THE PREPARATION OF METALLIC MIRRORS, SEMI-TRANSPARENT AND TRANSPARENT METALLIC FILMS AND PRISMS BY DISTILLATION[1]

By Otto Stuhlmann, Jr.

The necessity for a quick, reliable and practicable method for making gas-free and pure transparent metallic films, which were to be used for some photo-electric and optical research, lead to the development of the following simple device for making transparent, half silvered and opaque silver reflectors. Those who have gone thru the tedious process of making and polishing silvered and half silvered surfaces, especially such as are used in interferometers, will appreciate a method which allows one to arrest and observe the development at all times in the process of deposition.

The essential part of the apparatus consists of a wire, heated to incandescence by means of an electric current, while the object to be covered with the metallic surface is placed below it. If the whole is now placed in vacuo, the metal will vaporize and condense upon the object. If the incandescent wire is now moved with uniform speed across the object, a uniform coat of the metal will be deposited. The thickness of this condensed metallic film depends on the time of deposit, the temperature of the wire, the molecular weight of the vapor and its vapor pressure.[2]

The accompanying figure shows an apparatus in use for the deposition of silver on concave, convex, plane and irregular surfaces. The material to be silvered can consist of anything not having a pronounced vapor pressure at room temperature and 0.002 mm. of mercury pressure.

The working parts were mounted on a thick plate of rolled brass, grooved to retain a circular bell jar BJ. Mercury or hard sealing wax floated into this groove, formed the seal for the vacuum. This was never less than 0.002 mm. of mercury pressure. The vacuum was obtained by means of a Gaede rotary mercury pump. The bell jar had an internal diameter of eight inches and was five inches high.

The carriage HE, supports the metal to be evaporated in the form of a wire, about 0.017 inches in diameter. The wire is fastened between the

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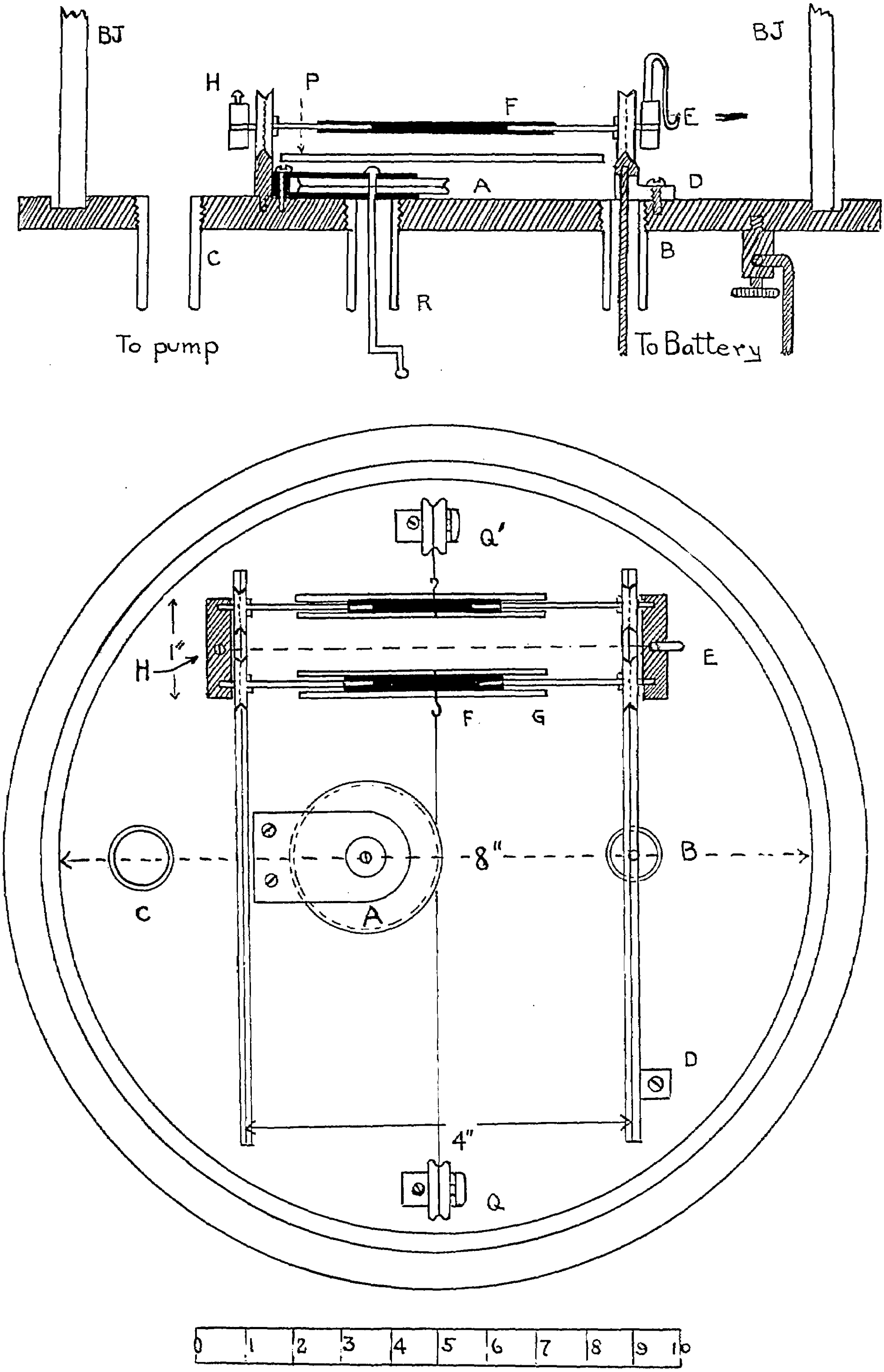
[2] Langmuir (Phys. Rev.V.2, p. 340, 1913) has shown that $m = \sqrt{\frac{M}{2 \pi R T}}\, p$ where m is the rate of evaporation in vacuo, M the molecular weight of the vapor and p its vapor pressure.

