# **Resistivity and Temperature Coefficient of Pure Molybdenum**

R. A. HOLMWOOD and R. GLANG IBM Components Division, Poughkeepsie, N. Y.

Resistivity and temperature coefficient of 99.999% molybdenum, zone purified in vacuum and nearly single crystalline, have been measured. The resistivities are 5.00  $\mu\Omega$  cm. at 0° C., 5.57  $\mu\Omega$  cm. at 25° C., and 740  $\mu\Omega$  cm. at 100° C. with a maximum error of +1.0, -0.5%. Between 0° and 100° C., the resistivity increases more than linearly. The closest linear approximation in this range is obtained with an average resistivity coefficient of 0.00482 per centigrade. The ratio of resistivities at -196° and 0° C. is 0.097. Comparison with data published previously indicates a purity of the material investigated which is higher than that of former single crystalline as well as spectroscopically pure products.

**D**URING the investigation of electrical properties of vacuum evaporated molybdenum films, the ideal resistivity of the metal was established. A survey of published data showed varying values depending on the purity and lattice perfection of the available products.

### **EXPERIMENTAL**

For this investigation, molybdenum rod zone purified in a vacuum was used (M. R. C. Manufacturing Corp., Orangeburg, N. Y.). The purity claimed is 99.999%. A typical analysis of triple pass material as given by the vendor lists the following impurities (p.p.m.): 5.0 carbon, 1.0 oxygen, 0.5 nitrogen, 0.3 hydrogen, 5.0 iron. From this material, a bar of approximately square cross section was cut, and damaged material on the surfaces was removed by lapping and etching. Metallurgical cross sectioning of the bar near both ends revealed no grain boundaries. Hence, the specimen must have been almost or fully single crystalline.

The density of the zone purified material was determined on a piece of nearly 18 grams by weighing in air and water and was in agreement with published data (3).

Two pairs of leads were soldered to the ends of the cut molybdenum bar. With a current of 500 ma. applied through the outer leads, the voltage drop along the bar was measured with an additional probe attached to a micrometer and travelling between the inner leads. The voltage vs. distance data were evaluated by the method of least squares to determine the spacing of the inner leads and to check the cross sectional uniformity of the bar.

The electrical resistance of the molybdenum bar was measured at 5° intervals from 25° to 100°C. with the sample and a calibrated thermometer submerged in a silicone oil bath. Measurements were also taken at 0° and  $-196^{\circ}$ C. in ice water and liquid nitrogen. The uncertainty of the temperature measurement was negligible at 0° and less than  $\pm 0.3\%$  at 100°C. The voltage between the inner leads was measured with a digital voltmeter, rated  $\pm 0.05\%$  (Hewlett-Packard, Model 2401 B). The sample current was determined with a d.c. milliameter (Greilbach Instr. Corp., model 700), whose accuracy is only 0.5%. However, this instrument had been calibrated for the particular range used here against a secondary standard (Singer Matrix Corp., voltage divider). Current readings have been

 $\pm 0.1\%$ . The measured experimental data and associated error limits are as follows:

corrected accordingly, and the error was thus reduced to

Weight of Mo har $(W)$	0.0564  gram + 0.1%
Density of Mo at $25^\circ$ (d.,)	$10.21 \pm 0.01\%$
Tetal length of her at $25^{\circ}$ C $(U_{2})$	$255 \text{ cm} \pm 0.1\%$
1 otal length of bar at 25 $C$ . ( $L_{25}$ )	2.00 cm. $\pm 0.176$
Average cross section at 25° C.	0.000 10=3 2 . 0.007
$[A_{25} = (W/d_{25}L_{25})]$	$2.093 \cdot 10^{\circ} \text{ cm}^{\circ} \pm 0.2\%$
Inner lead spacing at 25° C. $(l_{25})$	$2.157 \text{ cm}. \pm 0.1\%$
Measured voltage (V), maximum error	$\pm 0.05\%$
Measured current (I), maximum error	$\pm 0.1\%$

Resistivity values were calculated under consideration of dimensional changes of the bar owing to temperature:

$$\rho(T) = \frac{V}{I} \cdot \frac{A_{25}}{l_{25}} \left[ 1 + c \left( T - 25 \right) \right]$$
(1)

where  $c = 4.8 \cdot 10^{-6}$  per centigrade is the linear coefficient of thermal expansion of molybdenum in the interval 0° to 100° C. (4). Hence, the maximum random error of the resistivity data is  $\pm 0.5\%$ . In addition, variations of the actual cross section along the length of the bar can affect the measurements but only toward resistance values which are too high. The travelling probe data and microscopic measurements of the smaller bar dimensions indicate that the resistivity values may be at worst 0.5% high on account of deviations from the calculated average cross section. This would be a systematic error affecting all resistivities proportionately but not the temperature coefficient.

