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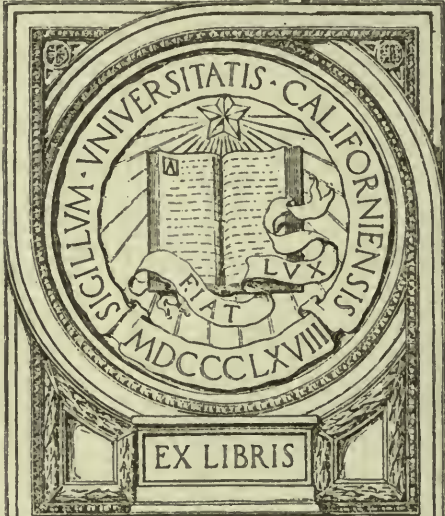
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EXCHANGE



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WIRE AT INCANDESCENT TEMPERATURES

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JUL 10 1924

A THESIS

PRESENTED TO THE FACULTY OF THE GRADUATE COLLEGE OF THE
STATE UNIVERSITY OF IOWA IN PARTIAL FULFILLMENT OF
THE REQUIREMENTS FOR THE DEGREE OF
DOCTOR OF PHILOSOPHY

BY

WILLIAM SCHRIEVER

JULY, 1921

UNIV. OF
CALIFORNIA

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BY WILLIAM SCHRIEVER

ABSTRACT

Rigidity of a 10-mil drawn tungsten wire to 2000°K.—To avoid errors due to end effects, two lengths of the same wire were mounted in series and heated by the same current so that the middle portions of both were at the same temperature. The apparatus was placed in an evacuated tube; the torque was applied magnetically and measured by the twist in a cold fine tungsten wire. The rotations of the mirrors attached to the ends of the heated wires were noted within a few seconds after application of the torque, since at the higher temperatures even a twist of 0.1° per cm exceeded the elastic limit. On the three successive runs made, the rigidity increased progressively due to the crystal growth in the temperature range above 1600°K. For the recrystallized or equiaxed wire the values of the modulus obtained on the third run are: 21.7 at 300°K, 21.0 at 1030°K and 3.4 at 1985°K, all times 10^{11} dynes/cm².

INTRODUCTION

UP to the present time, as far as is known to the writer, the simple rigidity of a drawn tungsten wire at incandescent temperatures has not been determined.

Koch and Dannecker¹ seem to have made the first determination of the simple rigidity of a metal at temperatures which approach the melting point of the material; they made observations on wires of some twenty-two elements and alloys using the usual torsion-pendulum method. They allowed for the changes in periods caused by the temperature gradients along the wires, by making several assumptions and approximations together with graphical interpolations and extrapolations—a very indirect method.

When the temperatures desired are not too high the whole apparatus may be heated in some sort of an oven and the temperature gradients in the wires, the "end-effects," thereby avoided. Since an apparatus which could be heated to 2000°K did not seem feasible, the following simple method of allowing for the end-effects was devised. The same torque was applied to two different lengths of the same piece of electrically heated wire whose central portions were at the same temperature. When the angle of twist of the shorter was subtracted from that of the

¹ Koch and Dannecker, *Ann. der Phy.* **47**, 197-227, 1915

longer the difference was the angle of twist produced by that torque in a uniformly heated wire of a length equal to the difference of the lengths of the two wires. This scheme necessitated using a static method for making the simple rigidity determinations. Since glowing tungsten is oxidized in the open air or in the presence of water vapour, the wires and attached apparatus were enclosed in an evacuated vessel.

Streintz² and Meissner³ have found that the simple rigidities of all the metals which they investigated are independent of the manner of heating. While it is possible, it is not probable that tungsten wire would show a difference. In fact Dodge⁴ has found that Young's modulus of tungsten wire is independent of the method of heating between 0° and 1000°C. The writer has thus far not attempted to check this point for higher temperatures experimentally.

