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THE EVAPORATION PROCESS AND ITS APPLICATION TO THE ALUMINIZING OF LARGE TELESCOPE MIRRORS

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ABSTRACT

A history of the evaporation process is given. This includes the contributions by Ritschl and others and describes the present technique in detail.

Formulae are developed which give the thickness of the film produced by a circular array of evaporation sources. Applications for the cases of 40-inch and the 108-inch tanks are discussed. Reference is also made to the application of non-uniform films in the figuring of mirrors. Methods of cleaning mirrors preparatory to coating are discussed. The technique of obtaining high vacuum in large tanks is treated.

The reflectivities and other properties are given for evaporated films of aluminum and silver, as well as a *Cr*, *Pt*, *Pd*, *Rh*, *Sn*, *Au*, and *Cu*. This includes observations on aluminized astronomical mirrors now in use for over three years.

The results of a study of the oxidation (corrosion) of aluminum by means of measurements on transmissivity and reflectivity of partial films is reported.

The different sets of equipment that have been developed in increasing size up to the 108-inch tank are described.

The history of the application of the process to astronomical mirrors is given.

INTRODUCTION

A process for coating glass mirrors with a reflecting material is described. It involves the evaporation of the material (usually a metal) in a high vacuum and is therefore called the "evaporation process." The metal is heated until it has a vapor pressure of the order of 1/10 mm of mercury. The vacuum is sufficiently high to give a long free path (greater than 100 inches), so that the evaporated molecules travel without intermolecular collisions from the hot metal source to the cold surface of the mirror. The attainment of

this vacuum during evaporation is evidenced by the sharp shadows cast in the evaporated film by obstacles placed between it and the evaporation source. The remarkable feature of the whole phenomenon is that the film, condensed on the mirror, has the same degree of polish as that possessed by the glass. A mirror freshly coated by this process does not, therefore, require any burnishing.

C. Hawley Cartwright and the author studied the application of this process to many metals, including aluminum, but it was not until the technique for aluminum was developed by the author that films of high quality were obtained which showed its potential value for coating astronomical mirrors.

The application to very large mirrors seemed to offer some difficulty, especially in the attainment of the necessary vacuum. Recent advances in vacuum technique and, in particular, the advent of oil diffusion pumps, have helped to make this application possible.

HISTORY OF THE EVAPORATION PROCESS

Pringsheim and Pohl[1] discovered that several metals, including aluminum, could be evaporated in vacuum and condensed on a glass surface to form a polished reflecting film. They used a magnesia crucible from which to distil the metal. R. Ritschl,[2] in 1928, in making an application of the evaporation method to the preparation of half-silvered interferometer mirrors, heated the metal in a bare tungsten coil. This change in technique has the advantage over the use of a magnesia crucible that the tungsten does not evaporate or out-gas so much in a vacuum.

Since then several physicists have used this technique for making mirrors for experimental use. Those with whom the author is acquainted are A. H. Pfund,[3] Joseph E. Henderson,[4] D. L. Webster,[5] W. W. Nicholas,[6] H. C. Burger and P. H. Van Cittert,[7] P. G. Kruger,

[1] *Verh. d. Deut. Phys. Ges.*, **14**, 506, 1912.

[2] *Tätigkeitsbericht d. Phys. Techn. Reichsanstalt*, 1928; *Zs. f. Phys.*, **69**, 578, 1931.

[3] *Rev. Sc. Instr.*, **1**, 397, 1930.

[4] *Evaporation of Nickel from Tungsten*, Yale University, 1926.

[5] *Proc. Nat. Acad. Sci.*, **14**, 679, 1928.

[6] *Circ. Bur. of Standards*, No. 389.

[7] *Zs. f. Phys.*, **66**, 218, 1930.

Robley C. Williams,[8-9] George B. Sabine,[10] C. Hawley Cartwright,[11-12] and Hiram W. Edwards.[13]

The author began his experiments at the University of Michigan in 1929, starting with a technique described by Professor L. S. Ornstein during his visit there. The experiments, originally started by Professor E. F. Barker and later carried on by the author, included not only the evaporation of metals but also their protection from tarnish by a thin film of evaporated quartz. This distillation of a non-conductor is a possibility of the evaporation process not shared by the sputtering method. Films of quartz, as well as of *AgCl*, were applied by this method to alkali halide prisms, used in infra-red spectroscopy, to prevent them from tarnishing with moisture. Silver chloride was found to give the best protection for these prisms.

Cartwright and Strong,[11] at the California Institute of Technology, in the autumn of 1930 developed a simple apparatus for carrying out the process and made a survey of its applicability to different metals. The usual technique of heating some of the metal to be evaporated in a helix of tungsten wire was found to be successful, except with aluminum and beryllium. They observed that the tungsten coil was dissolved by these metals. A similar failure for aluminum was recorded by W. W. Nicholas in the Bureau of Standards circular, *The Making of Mirrors by the Deposition of Metal on Glass.*

EVAPORATION TECHNIQUE FOR ALUMINUM

Other attempts[12] have since been made to develop the technique of evaporating aluminum because of its high ultra-violet reflectivity. Experiments were carried out here with crucibles of graphite, pure fused magnesia and alumina (sapphire), as well as with sintered and with fused crucibles of thorium oxide. They showed that the heating in a crucible was apparently impractical; either the metal reacted chemically with the material of the crucible or the latter evaporated when the aluminum was heated. The latter crucible material was

[8] *Phys. Rev.*, **41**, 255, 1932.
[9] *Ibid.*, **46**, 146, 1934.
[10] *Ap. J.*, **77**, 316, 1933.
[11] *Rev. Sc. Instr.*, **2**, 189, 1931.
[12] *Ibid.*, **3**, 298, 1932.
[13] *Phys. Rev.*, **43**, 205, 1933. *Rev. Sc. Instr.*, **4**, 449, 1933.

prepared by a method described by W. H. Swanger and F. R. Caldwell.[14]

The bare tungsten-coil method, however, proved to be the most practical after the discovery by the author that tungsten has a limited solubility in molten aluminum.[15] The burning-out of the tungsten coil was avoided by the simple expedient of making it of larger wire and arranging the charge so that the solubility of the molten aluminum for tungsten could be satisfied without dangerously reducing the diameter of the wire. A chemical analysis of the tungsten alloy formed when aluminum is fused on a tungsten coil showed the solubility of tungsten in aluminum to be about 3 per cent by volume.

It might be expected that some of the dissolved tungsten would boil away, especially since its spectrum has been observed in the bell jar during evaporation.[16] In order to test this point a coil was weighed before and after evaporating several charges of aluminum. Instead of a loss in weight, an increase was observed, indicating that some aluminum had diffused into the tungsten. Extended firing at a very high temperature decreased the weight, however, until, within the experimental error, it became the same as in the beginning. A chemical analysis of the condensed metal film gave no definite indication of tungsten. A concentration of 0.03 per cent by weight was judged detectable by Dr. T. F. Anderson, to whom I am indebted for the chemical analysis. He found 0.2 per cent of iron as the chief impurity of the film. The tungsten which is dissolved thus appears to be almost completely precipitated back on the coil as the evaporation proceeds. It may not deposit in exactly the same place, but it does compensate in a large measure for the decrease in diameter of the tungsten wire, especially if the coil is turned over after each charge. Ordinarily one coil lasts for the evaporation of about a dozen charges.

