An Electron Microscope for Filaments: Emission and Adsorption by Tungsten Single Crystals[1]

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A new and simple electron microscope has been designed for small cylindrical filaments. Die marks are found to be prominent in the electron images of all drawn wires. The activation of thoriated tungsten has been studied. Electron emission from single crystals grown in drawn pure tungsten filaments varies around the wire circumference and depends systematically on crystallographic direction. This variation of emission with crystallographic direction is more pronounced and complex when the filament is allowed to self-activate in cesium or potassium vapor.

THE MICROSCOPE

THE usual methods of electron optics[2] cannot be applied directly to study the emission from small round filaments, because of the conflicting symmetries of lens system and object. If only peripheral magnification is required, no electron lens is needed. The wire is mounted in the axis of a glass tube (Fig. 1) with fluorescent material on its inside wall,[3] and the emitted electrons are drawn over to the wall so rapidly that their initial nonradial velocity components have not time to take them far from a strictly radial path. On the wall is formed an electron image of the wire, magnified in circumference by the ratio b/a of tube radius to wire radius, not magnified in length. We estimate the resolving power by considering two electrons, one leaving the filament along a certain radius, the other with an initial velocity component $(2kT/m)^{\frac{1}{2}}$ normal to this radius and to the wire axis. The angular separation between the end-points of the paths is

$$\theta=2\left(\frac{300kT}{\epsilon V}\right)^{\frac{1}{2}}\left(\ln\frac{b}{a}\right)^{\frac{1}{2}}\int_0^{(\ln b/a)^{\frac{1}{2}}} e^{-u^2}du;$$

for operating conditions, a 5-mil wire at 2000°K in a 2-in. tube, with 10,000 volts on the screen, this is about 1°. A similar computation for the initial velocity along the axis shows that points about 0.2 mm apart on the wire length can be separated. These estimates assume that the tube wall is an equipotential. The voltage V is applied to a helix of nickel wire wound to fit the inside wall snugly. Slow secondary electrons are emitted by the screen between helix turns and drift to the turns under a weak field, keeping the fluorescent

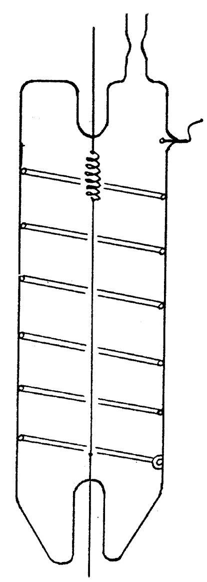


FIG. 1. Sketch of a typical tube.

[1] A report of this work was given at a meeting of the New England Section of the American Physical Society; abstracts in Phys. Rev. **48**, 973 (1935).

[2] Brüche and Scherzer, *Geometrische Elektronenoptik* (Berlin, 1934).

[3] After cleaning, the inside of the tube is coated with a ten percent solution of potassium silicate and the excess liquid is shaken off. This coat is allowed to dry for about two minutes, and willemite powder is blown on with a simple compressed-air dust gun, made of a glass bottle reservoir and a two-holed rubber stopper. Another method, which avoids the silicate binder, is to grind the inside with 120-mesh alundum, using a brass disk with about $\frac{1}{4}''$ clearance soldered on a long spindle which is turned in a lathe, and rub the willemite in dry. This gives a more uniform screen, but the difficulty of background radiation from the filament is more serious.

surfaces from charging up. Tests show that distortion of the image by potential gradients in the screen is negligible.

Observation is possible over the temperature range where enough electrons are emitted to excite the screen and not enough to damage it, from ~1700°K to ~2400°K for clean pure tungsten.

Cold Emission and Surface Contaminations

If, after evacuation and before any heat treatment of the filament, the anode potential is applied while the filament is at room temperature, the screen is covered with bright splotches (Fig. 2). On raising the filament temperature these squirm about, disappear and reappear, and usually vanish permanently when the filament is dull red. We attribute these splotches to field emission from rough regions on the wire which are covered with alkali impurities during drawing and handling. It has been suggested that these places of high electron emission from cold wires with small fields may be significant in Geiger-Müller counter operation: some tests are in progress. At about dull red, narrow bright rings often appear around the tube and persist to higher temperatures. A flash above 2500°K is always sufficient to remove them permanently from pure tungsten. Two platinum samples were raised to the melting point without cleaning off these rings. It seems certain that severe heat treatment is necessary before field currents or thermionic currents characteristic of a metal can be obtained.