APPARATUS AND PROCEDURE

A schematic drawing of the wires and attached apparatus, all of which was enclosed in a large glass tube, is shown in Fig. 1. The short length and the long length of the tungsten wire on which the simple rigidity determinations were to be made are shown at L_1 and L_2 respectively. L_3 is a relatively fine tungsten wire attached by means of a clamp to the lower end of L_2 , the axes of the wires being in the same straight line; L_3 was never heated and was used to measure the torque applied to L_1 and L_2 . The top end of L_1 was clamped securely in the end of the vertical round brass rod to which were attached the soft iron cross-bar Fe and the mirror M_1 . An electro-magnet acting through the glass tube on the iron cross-bar made it possible to rotate the rod and therefore to twist the series of wires.

The lower end of L_3 was held securely in a clamp to which were attached the mirror M_4 and the weight W ; the clamp had a rectangular cross section and could thus slide up and down, as the wires changed in length due to thermal changes, but could not turn. The weight of the clamp and W (170g) served to hold the wires taut.

To the clamp which held together L_2 and L_3 was attached the mirror M_3 and an inverted U of iron wire which dipped into the mercury in the annular iron mercury-cup Hg . The mercury-cup and the parts of the brass frame were so insulated that the heating current flowed in at P_1 , down R_1 to Hg , up L_2 and L_1 , and out at P_2 . L_1 and L_2 were one continuous piece of tungsten wire separated into two segments by a brass clamp to which was attached the mirror M_2 .

² Streintz, Pogg. Ann. 150, 368-380, 1873

³ Meissner, Ann. der Phys. Beibl. 34, 756, 1910

⁴ Dodge, Phys. Rev. 11, 311, 1918

Each mirror was made by depositing a film of platinum on a cover-glass by an evaporation method, covering the film side with another cover-glass, and mounting the two in a light copper frame. Silvered mirrors put together in the same manner were tried first, but the mercury vapor soon caused them to lose their reflecting power almost completely, while the platinized mirrors seemed to be in perfect condition after several months of exposure to mercury vapor. The asbestos tape which was wound around the three cross-heads to make the frame fit the glass tube snugly, had previously been heated to 350°C in a tube attached to a vacuum pump, in order to get rid of the organic matter which would otherwise discolor the glass tube of the final apparatus and thus make optical temperature measurements inaccurate.

After the frame had been placed in the tube and the mirrors were in proper adjustment, annealed coiled copper leads were pulled out of side-arms and made fast in the binding posts P_1 and P_2 . The top of the large tube was then closed by sealing on a cap to which was attached a pump connection. A side-tube of small diameter was sealed into the large tube so that mercury could be let into the mercury-cup through it. This apparatus was placed in a long electrically heated oven from which protruded the pump connection and the small side-tube to which was attached a bulb containing sufficient clean air-free mercury. A drying-tube containing phosphorus pentoxide was placed in the pump-line. The vacuum pump was operated for about eight hours during which time the oven was maintained at a temperature slightly above 325°C . After a side discharge-tube had indicated an x-ray vacuum for three hours, the apparatus was sealed off. As soon as the apparatus was cool enough to be handled, it was removed from the oven, set in a vertical position and the mercury cup filled. The side-tube was sealed off at once thus leaving the wires in a sealed glass tube.

The scales on which the angles of twist of the mirrors M_1 , and M_2 and M_3 were observed, were complete circles 360 cm in circumference, while the scale for M_4 was a short arc having the same radius. For each mirror was provided an illuminated wire and a lens for focusing its image

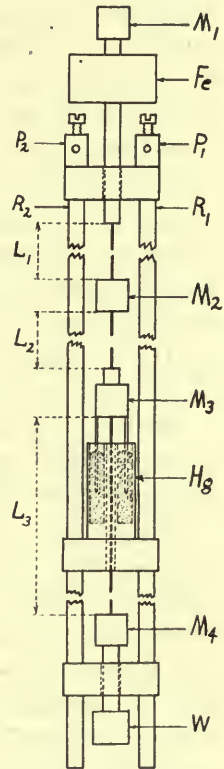


Fig. 1. Torsion system for determining the rigidity.

on its scale. Thus for each degree of twist of a mirror the image of the wire moved along the scale 2 cm.