	Resistivity of 99.999% Molybdenum Resistivity $(10^{-6} \Omega \text{ Cm.})$			
Temp., ° C.	Measured	Calculated	ed Differenc	
-196	0.486	•••		
0	5.00	4.98	+0.02	
25	5.57	5.58	-0.01	
50	6.17	6.18	-0.01	
75	6.78	6.78	0.00	
100	7.40	7.38	+0.02	

Table II. Resistivity and Temperature Coefficient of Pure Molybdenum

Material Investigated	Resistivity $(\mu \Omega Cm.)$		T. C. $\alpha$ ., 0° – 100° C.	$\alpha \rho(25^{\circ})$ (10 <sup>-10</sup> Ω Cm.	Lit.
	0° C.	25° C.	(Degree <sup>-1</sup> )	Degree <sup>-1</sup> )	Reference
Single crystal wire	5.03	5.62	0.00473	266	(1)
Pure Mo			0.00459		(1)
Pure single crystal	5.05	5.65	0.00476	269	(2)
Wire			0.00459		(8)
Wire, 99.85%	5.17	5.77	0.00462	266	(5)
Wire, 99.85%	5.25	5.85	0.00461	270	(5)
Spectroscop, standard					. ,
rod. Johnson-Matthey	5.05	5.65	0.00475	268	(7)
Zone purified rod.					
99.999% pure	5.00	5.57	0.00482	268	Author

## RESULTS

The experimental resistivity values are shown in column 2 of Table I. A linear approximation of the resistivity as a function of temperature has been calculated from the experimental data, applying the least squares method over the interval 0° to 100° C.:

$$(T) = \rho_o + \alpha \rho_o T \tag{2}$$

The average temperature coefficient of resistivity in this range was thus  $\alpha = 0.00482$  per centigrade. Equation 2 was then used to calculate the resistivity values which are shown in column 3 of Table I. The differences between measured and calculated values listed in the last column indicate that the true temperature function is not linear, which is reflected clearly if all experimental data are plotted graphically. This behavior is to be expected since molvbdenum has a relatively high Debye temperature of 360° to 380° C. (6).

A comparison of resistivities and temperature coefficients published by various authors is shown in Table II. The data of this investigation indicate the highest purity of molybdenum which has been reported so far. This is confirmed by the resistance ratio

$$\frac{R \ (-196^{\circ} \text{ C}.)}{R \ (0^{\circ} \text{ C}.)} = 0.097 \pm 1\%$$

For comparison, Kannuluik reported ratios of 0.1814 and 0.1705 for two molybdenum wires at the two temperatures -183° and 0° C.

As shown in the next to last column of Table II, Mathiessen's Rule is valid for all molybdenum samples of reasonably good purity. According to Geiss and van Liempt (2), this is also true for drawn wires with a considerable degree of cold deformation.

#### LITERATURE CITED

- Geiss, W., van Liempt, J.A.M., Z. Metallk. 17, 194 (1925). (1)
- Geiss, W., van Liempt, J.A.M., Z. Physik 41, 867 (1927). "Gmelins Handbuch der Anorg. Chemie," 8th ed. p. 44, 1935. (2)
- (3)
- Harwood, J., Ed., "The Metal Molybdenum," p. 10, Am. Soc. (4)Metals, Cleveland, Ohio, 1958.
- Kannuluik, W.G., Proc. Roy. Soc. London A 141, 159 (1933). (5)
- Meissner, W., Voigt, B., Ann. Physik (5), 7, 911 (1930). (6)
- Tye, R.P., "Report on Conference on Nb, Ta, Mo, W" in (7)Sheffield, p. 169, Elvesier, Amsterdam, 1961.
- Zwikker, C., Physica 7, 71 (1927). (8)

RECEIVED for review September 4, 1964. Accepted February 8, 1965.

## Volume Changes of Mixing of Some Ethanol-Hydrocarbon Systems

FRANCISCO PARDO<sup>1</sup> and H. C. VAN NESS

Chemical Engineering Department, Rensselaer Polytechnic Institute, Troy, N.Y.

Volume changes of mixing at 25° and 45° C. are reported for the liquid binary systems made up of ethanol with cyclohexane, toluene, o-xylene, m-xylene, and p-xylene.

 $\mathbf{P}_{ ext{RECISE}}$  measurements have been made of the volume changes of mixing at 25° and 45° C. for binary liquid systems consisting of ethanol with cyclohexane, toluene, o-xylene, m-xylene, and p-xylene. The apparatus used consisted of a dilatometer, which is fully described elsewhere (4). It

followed the general design of the apparatus described by Anderson (1), but incorporated several modifications. The purities of the materials used are indicated in Table I.

To test the results obtained with this dilatometer for agreement with those obtained by earlier investigators, data were first obtained for the carbon tetrachloride-cyclohexane system at 30°C. The excellence of agreement is shown in Figure 1.

<sup>&</sup>lt;sup>1</sup>Present address: Esso Engineering, Florham Park, N. J.