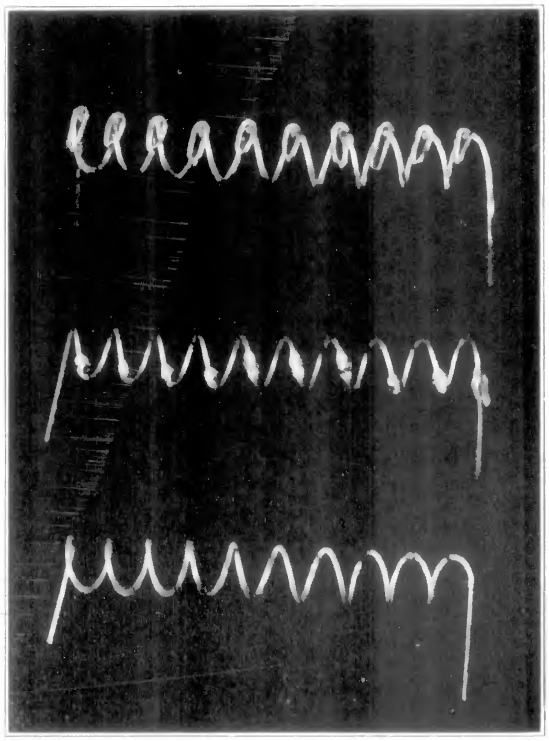
The final arrangement of the coil, which has been used for aluminizing all mirrors at the California Institute of Technology, is shown in Plate XIV. It is in the form of a helix, consisting of ten turns of 30-mil tungsten wire, $\frac{5}{16}$ inch in diameter and pitched four turns to the inch. A U-shaped piece of aluminum wire 1 mm in diameter and

[14] *Bur. of Standards J. of Res.*, **6**, 1131, 1931.

[15] John Strong, *Phys. Rev.*, **43**, 498, 1933.

[16] E. Gaviola and John Strong, *ibid.*, **38**, 136, 1935.

PLATE XIV



ALUMINUM COILS SHOWING METHOD OF CHARGING AND MELTING DOWN THE CHARGE

about 10 mm in total length is clamped to each turn, as is shown by the top coil in the illustration. A potential of 20 volts applied to the coil in vacuum for four seconds fuses these pieces, as is shown by the coil in the middle. At this stage surface tension keeps the molten aluminum from dropping. This fusion also serves to free it from oxide and other impurities. It is customary to make a separate run in order to effect this melting of the aluminum to the tungsten wires in all the tanks except the 40-inch. Here the coils are covered by a baffle during the preliminary firing. The aluminum is distilled from the coils by the same voltage applied to each coil for fifteen seconds, leaving the coil as is shown at the bottom of the illustration.

UNIFORM FILMS

In order to get a uniform coat on the mirror, it was early decided to distil the metal on the large reflector surface from several tungsten sources suitably arranged, rather than from one movable source.

The evaporation of polonium in a high vacuum from a point source has been investigated by Bonet-Maury.[17] He finds that the condensation on a plane surface is proportional to the inverse square of the distance from the source and to the cosine of the angle between the normal to the surface and the line connecting the surface with the source. We have no reason to suspect that such is not the case for other metals which have a low vapor pressure at room temperatures.

Starting with this, we may consider the distribution of the film thickness τ produced by various experimental arrangements. In the case of evaporation to the inside surface of a sphere of radius ρ from a point source of vapor at its center we get a uniform film whose thickness, τ_0, is

$$\tau_0 = \frac{m}{4\pi\delta\rho^2} .$$

Here m is the mass of metal evaporated and δ is its density.

The film thickness on a plane surface at the normal distance ρ from a point source at P is

$$\tau_P = \frac{m}{4\pi\delta r^2} \cos\theta = \tau_0 \left(\frac{\rho}{r}\right)^3 .$$

[17] *Ann. d. Phys.*, 11, 285, 1929.

Here τ_0 is the thickness at distance ρ, r is the distance from the source to P, and θ is the inclination of the surface at P to the molecular rays emitted by the source.

The film thickness produced on a plane surface by a circular array of vapor sources was studied. If there are N coils spaced uniformly around a circle at a distance ρ from the surface to be coated, the film thickness at P, which is at a distance a from the intersection of the axis of the circle with the face of the mirror, is given by the expression

$$\tau_P = \frac{M\rho}{4\pi\delta N} \sum_{i=1}^{N} \frac{1}{r^3} .$$

Here M is the total mass of metal evaporated and r is the distance from P to the respective ith coil.

Dr. Edward M. Thorndike made the same calculation, assuming a continuous circular source. The thickness is given in this case by

$$\tau_P = \frac{M\rho}{8\pi^2\delta} \int_0^{2\pi} \frac{d\theta}{r^3} .$$

This calculation involves the integration[18] of the expression

$$\int_0^{2\pi} \frac{d\theta}{r^3} = \int_0^{2\pi} \frac{d\theta}{(1+a^2+\rho^2-2a\cos\theta)^{3/2}}$$

$$= \frac{4}{[(a-1)^2+\rho^2]\sqrt{(a+1)^2+\rho^2}} E\left(\frac{2\sqrt{a}}{\sqrt{(a+1)^2+\rho^2}}\right) .$$

θ defines the position of the element $d\theta$ of the circular source involved in the integration. Values of this integral, calculated by Thorndike, are given in Table I.

We see from this table that for $\rho = 1$ the film is quite uniform as far out from the center as $a = 1$. This case was realized in the 40-inch tank by a circular array of twelve of the standard coils (Pl. XIV) spaced around a circle 36 inches in diameter, 18 inches above the face of the astronomical reflector to be coated (Fig. 1). Tests of

[18] D. Bierens de Haan, *Nouvelles tables d'intégrales définies*, Table 67, eq. 3, p. 102.

TABLE I*

VALUES OF THE INTEGRAL $\int_0^{2\pi} \frac{d\theta}{r^3}$ FOR VARIOUS PARAMETERS

a	$\rho=0.5$	$\rho=1.0$	$\rho=1.1$	$\rho=1.2$	$\rho=2.0$	$\rho=4.0$
0.00	4.50	2.22	1.91	1.65	.560	.090
0.25	4.82	2.24	1.93	1.65	.555	.090
0.50	3.96	2.29	1.93	1.63	.540	.088
0.75	7.74	2.28	1.89	1.57	.515	.085
0.80	.....	2.27	.....	.....	.....	.....
0.90	.....	2.22	.....	.....	.....	.....
1.00	8.28	2.11	1.74	1.45	.480	.082
1.50	3.40	1.38	1.09	1.02	.385	.072
2.00	1.20	0.74	0.67	0.61	.285	.068
3.00	0.28	0.24	0.23	0.22	.145	.050

* a and ρ are expressed relative to the radius of the source taken as unity.