The method of measuring the diameters of the wires has already been reported.⁵ L_1 , L_2 was a piece of 10-mil drawn tungsten wire of lot B24-3822 of the Nela Research Laboratory; L_3 was a piece of 6-mil tungsten wire of the same lot. Their diameters were respectively $252.3 \pm 0.3\mu$ and $156.72 \pm 0.16\mu$ and their lengths 11.91 cm, 26.62 cm and 25.03 cm at room temperature. The heating current for the 10-mil wire was furnished by large lead storage cells and was regulated by varying the number of cells and by three variable rheostats in parallel; it was measured by means of a 0.01 ohm standard resistance in conjunction with a Leeds and Northrup potentiometer. The temperatures of the wires were measured with a Holborn-Kurlbaum type optical pyrometer; corrections were made for the absorption of the enclosing glass tube. The pyrometer used and the methods employed have already been described.⁶

The length of L_1 was such that its mid-point was at the same temperature as the mid-point of the longer section L_2 when the same heating current passed through both. Another piece of 10-mil tungsten wire 17.6 cm long, mounted in the same frame, was heated by constant currents so that its mid-point assumed the following temperatures: 1065, 1200, 1338 and 1492°K. For each mid-point temperature the temperatures of various points along the wire were obtained. The curves in Fig. 2 showing the temperature as a function of the distance along the wire measured from the top clamp, gave the information necessary to determine the minimum length for L_1 .

The 10-mil tungsten wire was next heated to about 1600°K for just a few seconds in order to get rid of most of the graphite coating which always remains on a tungsten wire when its drawing is completed. Then a constant current of 4.200 amperes was passed through the wire and the brightness temperatures of the mid-points of L_1 and L_2 were found to be 1123 and 1119°K respectively. The lower section of the wire had been polished somewhat before mounting it, and consequently its graphite coating was more completely removed by the first heating; this may help to account for the observed 4° difference. At any rate the change in the simple rigidity caused by a 4° change in temperature could not have been detected since it was less than the experimental error.

The lengths of all the wires were measured and checked several times during the process of making the rigidity determinations; the measurements were always made at room temperature. The length of $(L_2 - L_1)$ at

⁵ Schriever, Proc. Iowa Acad. Sci. **24**, 235-240, 1917

⁶ Schriever, Proc. Iowa Acad. Sci. **28**, 69-82, 1921

a higher temperature was obtained by adding the expansion caused by the heating, the expansion being calculated by using the first term of Worthing's expansion formula.⁷ Allowance was also made for the expansion of the diameter of the wire. The effects of the two expansions tended to balance in the final calculations, and if they had been neglected the error would have been small.

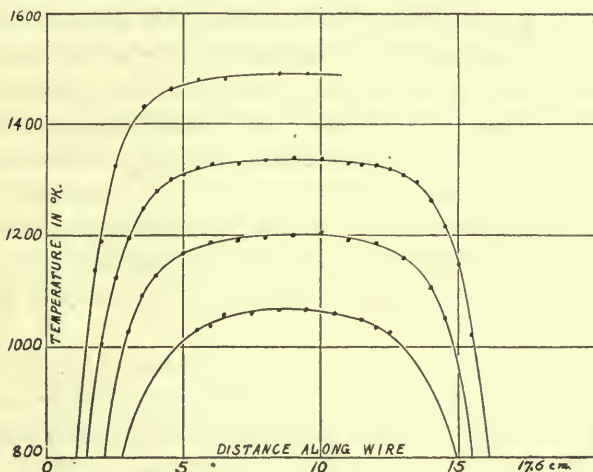


Fig. 2. Temperature distributions along a ten-mil heated tungsten filament.

After the wires L_1 and L_2 had been heated to a definite known temperature, the iron cross-bar, Fe in Fig. 1, was twisted through a small angle and the positions of the images reflected from the four mirrors on the four circular scales were recorded. The cross-bar was then twisted still further in the same direction and the final positions of the images were recorded. The cross-bar was then twisted back to the neutral position and the whole operation was repeated. From six to eight such sets of observations were made at each temperature. The temperature was kept constant by taking pyrometer readings at the beginning, after the third set of observations, and after the sixth set of observations, and making slight adjustments in the heating-current rheostats when necessary. At the higher temperatures the temperature was checked, and adjusted if necessary, after each set of observations. The temperature determinations were certainly correct to within 5° .