Stripes and Die Marks

The emission pattern of a clean drawn tungsten wire (Fig. 3) changes only in intensity over the observation range. The light and dark stripes, regularly spaced around the tube and continuous down its length, have appeared on the pattern of every drawn tungsten wire we have examined—more than a dozen samples, differing in size, manufacture, and purity. The stripes can be correlated with grooves, presumably die marks, on the wire, visible under an optical microscope. Neither grooves nor stripes are removed by hours

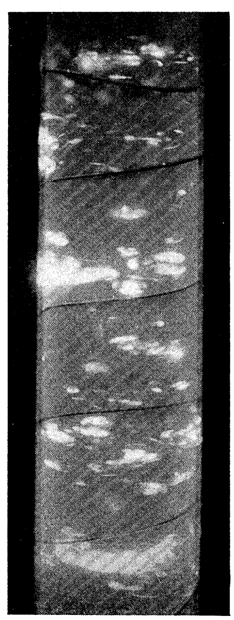


Fig. 2. Cold emission from a raw tungsten wire.

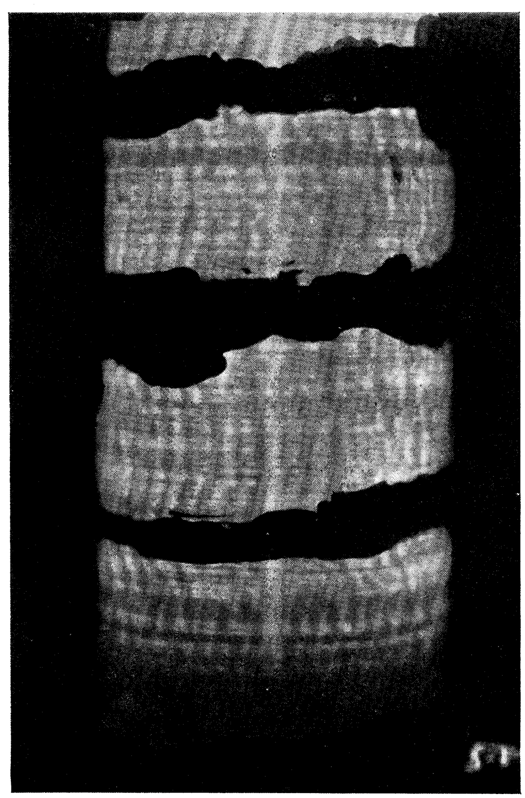


Fig. 3. Emission pattern of a drawn tungsten wire. In this tube the nickel helix was replaced by one of platinum paint.

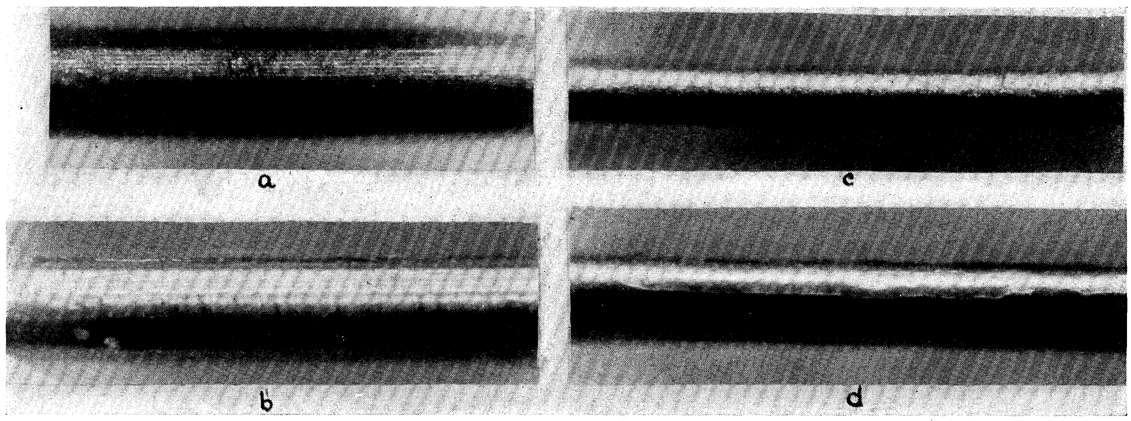


FIG. 4. Microphotographs of tungsten filament surfaces. (a) Raw wire from spool; (b) piece of same wire after about one hour at 2800°K; (c) wire smoothed by electrolysis; (d) piece of same wire after about one hour at 2800°K.