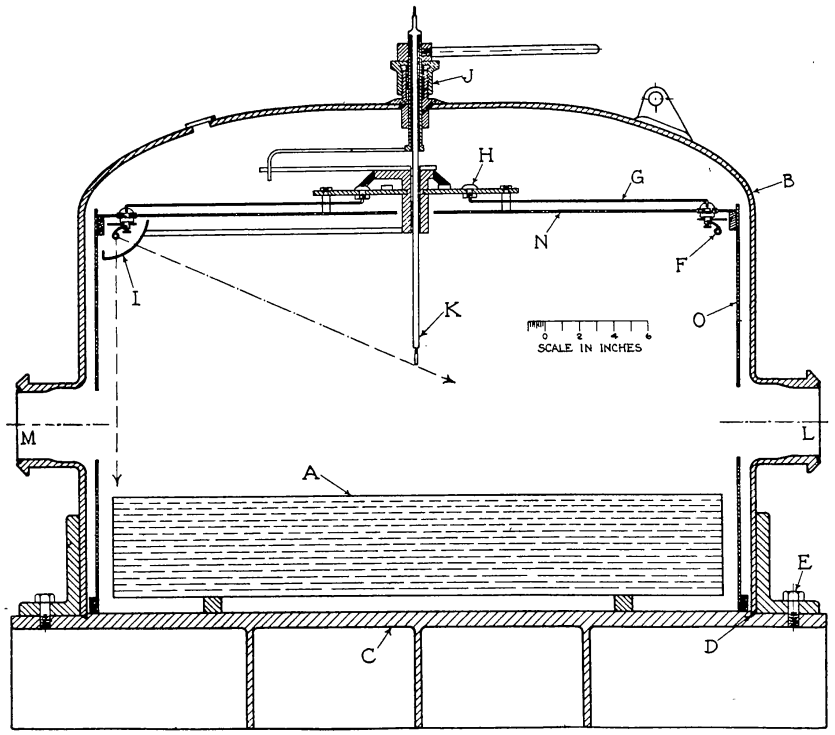


FIG. 1.—Diagram of apparatus for aluminizing mirror. *A*, mirror to be aluminized (36-inch diameter); *B*, bell-jar (39½ in. inside diameter); *C*, base plate; *D*, groove for lead fuse wire gasket; *E*, bolts to fasten bell-jar to base plate; *F*, filaments of tungsten wire from which aluminum is evaporated; *G*, conductors to supply current to filaments; *H*, switch; *I*, baffle; *J*, packing gland for switch control; *K*, electrode for cleaning the mirror face; *L*, observation window; *M*, pump connection; *N*, plate to carry filaments and switch; *O*, removable brass cylinder to carry *N*.

transmission of a film produced with partially loaded coils showed that the coat had the expected uniformity.

In the 108-inch tank it was not convenient to use a similar array of coils spaced 50 inches from the face of the mirror. Instead, three arrays were used, each about 20 inches from the mirror. From the expressions developed above, as well as from actual tests, it was found that with four coils in the center, twelve on a circle of 50-inch diameter, and twenty-four on a circle of 100-inch diameter, the film would have sufficient uniformity. The arrangement is shown in Plate XV.

The foregoing arrangement produced a film which was just opaque to sunlight. It was desirable to have this thickness (about 1000 A) since much thicker films are more easily scratched, while thinner ones may in time become transparent because of the gradual increase in thickness of the oxide layer which forms on the aluminum coat.

NON-UNIFORM FILMS

The film may be made non-uniform in thickness as desired, by a suitable arrangement of the evaporation coils or by the use of baffles, thus allowing the figure of a mirror to be altered. It is undesirable for the mirror to be refigured every time that it is given a new reflecting coat. Utilizing the insoluble nature of chromium,[25] we have devised a scheme whereby the mirror is figured with chromium. A uniform reflecting coat of aluminum may then be added or removed without affecting the true surface given by the chromium film. John Strong and E. Gaviola[19] have reported on the successful parabolizing of a 12-inch $f/7$ spherical mirror. This technique is described in an article appearing in the April, 1936, issue of the Journal of the Optical Society of America.

CLEANING THE GLASS

It is important that the surfaces to be aluminized be clean. Difficulty is met in freeing it from the contamination which is usually deposited by cotton used for drying and which is believed to be a film of fatty acid. If it is not removed, it reacts with the aluminum;

[19] Meeting of the Astronomical Section of the American Association for the Advancement of Science, June, 1935.

PLATE XV

INSIDE VIEW OF THE 108-INCH TANK, SHOWING ELECTRICAL LEADS AT LOWER RIGHT, OUTER ARRAY OF 48 COILS, INTERMEDIATE ARRAY OF 24 COILS, AND CENTRAL ARRAY OF 8 COILS AND ELECTRODE WITH ATTACHED AERIAL FOR ELECTRICAL CLEANING DISCHARGE

The tank was lined with Apiezon wax-*W*

a completed mirror which at first looks perfect will develop, in a day or two, countless tiny blisters. Even if these blisters do not develop, the adhesion of the aluminum is weakened. In former papers [20-21] a procedure was described for removing this film by a glow discharge.

The glass surface to be aluminized is cleaned by washing it with the detergent, Alphasol, then with *KOH*, and finally with concentrated HNO_3. The mirror is rinsed between each washing, with tap water. This order of the reagents is the reverse of that used in silvering because it has been observed that fatty acids adhere to the alkaline glass surface more readily than to the acidified glass surface.[22] After washing with tap water and finally with distilled water, the surface is dried and rubbed with absorbent cotton. The breath should condense on the glass, after this treatment, to form a uniform structureless gray film. If a pattern persists in the breath figure, it may be removed by a light rubbing with a rouge pad, after which the foregoing cleaning process is repeated. Small mirrors which cannot be conveniently held for drying without contamination from the fingers may be allowed to drain and dry by evaporation. Even though the breath figures in this case may have some structure, the use of the glow discharge will usually result in an excellent mirror. Very small mirrors may be simply treated in a beaker with the various reagents, rinsed and arranged to dry on a tuft of absorbent cotton.

The Alphasol[23] referred to above is one of the new detergents,[24] all of which have in common the constitution of sulphonated organic compounds of high molecular weight. These detergents have a neutral reaction and their advantage over soap lies in the fact that they may be used in neutral, caustic, or even acid solutions. Unlike soaps, the new detergents form soluble compounds with the magnesium and calcium salts common in tap water, and therefore rinse easily.

[20] John Strong, *Pub. Astr. Soc. of Pac.*, **46**, 18, 1934.

[21] John Strong, *Rev. Sc. Instr.*, **6**, 97, 1935.

[22] See also Katharine B. Blodgett, *J. Amer. Chem. Soc.*, **57**, 1007, 1935.

[23] The compound Alphasol *O T* is manufactured by the Selden Division of the American Cyanamid and Chemical Corp., Bridgeville (Pittsburgh), Pa.

[24] R. A. Duncan, *J. Ind. and Eng. Chem.*, **26**, 24, 1934.

The cleaning of astronomical mirrors is much more difficult than the cleaning of small pieces of plate glass, because of the rouge and oxidized pitch in the rough ground edges as well as in bubble holes which are often present in the face of the mirror. It is difficult to avoid spreading such contamination when the mirror is rubbed with cotton.