The temperature distribution along the wire at the top of L_1 was very approximately like that at the bottom of L_2 , and that of the bottom end

⁷ Worthing, Phys. Rev. 10, 638, 1917

of L_1 like that at the top end of L_2 . The twists in L_1 and L_2 may each be considered to have been made up of two twists, first, a twist in the end-sections in which there were temperature gradients, and second, a twist in the center section all of which was at a uniform temperature. Therefore the difference of the total twists in L_1 and L_2 gave the twist in a section of the wire of length $(L_2 - L_1)$, the temperature of all of which was that of the mid-point of either wire. Let $\alpha, \beta, \gamma,$ and δ be the angles turned through by the images reflected from M_1, M_2, M_3 and M_4 respectively. Then $(\alpha - \beta) = \varphi_1$ was twice the twist in L_1 ; $(\beta - \gamma) = \varphi_2$ was twice the twist in L_2 ; and $(\gamma - \delta) = \varphi_3$ was twice the twist in L_3 . Therefore $(\varphi_2 - \varphi_1)$ was twice the twist in a uniformly heated section of wire of length $(L_2 - L_1)$; φ_3 was twice the twist in a length L_3 of the cold wire. $\varphi_3/(\varphi_2 - \varphi_1)$ obviously should be a constant for twists which do not exceed the elastic limit. The average value of this ratio for each six to eight sets of observations taken at a constant temperature was determined and its probable error calculated. $(\varphi_2 - \varphi_1)$ was kept small; it was always of the order of 0.1 degrees per centimeter.

The mirrors M_2, M_3 and M_4 were displaced from the axis of rotation 0.27, 0.38 and 0.23 cm respectively. Therefore the observed angles of deflection were not twice the angles through which the mirrors were turned. Corrections for M_2 and M_3 were calculated and applied in determining the correct values of β and γ for each set of observations. The deflections of M_4 never exceeded one degree and in most cases were too small to be observed.

The torque required to twist a wire of radius R and length L through an angle θ is

$$T = \frac{1}{2} \pi \eta \theta R^4 / L$$

where η is the modulus of simple rigidity of the wire. In the apparatus employed in this work the same torque acted on the cold and hot wires. Therefore we have

$$\eta_h = \frac{\theta_c}{\theta_h} \left(\frac{R_c}{R_h} \right)^4 \frac{L_h}{L_c} \eta_c$$

where the subscripts h and c refer to the hot and cold wires respectively. L_h/R_h^4 was calculated for each temperature, due allowance being made for the expansion as explained above. $R_c^4 \eta_c / L_c$, a constant for the apparatus, was found to be 215.9 ± 0.8 .* θ_c / θ_h is obviously the average $\varphi_3/(\varphi_2 - \varphi_1)$, the determination of which has already been explained.

Moduli of rigidity, as given in tables of physical constants, have, in general, been obtained from angles of twist which were not great enough to

* The value of $\eta_c = 14.34 \pm 0.04 \times 10^{11}$ dynes per cm^2 was obtained from Dr. L. P. Sieg; he used a static method on a piece of the 6-mil wire taken from the same spool.

cause the elastic limit of the material to be passed. In this work on tungsten the angles of twist were less than 0.1 degree per centimeter and yet, at the higher temperatures, this was considerably beyond the elastic limit. This was shown by the fact that, after the final twist was given, the mirrors M_2 and M_3 turned slowly back toward the original positions. Twists which would not strain the wire beyond the elastic limit at the highest temperatures, could not be measured accurately enough with this apparatus. In order to obtain consistent and fairly accurate results the following method of taking scale readings was used. Two card sliders to indicate the position of the beam of light reflected from a mirror, were placed on each circular scale. Four of the sliders were set at the

