in combined bending and torsion at 90°K and found a value of 11.5×10^{10} dynes/cm². The modulus decreases as the temperature is raised; a reasonable variation between 90°K and 300°K is about 20%. Thus Bridgman's value would be about 9.2×10^{10} dynes/cm² at room temperature.

The value determined in the present experiments agrees as to the order of magnitude. It is believed to represent a somewhat better value for polycrystalline lithium.

Values of the single-crystal elastic constants c_{11} , c_{12} , and c_{44} were determined by Nash and Smith¹⁶, and, as quoted by Huntington¹⁷, are 14.8×10^{10} , 12.5×10^{10} , and 10.8×10^{10} dynes/cm² respectively at 78°K. From these values Huntington calculated the elastic compliances s_{11} , s_{12} and s_{44} and found the values 29.5×10^{-12} , -13.5×10^{-12} , and 9.26×10^{-12} cm²/dyne respectively. As Huntington points out, the calculation of the elastic modulus for a polycrystalline aggregate from the single crystal constants is difficult both theoretically and experimentally.

Voigt¹⁸ derived an expression for averaging the elastic constants for a cubic crystal:

$$E = \frac{(c_{11} - c_{12} + 3c_{44})(c_{11} + 2c_{12})}{2c_{11} + 3c_{12} + c_{44}}.$$

Reuss¹⁹ derived an expression for averaging the elastic compliances to calculate E:

$$E = \frac{5}{3s_{11} + 2s_{12} + s_{44}}.$$

Using Voigt's expression, E for lithium turns out to be approximately 18×10^{10} dynes/cm². By Reuss' method, E is approximately 7×10^{10} dynes/cm². The difference between these two values is not too surprising because of the differences between the methods of calculation.

The elastic modulus decreases as the temperature is raised, so that the room temperature values of the quantities just calculated would probably be about 20% lower than at 78°K. Because of the uncertainties involved in the calculations with the single crystal constants, these calculations would not invalidate the results obtained in the present experiments.

CONCLUSIONS

1. Measurement of the elastic modulus of lithium by extruding wire into a controlled atmosphere chamber and leaving the wire supported by the die was found to be feasible experimentally.

2. The elastic moduli of natural lithium (92.5% Li-7, 7.5% Li-6) and of lithium-6 are identical at room temperature (25°C) within the experimental error.

3. The elastic modulus of natural lithium was found to be $7.98 \pm 0.33 \times 10^{10}$ dynes/cm²; the elastic modulus of lithium-6 at room temperature was found to be $7.93 \pm 0.21 \times 10^{10}$ dynes/cm². An average of both sets of measurements gives a value for the elastic modulus of lithium of 7.96×10^{10} dynes/cm² at room temperature.

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