The glow discharge necessary for the final cleaning of the mirror is conveniently produced throughout the inside of the tank by a spark coil, or a 1-kw transformer in the case of the 108-inch tank. The discharge is produced by an electrode, or an aerial in the case of the 108-inch tank (Fig. 1 and Pl. XV).

One advantage of the chroluminum process developed by Williams is that it facilitates the aluminizing of mirrors which are otherwise difficult to clean. This can also be attained with double films where the underlying one is not chromium. The chromium, however, exhibits strong adhesion to glass,[25] without the extreme cleanliness that is required for good adhesion with aluminum. Furthermore, the chromium film is very hard. The aluminum adheres strongly to the freshly formed chromium surface. It is not, however, necessary to coat the mirrors with a double film if the cleaning technique described above is employed.

After installation in the telescope it is advisable to wash aluminized mirrors from time to time to remove dust and other contamination which accumulates with use. This is done with Alphasol, as this detergent may be rinsed off with tap water without leaving any stain. Surface film not removed by this washing will frequently dissolve in concentrated nitric acid. Sometimes it is possible to remove faint bloom with a rouge pad such as is commonly used to burnish silvered mirrors.

VACUUM TECHNIQUE

The first apparatus constructed for the application of the evaporation process was equipped with high-speed mercury pumps. This

[25] It has been discovered here that chromium films may be removed with cold hydrochloric acid if a little zinc dust is first sprinkled on the metal, otherwise it is not easy to remove them. Another technique involves making the film cathode in concentrated *HCl*. Once the solution is started, it will proceed regularly over the surface of the mirror.

will be referred to later. Cartwright and Strong later recommended charcoal absorption since, unlike most other cases where high vacuum is required, the absorption capacity of charcoal is entirely adequate for the short time involved.

One plan for evacuating the 40-inch tank involved the use of the charcoal absorption trap shown in Figure 2. The charcoal was outgassed with the heater unit shown in the figure. The water-cooling coils dissipated the heat radiated by the charcoal. After this the heater units were withdrawn; the charcoal was cooled to room temperature with tap water and then to a very low temperature with liquid air. About $2\frac{1}{2}$ pounds of liquid air were used in making one evaporation with the 40-inch tank. The vacuum attained, although not as good as that obtained with diffusion pumps, was sufficient for carrying out the evaporation. A rough estimate of the pumping speed of this trap indicated a value for air of about 30 liters per second for several minutes. However, the method was abandoned in favor of Apiezon-oil diffusion pumps, because they have a high pumping speed for hydrogen, one of the gases evolved during evaporation.

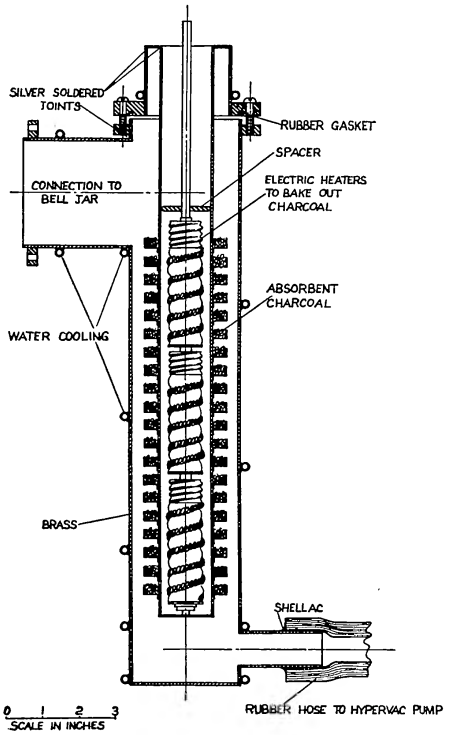


FIG. 2.—Charcoal absorption trap for obtaining high vacuum.

The Apiezon-oil pumping systems will be treated in a separate paper to be published in the *Review of Scientific Instruments*. It will be sufficient here to describe the general features of the pumping system for the 108-inch tank.

The original plan called for a charcoal trap with this pumping system, similar to the one described above, which could be cooled with "dry ice." However, it was not found necessary to use it. A long

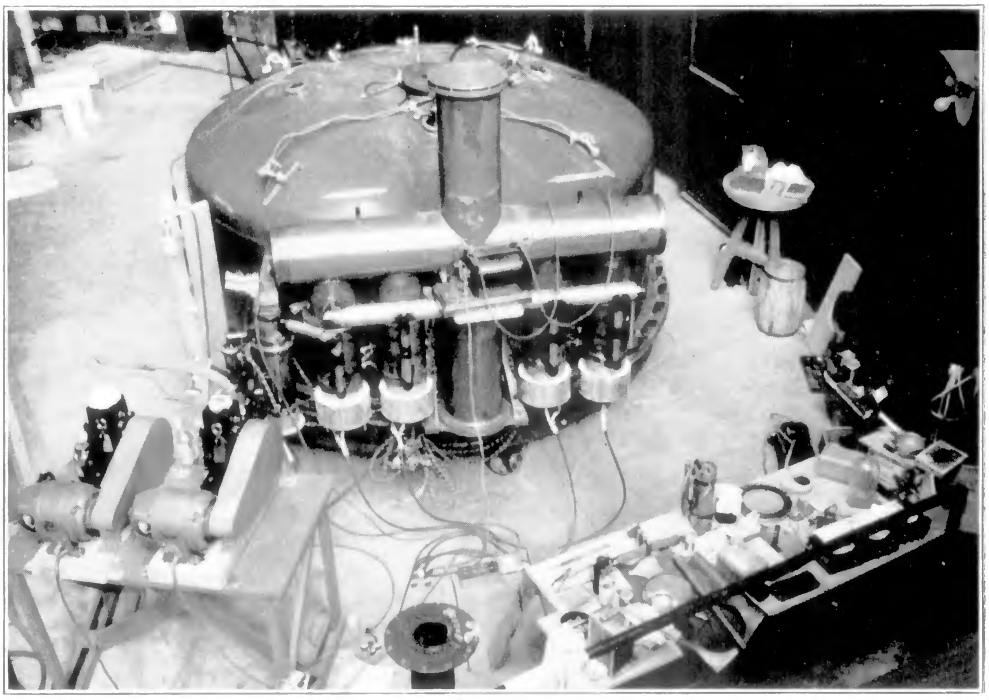
vertical pipe (Pl. XVI) was constructed to contain this trap. This pipe was connected with the 108-inch tank as well as with the horizontal manifold which supports the diffusion pumps. The manifold was connected to the three 8-inch diffusion pumps on the right, which discharge into a chamber above the 8-inch pump on the left through two manifolds, one in front and the other behind the pumps. This pump is, in turn, backed by a 6-inch and a $2\frac{1}{2}$-inch pump. Two Hypervac mechanical pumps serve to maintain the fore-vacuum for these diffusion pumps. They were capable of evacuating the tank to a pressure of 10^{-2} mm in approximately eight hours. The glass discharge tubes for testing the vacuum were connected, respectively, with the inlet and the exhaust of the last stage of the pumping system, i.e., with the three 8-inch pumps at the right, which were arranged in parallel connections.