two points H and E as shown by the dotted line in the diagram. At H it is attached to a screw at the top of the rod connecting the two wheels, from where it passes down and then thru a large hole in the rod between the wheels, then across and thru a similar hole in the rod at E, thence to the spring brass hook. This springy hook supplies the necessary tension to the wire to keep it taut. Upon heating the wire, it expands and the slack is further taken up by the spring in the hook. A 110 volt alternating or direct source can be used for the heating current. Under these conditions a 0.017 inch silver wire needs about eight amperes to heat it enough to produce the desired vaporization.

The heating current enters the inclosure thru the baseplate at the binding post to the right. It then passes into the metallic track at H which is screwed to the baseplate. From here it passes thru the left side of the carriage, thence thru the wire across to E into the right hand track. This track is insulated from the base by means of two fibre L-shaped supports. The current passes from this insulated track thru the rod B back to the battery. The rod B passes thru a hole in the base and is sealed in place with hard red sealing wax, which serves to insulate it from the base and at the same time seal the opening air tight.

To prevent the wire from being short circuited thru the rods connecting the two ends of the carriage, they were separated at the middle by hard fibre. This joint F was then covered with a tight fitting glass tube extending about half an inch beyond each end of the fibre insulations. This prevented the fibre from becoming coated with a layer of the conducting metallic deposit which would eventually accumulate on it and short circuit the wire.

The glowing silver wire, as mounted on the carriage, gradually evaporates and the vapor condenses on the material placed at P. This plane is about one inch below that of the wire. In order to cover a small area with an opaque deposit, it is only necessary to roll the carriage along its tracks so that the glowing wire is over the center of this area. A larger area can be covered when the wire is moved with uniform speed across it, so that the metallic vapor may condense uniformly. In order to move the heated wire it was necessary to have a mechanical control outside of the apparatus. This was supplied by means of a small crank which passed thru a ground glass stopper, sealed in the tube at R. This crank turned the horizontal, V-grooved wheel at A. Around this wheel passes a belt



Scale 2 cm = 1 inch.

to the small wheel Q, from where it passes to a hook on the carriage at F. The other end of the belt passes over a similar wheel at Q, and thence to a similar hook on the other side of the carriage.

By turning the crank R, the carriage can be rolled with any desired speed back and forth across the area between the tracks. This allows an area approximately equal to the area inclosed by the tracks to be covered with the metallic deposit.

In order to get a very pure deposit, it is necessary to use silver free from other metals. The occluded gases, and absorbed impurities that naturally accumulate on the surface in the process of manufacturing and mounting the wire can be removed by heating it, when at one end of the tracks and away from the surface to be covered with the material. The gases thus driven off are removed by the pump and the impurities condense on the apparatus in the immediate vicinity of the carriage. The wire thus cleaned can now be rolled over the surface to be plated. To facilitate the deposition of opaque surfaces of silver especially where a slight admixture of platinum is not objectionable, it was found practicable to use an alloy of 15 per cent. platinum and 85 per cent. silver. Since the boiling point of silver is about 800°C. below that of platinum, it follows that silver can be rapidly removed by evaporation without melting the wire.