of heating above 2800°K (Fig. 4). Wires smoothed free of grooves by electrolysis or abrasion[4] give an electron image free of stripes. The pattern and optical appearance of tantalum are similar to those of tungsten; platinum and nickel (oxide-coated) show less prominent stripes and grooves.

THORIATED TUNGSTEN[5]

Immediately after a flash to 2800°K the pattern of a thoriated tungsten filament at 1200°K consists of intensely bright spots (Fig. 5). Many of these reappear in the same places after a subsequent flash to 2800°K, no matter what the intervening thermal history of the wire. During activation at 1850°K the thorium appears to migrate from these spot-sources and eventually to cover the surface almost completely. At 1200°K the activated filament shows very brilliantly the same pattern, with stripes due to grooves, as at 2000°K without a thorium coat.

CESIUM ON TUNGSTEN SINGLE CRYSTALS

When the tube contains cesium vapor in equilibrium with cesium metal at about room temperature, there is a lower observation range ($\sim$700°K to $\sim$900°K) where the work function is greatly decreased by adsorbed cesium.[6] Above 900°K, where the equilibrium coverage of cesium is very small, the screen is dark until we reach the usual clean-tungsten range, 1700°K to 2400°K. Using smoothed drawn wires of pure tungsten (G. E. 218) containing large single crystals which in growth have followed the fiber grains in having a 110 axis parallel to the wire axis,[7] we find that the emission pattern on the tube, around

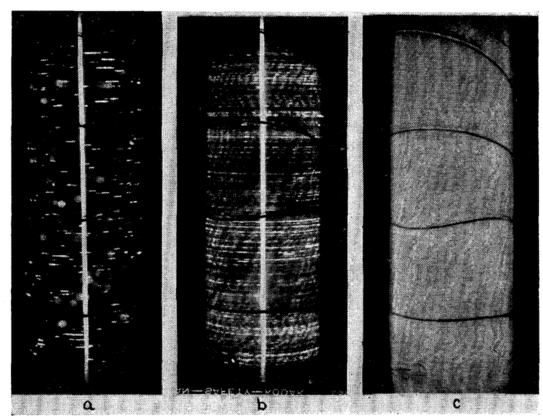


FIG. 5. Typical patterns of thoriated tungsten (1200°K). (a) immediately after flash to 2800°K; (b) after first 4 minutes of activation at 1850°K; (c) after 40 minutes of activation at 1800°K.

[4] Rapid electrolysis in NaOH or KCN will give a smooth, glassy surface, but the rate requires careful control, and it is impossible to keep the diameter uniform. A grinding engine has been constructed and is now in use, which polishes wires up to 40 cm long, leaving them uniform in cross section, round, and microscopically smooth.

[5] For a review of this subject, see I. Langmuir, J. Frank. Inst. **217**, 543 (1934).

[6] J. B. Taylor and I. Langmuir, Phys. Rev. **44**, 423 (1933).

[7] M. Ettisch, M. Polanyi and K. Weissenberg, Zeits. f. physik. Chemie **99**, 332 (1921).