After the tank is sealed the mechanical pumps are started. In about eight hours they lower the pressure to a point at which the diffusion pumps may be turned on. When the diffusion pumps are hot, about three-quarters of an hour after the current is switched on, they begin to pump. This is shown very clearly by the discharge tubes. The tube to the right has a greater conductivity and shows a stronger discharge than the left one when they are both at the same pressure. When pumping starts, the discharge at first equalizes and finally disappears completely in the right tube. Soon after, the discharge also disappears in the other tube.

The purpose of this rather elaborate pumping system is not so much to realize a degree of vacuum at which the evaporation process can be performed, for one hypervac pump gives a vacuum of 5×10^{-4} mm of mercury in the 108-inch tank. Its purpose is the maintenance of a high vacuum after evaporation is started, in spite of the outgassing induced by the lighted filaments. The aluminizing process has a tendency to improve the vacuum by the "getter" action, and if the evaporation can be done rapidly enough, the pumping speed required can be materially reduced. Another advantage is that with such fast pumps the time involved in evacuation is less, and also considerable leaks may be tolerated. It is planned to use the same pumping system for evacuating the 200-inch tank when it is built.

The performance of this pumping system is illustrated by the log

PLATE XVI



VACUUM PUMPING SYSTEM AND GENERAL VIEW OF INSTALLATION IN THE 100-INCH TELESCOPE DOME

of the run in which the 100-inch mirror was aluminized. This is given in Table II.

It was found useful to line the 40-inch and 108-inch tanks with Apiezon wax "W," which has a vapor pressure so low that it is possible to evaporate aluminum directly on it.[26] It stops all leaks and covers up such "dead alleys" as are common in steel, so that the tank has, in effect, the strength and convenience of steel and the vacuum behavior of a glass tank. It is believed that this technique of coating the inside with Apiezon wax may be of value elsewhere in

TABLE II

LOG OF THE VACUUM WHEN THE 100-INCH MIRROR WAS ALUMINIZED

8:10 P.M.	Pumping started with two Hypervac pumps.
7:55 A.M.	Vacuum 5×10^{-3} mm mercury
	High-tension discharge from kilowatt transformer for 15 minutes at 6×10^{-2} mm of mercury air pressure
8:10 A.M.	Diffusion pumps turned on
9:33 A.M.	Vacuum 2×10^{-5} mm of mercury
	Mirror aluminized
	40 coils fired 15 seconds each
10:45 A.M.	After aluminizing
	Vacuum 5×10^{-5} mm of mercury
	Mean free path about 150 inches
	Diffusion pumps turned off
2:00 P.M.	Vacuum 3×10^{-3} mm of mercury
	Air introduced and mirror removed from tank

obtaining high vacua in large steel tanks. When first evacuated with a single Hypervac pump, a pressure of 5×10^{-4} mm of mercury was attained in the tank, indicating that there were no significant leaks. After this it was coated with two coats of Glyptal on the outside, one red and one machine blue, to insure complete coverage.

REFLECTIVITIES AND PROPERTIES OF EVAPORATED FILMS

Reflectivity measurements have been made on several films with monochromatic light obtained with a Müller-Hilger double-quartz monochromator. A Nernst filament as well as the sun served as light-source, and quartz sodium and potassium photo-cells as receivers. The photocurrent was measured with an electrometer by a

[26] At 180° C the vapor pressure of this compound is 10^{-3} mm of mercury.

null-deflection method, as well as with an amplifier and galvanometer. In every case the square of the reflectivity was measured, thus giving a higher precision in the result. The probable error of the measurements is smaller than 1 per cent for R greater than 85 per cent, and is about 2 per cent elsewhere.

The results of the measurements on various films are shown in Figures 3, 4, and 5. They include measurements on silver films which have tarnished for two and three weeks and also films which have been burnished.

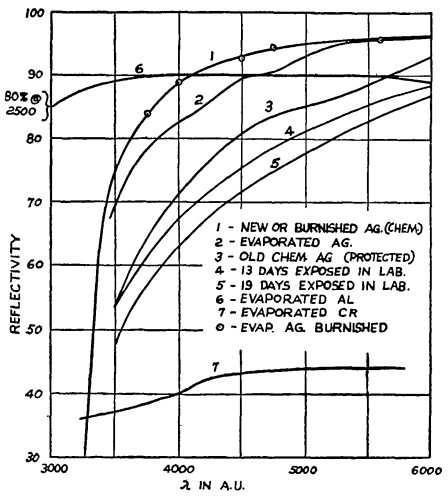


FIG. 3.—Reflectivity measurements made with the Müller-Hilger double spectrometer-monochromator, Nernst filament light-source, sodium-quartz photocell and electrometer.

W. W. Coblentz and R. Stair[27] have recently measured the reflectivity of fresh silver, and have obtained results which are definitely higher than the values obtained by Hagen and Rubens.[28] The author's measurements are in agreement with those of Coblentz and Stair in indicating definitely higher reflectivity for fresh silver than that obtained by others.

It is important to point out that differences were observed for gold, copper, and silver between evaporated and polished massive metal. This is shown, in the case of silver, where burnishing with a rouge pad increases the reflectivity from that characteristic for the evaporated metal (curve 2, Fig. 3) to that characteristic for burnished "chemical" silver (points on curve 1). Freshly silvered mirrors (by the chemical method) are frequently "toughened" by rubbing them with cotton before they are burnished with a rouge pad. Otherwise they exhibit a faint reddish color, indicating that they are charged with rouge. It is probable that both chemical and evaporated silver require burnishing to form the metal into a compact film

[27] *Bur. of Standards J. of Res.*, **2**, 343, 1929.

[28] *Ann d. Phys.*, **1**, 352, 1900; **8**, 1, 1902.

which exhibits the higher reflection coefficient. Figures 3 and 4 show also the reflectivities of *Cr*, *Pt*, *Pd*, *Rh*, and *Sn*.

The reflectivity of a 12-inch telescope mirror coated in October, 1932, and used continuously since that time, was recently tested by the substitution method by comparing it with a freshly aluminized mirror of equal radius of curvature. An incandescent lamp filtered with A, B, and C Wratten filters, respectively, was used as the light-source for these tests. It showed no decrease of reflectivity in the red and green and a decrease of 1 per cent in the blue.

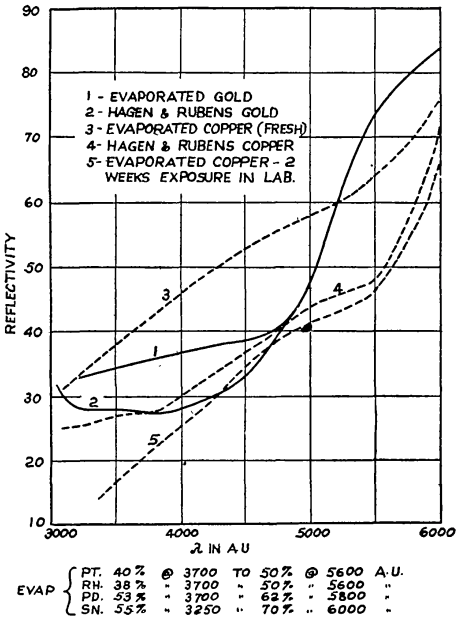


FIG. 4.—Reflectivity measurements made with the Müller-Hilger double spectrometer-monochromator, Nernst filament light-source, sodium-quartz photocell and electrometer.