All the metals having a low melting point like zinc or tin, can be deposited very rapidly and with much ease from the well known alloys of brass and phosphor bronze. While those having a high melting point can be deposited from wires made out of the pure metal.

The advantage of this method over the chemical reduction process of depositing silver becomes apparent when one recalls the excessive care that must be exercised in cleaning the surfaces upon which the deposit is supposed to take place. In addition the often hopeless task of controlling the thickness of the deposit, when semi-transparent mirrors are required, is well known. The distillation method only requires the surfaces to be free from grease. This is easily accomplished by washing them in a bath of sodium hydroxide and then in some dilute acid. Rinsing off the acid and drying the surface with a clean lintless cloth completes the operation.

The surfaces upon which the deposition is to take place can be either plane, concave, convex or irregular. They can be any material, and even cardboard or paper can easily be covered with a mirror like surface if care is taken not to deposit the material too rapidly or at too high a temperature. This method lends itself especially well to the making of galvanometer mirrors. In this apparatus the whole area between the tracks can be filled with the disks of glass and all simultaneously covered with a thick silver deposit, evaporated from the silver platinum alloy, in about one minute after the desired vacuum has been obtained.

The metal deposited by evaporation need not be polished, which is of great value when pure optical surfaces are wanted. Those made by other methods involving a chemical process always need some polishing with the inevitable adhesion of impurities.

Where semi-transparent silvered surfaces are desired, as those used in all interferometers, the procedure is as follows. A silver wedge is deposited by allowing the wire to stand over a microscope slide until the region just below the wire is opaque. In both directions at right angles to the wire the thickness diminishes with the distance. This wedge can then be examined for the required thickness which will produce the best combination of transmission and reflection. This region has a definite color by transmission which can be matched with the interferometer films undergoing deposition in the apparatus. To facilitate the color matching, place a white card under the interferometer plates. Since several plates can be silvered simultaneously it follows that all plates exposed to the vapor for the same length of time, have equal densities of metal deposited upon their surfaces.

In the chemical reduction of silver and its deposition on the plates no such exact results can be obtained. Here a thick film must be deposited and the surface gradually polished down to the required thickness. This usually requires great skill, and more often than not results in variations in thickness of the deposit and the adhesion of impurities. If the polishing is carried too far, the whole process must be repeated.

Similar deposits can also be produced by cathodic sputtering in vacuo. The objection to this method is not apparent at once. Its chief objection is, however, the slowness of deposition, and the penetration of the particles into the glass itself, which in turn results in a strain and a distortion of the reflected image, making the surfaces optically useless. The deposit also contains all the impurities found in the cathode.

The distillation method also allows one to resilver mirrors or surfaces permanently attached to instruments without the necessity of removing them from their often delicately adjusted supports.

The instrument was built by Mr. Kalmbach, the laboratory mechanician, to whom I am indebted for some valuable mechanical suggestions.

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A NEW TYPE OF COMPARISON PHOTOMETER

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The Martens comparison photometer* has for years been the favorite instrument for precision work in measuring relative transmission, reflection and relative brightness in general. It gives a good comparison field free from any perceptible dividing line. It gives the highest precision of which a visual instrument is capable and is so compact and well protected that it is seldom injured or thrown out of adjustment.

This is a polarization instrument, however, and if the light enters it in a partly polarized condition, either serious errors result or a tiresome duplication of readings is necessary to eliminate such errors. A much more serious objection, however, is that the instrument is no longer on the market, since the calcite for making the Wollaston and nicol prisms used for varying the light intensity in a known manner is not available. The new instrument was designed to do the precision work for which the Martens photometer is ordinarily used, but with more flexibility to take care of a wider variety of work. The use of calcite and of the polarization principle is avoided in its construction.

In the new type of instrument, the light beam is divided and recombined by means of bi-prisms and the density is controlled in a known manner by means of a precision iris diaphragm or neutral gray wedge inserted at the precise apex of the field pencils. The field viewed is bi-lateral and circular. The scale is empirical and very nearly linear. The two instruments constructed for the Research Laboratory of the Eastman Kodak Company have shown high sensibility and precision.

The construction of the instrument is as shown in the figure. The instrument is symmetrical about a longitudinal plane. In each beam a

*Phys. Zs. Vol. I, page 299, 1900.