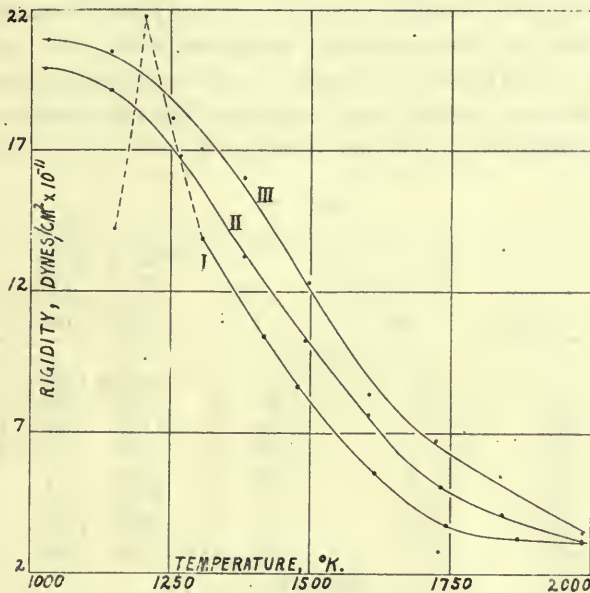


Fig. 3. Rigidity as a function of temperature for three successive runs.

initial positions of the reflected beams on the four scales. The final twist was given and the other four sliders were adjusted in the following order: first for M_3 , second for M_2 , third for M_4 , and last for M_1 . The wire was then twisted back to the initial position and the positions of the sliders were recorded. M_1 and M_4 did not change their positions, and M_2 twisted back only very slowly relative to the rate of M_3 ; this will explain the order of making the slider adjustments. The second slider for M_3 was in position in about two seconds after the final twist was given. The variations in the value of $\varphi_3/(\varphi_2 - \varphi_1)$ for a given set at the higher temperatures, are probably largely due to the inaccuracy of the settings of the second slider for M_3 .

The wire was heated for about an hour at each of the nine different temperatures of the first series, and about three quarters of an hour at each temperature of the second series. The last series was taken more rapidly; each temperature was maintained for about thirty-five minutes.

The probable error of the modulus of rigidity for each temperature was calculated from the probable error of the mean of $\varphi_3/(\varphi_2 - \varphi_1)$, that of the constant of the cold wire, and that of the radius of the hot wire. The probable errors of the length-measurements were neglected since they were relatively small compared to those of the fourth powers of the radii.

RESULTS

Curves showing the modulus of rigidity of the 10-mil drawn tungsten wire as a function of the temperature, are shown in Fig. 3. The data for each curve were obtained at a series of increasing temperatures; the curves were obtained in the order indicated by the numbers. A recapitulation of the results is shown in Table I.