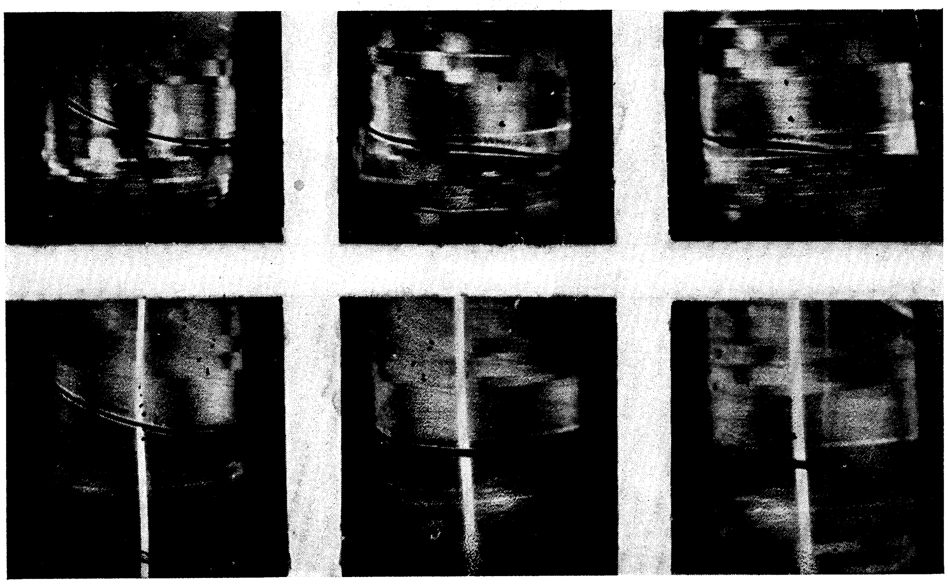


FIG. 6. Emission patterns of a smoothed tungsten wire containing several large single crystals, in equilibrium with saturated cesium vapor at room temperature. Upper row at ~825°K (intermediate between *C* and *D*, Fig. 7), lower row at 2000°K. Left column, looking along 100 axis of large crystal; right column, looking nearly along 110 axis of large crystal; center column, intermediate direction. Black dots are ink spots on the tube for reference.

each single crystal and over both observation ranges, is divided into four quadrants, the pattern in any quadrant being a mirror image of the patterns in the adjacent quadrants (Figs. 6, 7). This is just the symmetry of the tungsten lattice about the 110 axis, with the quadrants lying between another 110 and a 100 axis, both perpendicular to the wire axis. X-ray Laue pictures allowed us to identify the axes marked on the diagrams. As the temperature is lowered from the hot tungsten range, the first regions to become bright (due to adsorbed cesium) are those which were darkest on the hot tungsten pattern. At still lower temperatures the patterns become more complicated, and "fine structure" develops, but in general regions of high emission from hot tungsten are regions of low emission, indicating low cesium coverage, from the Cs–W surface, and *vice versa.*