The superior reflectivity of aluminum over silver for short wavelengths not only gives a more even exposure of the stellar spectra in the violet but extends the range of the observable spectrum some 250 A, making observations possible to the limit of the transmission of the atmosphere. For the demonstration of the excellence of aluminum mirrors for photographing stellar spectra in the ultra-violet we are indebted to the pioneer work of Williams and Sabine, and later of Williams and Boothroyd, as well as to the work of W. H. Wright.

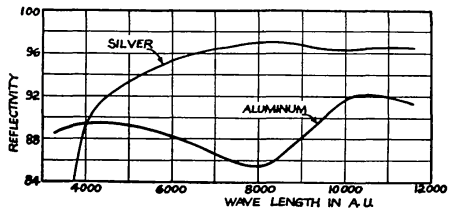


FIG. 5.—Reflectivity measurements made with the Müller-Hilger double spectrometer-monochromator, potassium photocell with amplifier and galvanometer. Sunlight used as light-source.

Figure 5 shows that aluminum is inferior to silver in reflectivity in the red and near infra-red. For spectroscopic studies in this spectral range where the light is weak this is serious. It is suggested that for this work the aluminized mirror may be temporarily coated with

silver by the chemical process. Afterward it may be removed with concentrated nitric acid, leaving the aluminum unaffected.

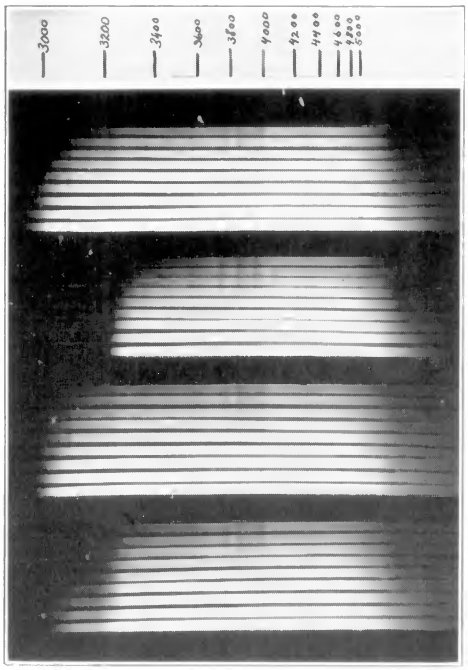
In this connection the photographs of the sky spectrum shown in Plate XVII are of interest. The spectral distribution here corresponds to that of a hot star. These photographs were taken with a Hilger E-30 quartz spectrograph on Eastman 40 plates. As the slits were 0.0055 inch in width and the dispersion at H and K was only 11 A/mm, the absorption lines are not very distinct. Nevertheless, the features of these spectra as they are influenced by reflection from silver and aluminum are evident. The exposure times within each of the four series were increased by factors of 2, the shortest being 4 seconds. The top series was exposed to the zenith at 2:00 P.M. on July 30, 1935, with three reflections from aluminum at an incidence of 45°. The second series was taken at 2:30 P.M. with three silver reflections. The two series at the bottom were taken at 4:00 and 4:30 P.M. with one aluminum and one silver reflection, respectively. After account is taken of a gradual decrease in the strength of the light-source during the afternoon, it is seen that the blackening beyond 5000 A is sensibly the same in all four series. The gain of aluminum over silver in the violet and ultra-violet is greater in the case of three mirrors, the extension of the spectrum by aluminum over silver being about 250 A.

This increase in the length of the spectrum is also shown by the photographs (Pl. XVII) of a planetary nebula taken by W. H. Wright with the Crossley reflector before and after aluminizing. Dr. Wright[29] states that photographs of the north polar sequence taken by Dr. Mayall before and after the mirror was aluminized show a limiting magnitude of 19.3 with silver and of 19.8 with aluminum. Tests made after one year of service did not indicate any change in this limit.

If we choose one of the various reflectivity-curves for silver shown in Figure 3, in such a way that aluminum gives a gain of $0^{M}.5$ (photographic) for an Ao star, we compute that the gain for a Cassegrain arrangement with three reflections is about 1 mag. This calculation is made for an Eastman 40 plate and for the normal transmission of the atmosphere. It will be of interest to see how this cal-

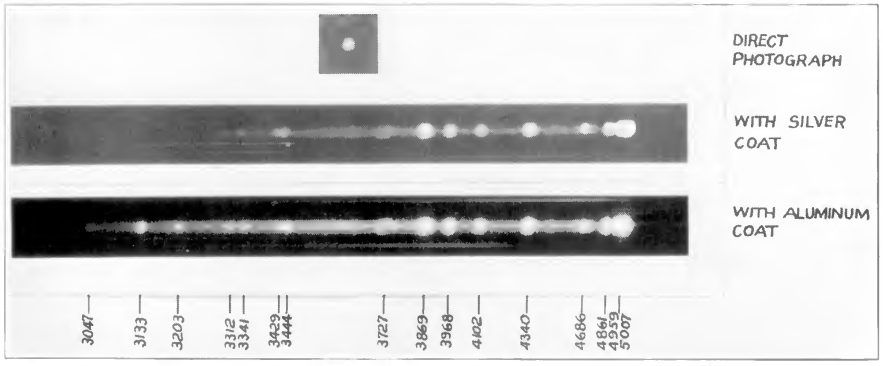
[29] *Pub. of Astr. Soc. of Pac.*, **46**, 32, 1934.

PLATE XVII



SKY SPECTRA

Upper series taken with three reflections from aluminum mirrors at 45° incidence. Each successive spectrum has double the exposure of the preceding one. Second series, three reflections from silver. Third and fourth series, one reflection at the same angle of incidence, 45°, from aluminum and silver, respectively.



SPECTRA OF NGC 7662

Taken with the Crossley reflector of the Lick Observatory by W. H. Wright

culation will agree with the results obtained with the 60-inch and the 100-inch telescopes on Mount Wilson.

Aluminized mirrors scatter less light than silvered mirrors, as the latter have to be burnished and this process leaves many small scratches upon the silver. We may appreciate this difference from the fact that with the fresh aluminum coat it was possible to photograph[30] the companion of Sirius at the Cassegrain focus of the 60-inch telescope on an unbacked plate.

The effects of coating speculum gratings by evaporation is to increase their reflection about 50 per cent. Other effects have been made the subject of a separate paper published in the February 15 issue of the *Physical Review*. Successful results have been obtained by coating replica transmission gratings to change them to reflection gratings.

OXIDE FILMS ON ALUMINUM

The aluminum film is automatically protected from tarnishing by an oxide film (presumably corundum, Al_2O_3, or bauxite, $Al_2O_3 \cdot 2H_2O$) which starts to form as soon as the aluminum comes in contact with the air. This oxide becomes thicker with time for about sixty days, when it is very hard and tough, forming a surface not easily scratched when it is being dusted and cleaned.