TABLE I

Set No.	Length of hot section (cm)	R_h^4 (10^{11}cm^4)	Ω $= 215.9L/R^4$ (10^{-10})	Γ $= \frac{\varphi_3}{\varphi_2 - \varphi_1}$	Brightness temperature	True temperature	$\eta_h = \Omega T$ ($10^{-11}\text{dynes/cm}^2$)
<i>Series I</i>							
1	14.73	2576 ± 16	$12.33 \pm .10$	$11.55 \pm .09$	1094°K	1150°K	$14.23 \pm .16$
2	.74	2576	.33	$17.72 .43$	1144	1205	$21.80 .52$
3	.75	2585	.33	$11.28 .20$	1238	1308	$13.88 .25$
4	.76	2585	.32	$8.46 .13$	1336	1418	$10.41 .16$
5	.77	2592	.32	$7.04 .12$	1389	1479	$8.66 .15$
6	.78	2592	.32	$4.53 .23$	1506	1615	$5.57 .27$
7	.79	2602	.29	$3.01 .15$	1618	1743	$3.70 .18$
8	.80	2608	.25	$2.62 .13$	1727	1870	$3.22 .16$
9	.81	2615	.21	$2.54 .06$	1823	1985	$3.10 .08$
<i>Series II</i>							
10	14.75	2569 ± 16	$12.41 \pm .10$	$16.08 \pm .06$	985	1025	$19.92 \pm .16$
11	.76	2576	.36	$15.46 .25$	1089	1143	$19.15 .32$
12	.76	2576	.36	$13.57 .08$	1191	1268	$16.81 .16$
13	.77	2585	.31	$10.73 .09$	1301	1380	$13.21 .14$
14	.78	2592	.32	$8.35 .12$	1401	1492	$10.27 .16$
15	.79	2592	.33	$6.19 .03$	1499	1605	$7.61 .07$
16	.80	2602	.30	$4.15 .04$	1610	1734	$5.10 .06$
17	.82	2608	.28	$3.30 .02$	1704	1843	$4.06 .03$
18	.86	2615	.28	$2.58 .05$	1823	1985	$3.17 .06$
<i>Series III</i>							
19	14.75	2538 ± 16	$12.54 \pm .10$	$17.39 \pm .14$	—	296	$21.73 \pm .22$
20	.80	2569	.45	$16.84 .11$	988	1030	$20.98 .21$
21	.81	2576	.40	$16.55 .18$	1089	1143	$20.51 .27$
22	.82	2576	.40	$14.66 .14$	1187	1252	$18.17 .22$
23	.83	2585	.38	$12.94 .20$	1301	1380	$16.05 .27$
24	.84	2592	.38	$9.94 .11$	1405	1497	$12.31 .16$
25	.85	2592	.38	$6.72 .18$	1499	1605	$8.34 .22$
26	.87	2602	.36	$5.40 .15$	1603	1726	$6.70 .18$
27	.89	2608	.31	$4.45 .11$	1702	1841	$5.48 .13$
28	.91	2615	.30	$2.78 .07$	1823	1985	$3.42 .09$

Some qualitative determinations of the simple rigidity of a 10-mil drawn tungsten wire 30.5 cm long were obtained previously.⁸ No corrections were made for the end-effects but, since each temperature was calculated from the total amount of expansion, the error in the rigidity calculation tended to balance the error in the temperature calculation in such a way that the curves are more nearly correct than one might at first thought expect. The curves which were obtained are shown in Fig. 4. The data for Curve I was obtained by starting at room temperature; at the higher temperatures the elastic limit was passed so easily that observations became very inaccurate. The wire was then heated to about 2000° K for 100 minutes, allowed to cool to room temperature, and the observations for Curve II were started. Observations for Curve III were started at the highest temperature used for Curve II and completed at room temperature. A shorter length of the wire taken from the same spool gave curves having the same general shapes as those of Fig. 4, the irregularities occurring at the same temperatures.

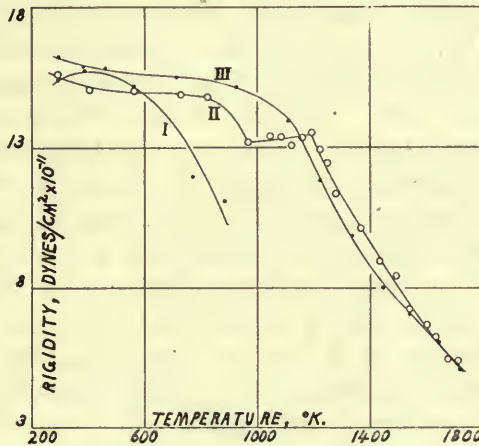


Fig. 4. Results of some preliminary measurements.

Jeffries' work⁹ on the change of crystal structure of tungsten wire by heat treatments at various temperatures makes it possible to interpret the shapes of the Curves in Figs. 3 and 4. Dr. L. P. Sieg found the value 13.99×10^{11} dynes/cm² for the rigidity of the wire at room temperature; the wire had been given no heat-treatment other than that which it received when it was drawn. In the present research the temperature at which the apparatus was baked out never exceeded 330°C, and the very short heating at 1600°K (to remove the graphite film) certainly changed the structure very little. The first rigidity determination was

⁸ Schriever, Master's Thesis in Uni. of Iowa Library

⁹ Jeffries, Trans. Am. Inst. Mining Eng. 1037-1092, 1918

made at 1150°K (Curve I of Fig. 3); the value was $14.2 \pm 0.3 \times 10^{11}$ dynes cm^2 which, within the experimental error, is the same as found by Dr. Sieg at room temperature. The structural changes caused by the heating at 1600°K and especially by the heat treatment at 1150°K during the process of making the first rigidity determination, will account for the constancy of the rigidity from room temperature to 1150°K for the first heating. If observations starting at room temperature had been taken, a rapid falling off of the rigidity would have been observed before a temperature of about 1100°K was reached, as was shown by the wire giving Curve I of Fig. 4.