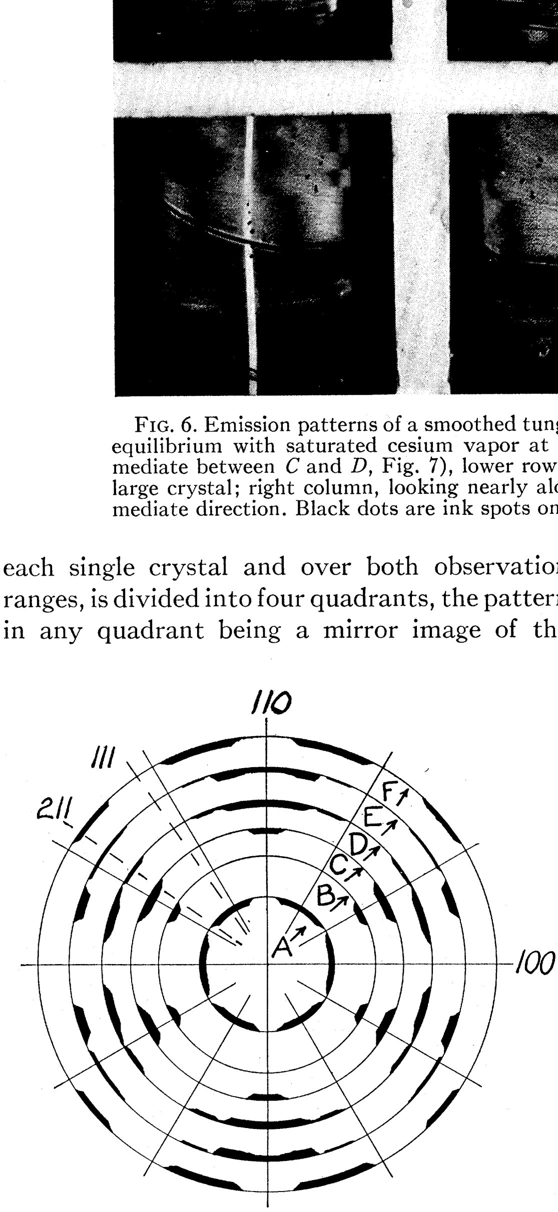


FIG. 7. Diagrams of patterns for wire of Fig. 6: dependence of brightness on direction, for a given filament temperature, is shown by thickness of line. Brightness of brightest parts of Cs-W patterns increases toward higher temperature. Wire axis, 110, is perpendicular to plane of diagram; prominent lattice directions in the plane are indicated. *A*~2000°K; *B*~900°K; *C*~850°K; *D*~800°K; *E*~750°K; *F*~700°K.

POTASSIUM ON TUNGSTEN

With the single-crystal filament in a potassium atmosphere symmetrical low temperature patterns were again found, but much fainter than with cesium. If the cold filament, surrounded by potassium vapor, is given a "bombing" treatment[8] for one minute, and then is raised for one

[8] Alkali vapor in a tube attacks the screen and diminishes the fluorescent yield. After this decomposition has begun, setting the anode voltage above 6000 v produces rapidly changing splotches and rings on the tube wall accompanied

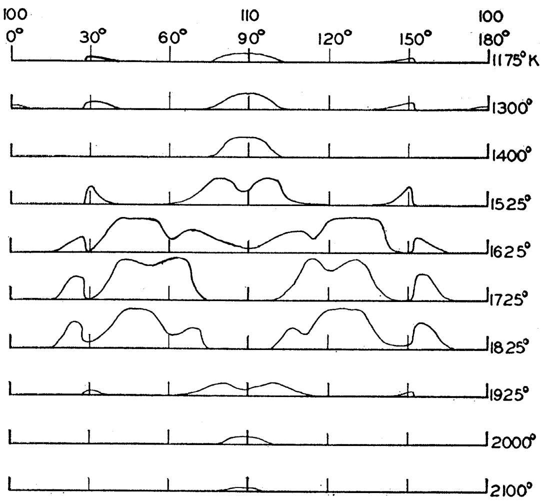


FIG. 8. Influence of preparation temperature on single-crystal patterns at ~775°K. Filament in equilibrium with saturated potassium vapor at 220°C; preparation temperatures at right. The curves, sketched during observation, show dependence of emission on angle around a semi-circumference of the tube with the 100 axis as diameter.

minute to a "preparation temperature" between 1200°K and 2100°K, the low temperature equilibrium patterns depend markedly in intensity, structure, and persistence on the preparation temperature (Fig. 8). The following hypothesis explains qualitatively all the observations: bombing coats the filament with a thick layer of oxygen,[9] which at a preparation temperature evaporates from the filament nonuniformly, the rate depending on crystallographic direction. When the partially oxygenated filament is then allowed to coat with potassium, the potassium coverage depends on lattice direction and oxygen coverage, and the electron emission depends on all three.

RECRYSTALLIZATION

We have used this electron microscope, with cesium, to observe grain growth in a smoothed pure tungsten wire (G. E. 218). The Cs-W pattern of the raw wire was uniform, indicating a microcrystalline surface structure below the resolving power of the instrument. A flash to 3100°K produced several single crystals long compared with the wire diameter, and the growth of these during subsequent flashes could be readily followed on the Cs–W patterns at ~800°K, well below the recrystallization temperature (Fig. 9).

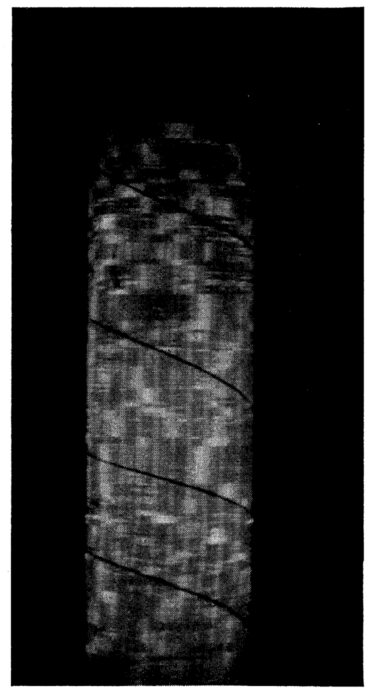


FIG. 9. Cs-W pattern at ~800°K, showing the degree of recrystallization produced by several flashes to 3100°K. (G. E. 218 wire, smoothed by abrasion.)