The formation of the aluminum oxide film on an exposed metallic aluminum surface has been studied by Vernon,[31] who weighed the sample to a precision of 1/100 mg and plotted the weight increment due to oxidation as a function of time. The period of time required for the oxide film to attain its natural thickness was seven to fourteen days. After this the thickness of the film remained nearly constant at 100 A.

This oxide layer is, of course, too thin to give interference effects. It could be much thicker, however, and still not give interference, because of its transparency and the high reflectivity of the underlying aluminum.[32]

[30] M. H. Humason, *ibid.*, **47**, 83, 1935.

[31] *Trans. Faraday Soc.*, **23**, 113, 1927.

[32] For a discussion of colors of transparent films on metallic surfaces see Robert W. Wood, *Physical Optics*, p. 206, 1934.

It has been observed for a long time that nitric acid does not dissolve an aluminum film. Experiments have shown, however, that treatment with this acid will apparently increase the thickness of the oxide film by about 50 per cent. These conclusions were inferred from measurements of changes in transmission and reflectivity of transparent films, and are based on the assumption that the nitric acid forms aluminum oxide on the surface of the film. The measure-

TABLE III

EFFECT OF OXIDATION OF ALUMINUM ON REFLECTION AND TRANSMISSION

Mirror	Age	Remarks	R	T	$R+T$
A and B.....	5 min	90 A in thickness	62	18	80
A.....	10	Washed 45 sec with HNO_3	56	22	78
A.....	15	Washed 60 sec with HNO_3	53	25	78
A.....	4 hr	.....	47	29	76
A.....	18	.....	46	31	77
A.....	46	.....	46	32	78
B.....	56	Not washed	53	26	79
A.....	71	.....	45	31	76
B.....	71	.....	52	26	78
A.....	5 days	.....	43	32	75
B.....	5	.....	51	26	77
A.....	12	.....	42	33	75
B.....	12	.....	50	28	78
B.....	12	Washed 60 sec with HNO_3	50	28	78
A.....	16	.....	41	35	76
B.....	16	.....	49	28	77
A.....	41	.....	37	36	73
B.....	41	.....	44	30	74
A.....	61	67 A in thickness	38	35	73
B.....	61	73 A in thickness	44	30	74

The reflectivity was measured by comparison with an opaque aluminum film, with green light. The reflection of aluminum was taken as 89 per cent. The transmission measurements are uncorrected for glass backing.

ments of transmission, T, and reflection, R, given in Table III, are for two plates coated simultaneously with the same thickness of aluminum. One of these, Plate A, was treated with acid, and the other, B, was allowed to oxidize naturally. One sees from the table that the reflection and transmission coefficients, originally $R=0.62$ and $T=0.18$, are changed to $R=0.56$ and $T=0.22$ by one nitric acid bath, and to $R=0.53$ and $T=0.25$ by the second bath.

The film was originally 90 A thick. If we interpret the changes of T and R as due simply to a decrease in the thickness produced by oxidation of the superficial aluminum, then film A which stabilizes

at $R=0.44$, $T=0.30$, is about 67 A thick. Film B stabilizes at $R=0.38$, $T=0.35$, or at a thickness of approximately 73 A. Assuming the oxide layer to be hydrated alumina ($Al_2O_3 \cdot 2H_2O$), it turns out that the thicker oxide film, A, is 62 A and the thinner one 46 A.[33]

The method of modifying the oxide film by treatment with HNO_3 would seem to have practical application to telescope mirrors in

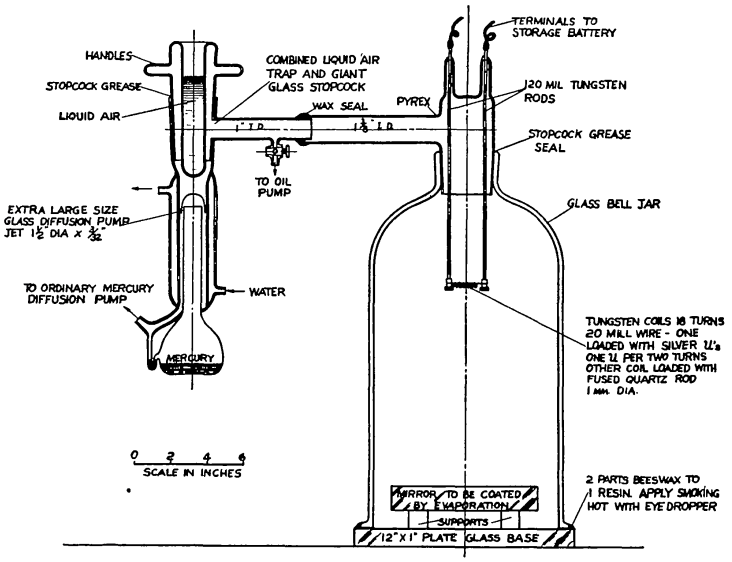


FIG. 6.—First evaporation apparatus constructed at the University of Michigan in 1929.

making the film more resistant to abrasion. The column $R+T$ in Table III, giving the efficiency of transparent aluminum films, is of interest in connection with their use on interferometer mirrors for dividing a beam of light.

EQUIPMENT

The apparatus constructed by the author at the University of Michigan in 1929–1930 is shown in Figure 6. This is, to my knowl-

[33] This thickness was computed, assuming the inverse-square law to hold for the intensity of the evaporated molecular beam. Also zero reflection was assumed for the aluminum beam. It must be remembered that although we have considerable evidence to support these assumptions, they must, nevertheless, be regarded strictly as assumptions. We have, on the other hand, some evidence that the thickness of the aluminized film produced by evaporating a given mass of metal is less when the vacuum is inferior, and at the same time granular aluminum deposits appear on surfaces not exposed to the evaporation sources. Although in the present case the vacuum was above 10^{-4} mm, the numbers associated with these film thicknesses are to be considered as only approximate.

edge, the first apparatus constructed for the general application of the evaporation process to the coating of mirrors. The special large mercury diffusion pump for evacuating the glass bell jar in which the evaporation is carried out was constructed with a large built-in stopcock and a liquid-air trap which permitted the recharging of the evaporation coils and the introduction of new mirrors without destroying the vacuum in the diffusion pumps. Four electrodes allowed several coils of tungsten to be fired. The base plate for the bell jar was of glass. After the assembly of the bell jar on the base plate with the pumping manifold and filament electrodes, the whole was attached to the pumping system by means of a wax joint. In a few minutes, when the wax was cool, the bell jar was evacuated by the vacuum pump through the by-pass. After this, the by-pass was closed, liquid air was introduced into the trap, and the large valve was opened. In a few minutes the vacuum was good enough (i.e., 10^{-4} mm of mercury or better) for the coils of tungsten, containing the metal (or non-metal), to be fired. A minute or two were required for the evaporation of the silver, during which time the bell jar became a beautiful blue as the silver condensed on it, forming a film of increasing opacity.

The next apparatus, developed by Cartwright and Strong, used the absorption of charcoal at liquid-air temperatures to obtain the necessary high vacuum. The evaporation was carried out in a glass bell jar sealed to a steel base by means of a mixture of beeswax and resin (2:1). This apparatus was later equipped with a mercury vapor pump. This is shown in Figure 7, together with the 14-inch cast-aluminum bell jar later constructed for use with it.