Heat treatment at 1150 to 1300°K will cause the structure of a new wire to change rapidly. The wire (Curve I of Fig. 3) was held at 1150°K for slightly more than two hours, at 1250° for an hour, and at 1308° for about an hour. Although these temperatures are below the five-minute equiaxing or recrystallization temperature, namely 1600°K, the long exposures at these temperatures probably allowed crystallization to take place. This evidently was the case because the rigidity at 1308°K was only a little less than it was at 1150°K. From 1300 to 1700°K the curve is almost linear which indicates that no radical change in the structure took place. Above 1700°, the region of rapid grain growth, the slope of the curve decreases rapidly. Unless the crystal grains enlarged a great deal it would be difficult to explain the relatively small decrease in rigidity between 1740 and 1985°K for the first heating of the wire to such high temperatures. The fact that large crystal grains were formed would also account for the high value of the rigidity at 1025°K (first point of Curve II, Fig. 3) and again at 1030°K in the third series of measurements. Since the change of slope near the lower end of each curve is less rapid in each succeeding curve, it seems probable that, after the grain-growth is completed, the rigidity will decrease regularly with temperature-increase, and that the curves obtained thereafter will not show a decrease of slope at the higher temperatures.

If the scale readings for the final positions of M_3 at the higher temperatures had been made less rapidly, the deflections for this mirror would have been smaller, and smaller values for the modulus of rigidity would have resulted. Consequently the change of slope near the lower end of each curve cannot be due to the yielding of the wire. Thus this decreased rate of falling off of the rigidity with temperature-increase must be real and, in all probability, due to the rapid grain-growth at the higher temperatures.

It is known that the modulus of rigidity when calculated from small twists, is greater than when calculated from large twists. This effect

was observed in this research, and for this reason the twist per centimeter was kept approximately constant.

It is observed that Curve III of Fig. 4 lies below Curve II for temperatures above 1175°K. This peculiar result was probably due to the wire having formed a crystal structure which made an exceptionally rigid wire but one which was not stable at the highest temperature. The second point for Curve I of Fig. 3 is probably incorrectly located although the data do not indicate this for they are consistent among themselves.

Since in small tungsten wires a single crystal grain may occupy a very large part of the cross-section of the wire,⁹ the modulus of rigidity determined from such a wire may be quite different from that obtained from observations made on a relatively large wire. An increase of rigidity with decrease in size of wire has been observed in the case of freshly drawn tungsten wires.¹⁰ The results given in this article may therefore be different from those which a bar of the metal would yield even if the bar had the same grain-size as the wire. Wires smaller than the one used would undoubtedly give still different values for the modulus of rigidity. The determination of the change of the rigidity with the size of the wire would make very interesting work but the labor involved would be very great.

In conclusion I wish to express my appreciation to Professor L. P. Sieg for his many valuable suggestions and keen interest in this work; and to the Director of the Nela Research Laboratory who, through Dr. A. G. Worthing, so kindly furnished the tungsten and sealing-in wires, the calibrated standard amp and the pyrometer lamp. I am also indebted to J. B. Dempster and M. Teeuwen for much valuable information regarding workshop methods, and to J. C. Steinberg who generously helped me prepare the platinum-coated mirrors with his evaporation apparatus.

PHYSICAL LABORATORY,
STATE UNIVERSITY OF IOWA,
June 6, 1923.¹¹

¹⁰ Sieg, Proc. Iowa Acad. Sci. **24**, 207, 1917

¹¹ This work was completed in July, 1921. Present address is University of Oklahoma, Norman, Oklahoma.

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