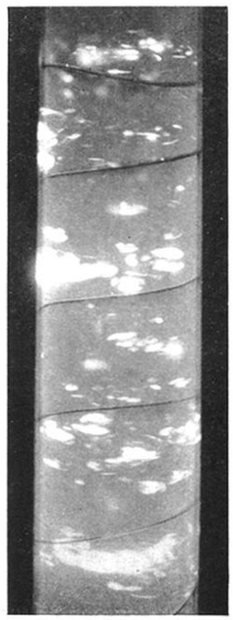
In recrystallized drawn tungsten wires long crystals are unusual, and the orientation is presumably influenced by the drawing. Preliminary investigations with single-crystal Pintsch filaments,[10] where the crystal orientation is random with respect to the wire axis, encourage the hope of obtaining an emission and adsorption map for all the lattice directions in tungsten.

We are greatly indebted to Professors W. B. Nottingham and B. E. Warren, to Dr. Irving Langmuir, and to Mr. W. E. Hazen for assistance and advice.

by red sparkles at the wire, as if the wire were being struck by particles liberated from the wall through electron impact. (Stable patterns can be maintained for anode voltages below 4000 v.) After this bombing treatment the pattern below 1000°K consists of irregular bright splotches and rings, like that of a raw wire.

[9] Oxygen is assumed, since the preparation temperature range resulting in high emission is almost the same as that observed by K. H. Kingdon (Phys. Rev. **24**, 510 (1924)) to result in high emission from a Cs-O-W surface.

[10] C. J. Smithells, *Tungsten: Metallurgy, Properties and Applications* (New York, 1927), in particular, p. 68.



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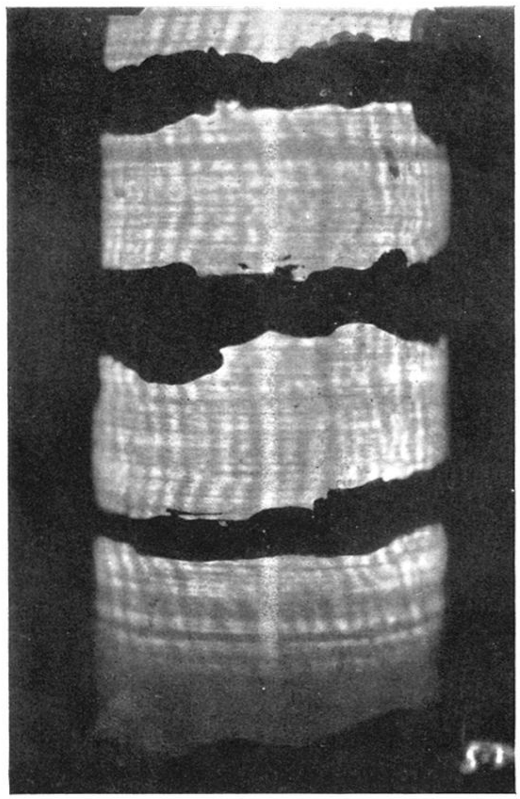

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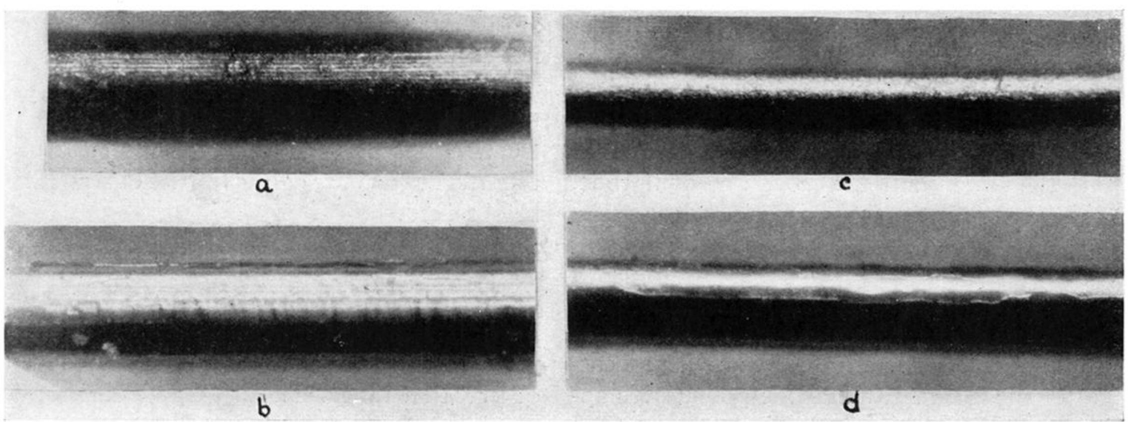