In May, 1932, experiments were started preparatory to coating, by evaporation, the 100-inch Hooker mirror of the Mount Wilson Observatory. The first plan considered was to use a small bell jar which would be placed directly on the mirror surface, thus using the mirror as a base plate to seal the vacuum system. This would make it possible, with the help of a baffle, to coat an area of hexagonal shape. By repeating these hexagons side by side it would be possible to cover the entire surface of the mirror. Difficulty was met in finding a material to use for the rim of the bell jar which would give a vacuum-tight seal without injury either to the film already deposited or the mirror face. After considerable experimentation a lacquered hard-

rubber foot about $\frac{1}{8}$ inch wide was found satisfactory. The rubber was mounted in a groove in the rim of the brass bell jar. It was coated with several thoroughly dried layers of colorless brushing lacquer to keep it from sticking to the metallic film already laid down. Preliminary tests were made on a piece of plate glass, indicating that this technique could be made successful.

The plan was abandoned, however, when it became apparent that the construction and evacuation of a tank large enough to coat the whole mirror at once was possible.

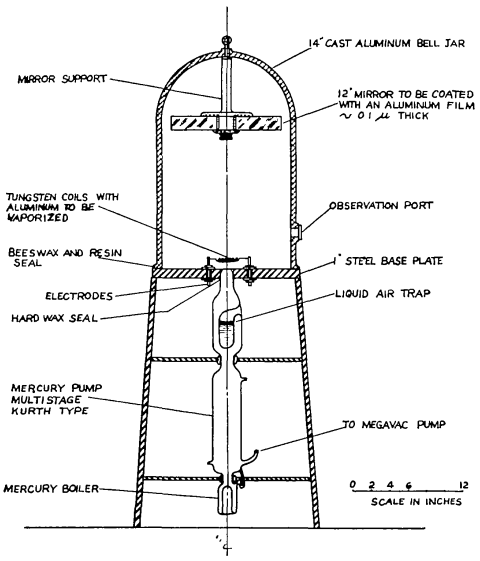


FIG. 7.—Small evaporation apparatus used to coat mirrors to 12-inch diameter as well as for general experimental work.

The next apparatus is shown in Figure 1. This was constructed with the possibility in mind of coating the 36-inch Crossley reflector. It was different from any constructed before in two important respects: it was made of ordinary boiler-plate steel throughout and was arranged to coat the mirrors face upward from coils directly above them. The operation of this tank has proved that the simple method of laying the mirrors on the base plate face upward is entirely successful. It has been used more than a hundred times and we have never had molten aluminum fall upon a mirror.

The 108-inch tank for the 100-inch Hooker mirror is shown in Plate XVI, together with the high-vacuum pumping system constructed for use with it. The tank was made from a steel cylinder 108 inches in diameter, 36 inches high, and $\frac{5}{16}$ inch thick. It was closed at the top with a bumped tank-head $\frac{3}{8}$ inch thick. The cylinder was fitted with two 8-inch ports, one for the electrical connections and the other for the pumping system. At the bottom it was connected to a ring on which a $\frac{1}{2}$-inch tongue was machined. This tongue was seated on a soft rubber gasket, $\frac{1}{2}$ by $\frac{1}{4}$ inch, retained in a groove machined in the base. The base was made from $\frac{5}{16}$-inch plate

reinforced beneath by *I*-beams. A ring was welded to it around the periphery, for strength, and the groove was machined in this to fit the tongue referred to above.

All the joints in this jar were electrically welded with three passes. The jar was X-rayed and stress-annealed before machining. After machining it was tested with 20 pounds per square inch of internal pressure for strength and leaks. The latter were detected with soapy water and repaired by peaning.

Plate XVI shows the 108-inch tank, with pumping system connected, installed in the dome near the 100-inch telescope. The charcoal trap, not used, is shown in the foreground. The four cables connected to the top of the tank lift both tank and pump from the base plate for introducing the mirrors and loading the coils.

HISTORY OF THE APPLICATION OF ALUMINIZING TO ASTRONOMICAL MIRRORS

It is not known to me when the application of the evaporation process to astronomical mirrors was first conceived, but, to my knowledge, the first astronomical mirror to be coated by the process was that of Professor Philip S. Fogg, Registrar of the California Institute of Technology. The author coated his 6-inch mirror, which he had ground and figured himself, with silver and a protecting layer of quartz by the evaporation process on January 14, 1931. Soon after this Cartwright coated several mirrors with silver and quartz for the Hale spectrohelioscope at the Brackett Observatory, Claremont, California.

The first group of mirrors aluminized by the new technique were coated June 19, 1932, by the author. One of these mirrors, which was sent to Albert G. Ingalls of the Scientific American, was not tarnished two years later.

The work at Cornell University on the application of the evaporation process to large astronomical mirrors was started in the summer of 1931, culminating in the coating, with chromium, of the 10-inch concave mirror of the Physics Department, and of the 15-inch mirror of the Lowell Observatory, in July, 1932.

During October, 1932, I coated with aluminum the 12-inch mirror of the Cassegrain telescope of the California Institute. This mirror is now the oldest aluminized telescope mirror, being three years old. The deterioration of its reflectivity has been less than 1 per cent.

R. C. Williams undertook the coating of astronomical mirrors with aluminum in the autumn of 1932, leading to the coating of the 15-inch mirror of the Lowell Observatory in August, 1933. His work ultimately led him to the conclusion that by first making a chromium mirror whose surface is then brightened with aluminum a reflecting film is obtained that is superior to a plain aluminum mirror with respect to hardness and adhesion. He has suggested the name "chroluminum" for this combination.

Hiram W. Edwards has applied the evaporation technique to magnesium alloys of aluminum which are reported to be superior in reflectivity to pure aluminum. He uses the name "panchro" for this coating.

In September, 1933, at the California Institute of Technology, the 18-inch coelostat mirror of Dr. Hale's solar telescope was aluminized by the author. In November, 1934, the mirrors of the 60-foot and 150-foot tower telescopes, as well as several of the Cassegrain, Coudé, and Newtonian auxiliary mirrors of the 60-inch and 100-inch telescopes of the Mount Wilson Observatory, were aluminized. In December, 1933, an aluminum coat was applied by the author to the Crossley reflector of the Lick Observatory. In January, 1934, the 24-inch reflector of the Yerkes Observatory was aluminized.

The largest telescope mirrors that have been coated are the 60-inch (on February 27, 1935) and the 100-inch (on March 1, 1935) of the Mount Wilson Observatory.

I wish to acknowledge the many helpful suggestions I have received from Dr. John A. Anderson, and the assistance of Professor E. Gaviola and of the staff of the Mount Wilson Observatory, during the coating of the 60-inch and 100-inch mirrors; and to thank Director W. H. Wright of the Lick Observatory for supplying the photograph used in Plate XVII. I also wish to acknowledge the valuable advice of Mr. G. W. Sherburne, Mr. Mark Serrurier, and Mr. S. Hart in the design and construction of the 108-inch tank.

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PASADENA, CALIFORNIA
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