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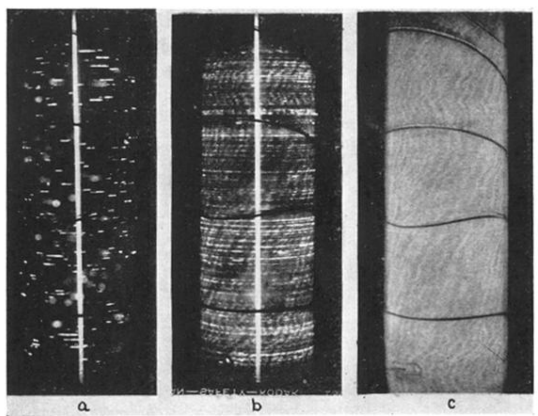

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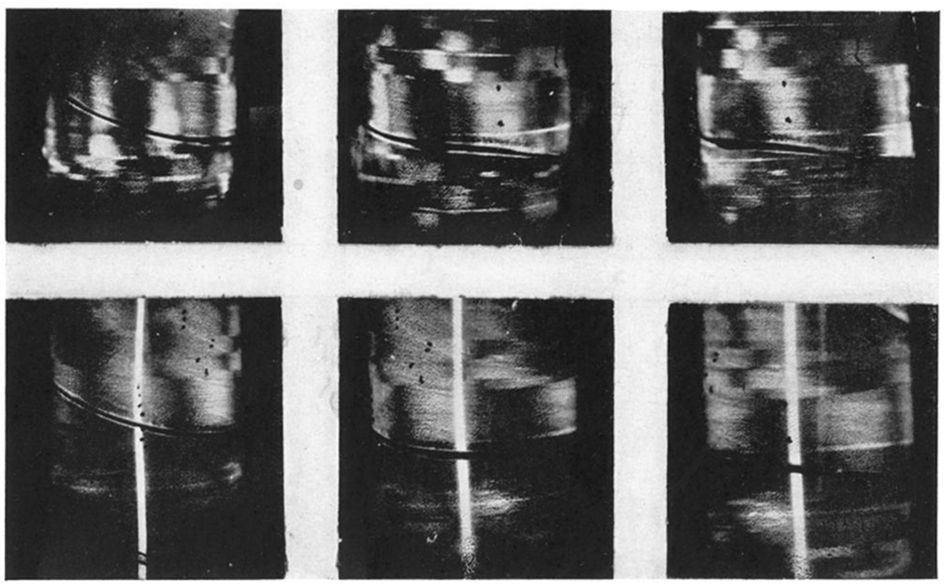

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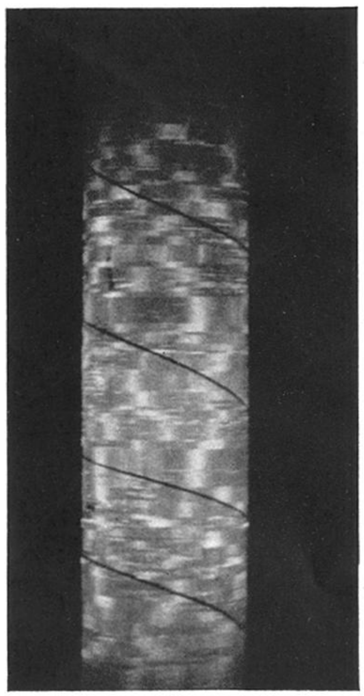

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