# The Application of Metallic Fluoride Reflection Reduction Films to Optical Elements

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#### Foreword



THIS PUBLICATION is neither a text nor a manual. It is a record of a Conference by members of The Optical Instrument Committee on the Application of Metallic Fluoride Reflection Reducing Films to Optical Elements. As such it comprises valuable information on the techniques of quantity production of evaporated films.

The widely varying experiences of the speakers and the diversity of the subjects they discussed provide the reader with a copious source of information from which he may select much that can be successfully applied to his own processes.

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### History



**Col. Welch:** Frankford Arsenal

This whole business of non-reflective film apparently started the year I was born so I probably grew up with it although during most of

that time, I didn't know it. However I suppose it remained more or less a laboratory phenomenon until the first World War, when certain of the manufacturers whom I think may be present here today, experimented with means for improving the performance of optics by non-reflection coating. Fred Wright¹ also experimented with these coatings during that war. I think he was stationed with Bausch & Lomb at that time. These films came to my attention about fifteen years ago during a discussion of methods of improving optical performance. At that time many people reported that the use of films could really accomplish that end. A few years later, a good many people were working on it, among whom were the Messrs. Cartwright and Turner. Some of them may be here today.

It was to outline the method that brought us together in 1939. In that same year, Keuffel & Esser Co. submitted to Frankford Arsenal a Height Finder Tracking Telescope, which had been treated by a non-reflective coating process, and in July of that year General Wells, who is now our Chief of the Artillery Branch in Washington, then in the Design Section of Frankford Arsenal, took cognizance of the military possibility of this phenomenon and started a program to determine its performance and durability. In 1940, our own laboratory at the Arsenal submitted a report in the furtherance of this program, but at that time questioned the durability and stated that it was too early to make any definite recommendations. In 1941, the Washington Navy Yard started a definite program to secure the benefits of the well-known scientific process and developed at that yard what seems to us the most useful and successful procedure yet obtained for military instruments. That is the process, procedure and final results that Mr. Goetzenberg spoke of a while ago.

During this period Frankford Arsenal continued its investigation, largely under the leadership of Major Sturr, until the end of 1942 when certain factors caused us to intensify our work. These factors revolved primarily about sights, telescopes and periscopes for Tank and Combat Vehicle use. Due to size limitations and other restrictions in Tank and Combat Vehicles, this type of tele¹Dr. P. E. Wright, Technical Adviser Joint Optics Conm. ANMB.

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scope was rather limited as to power and dimensions. At the same time, it had to be used both at dawn and dusk and under many other conditions of poor visibility. It was essential therefore, that every practical means be used to make the best telescope possible within these limitations. We therefore first applied coated optics in production of telescopes for Tank and Combat Vehicles. The early orders were placed with RCA. At that time, we had set up the manufacture of telescopes so that optical elements were produced in one place, coated in another and transmitted to a third plant for assembly. Everyone concerned expressed considerable doubt as to whether such a chain of handling could be made to work. A thorough test at the Westinghouse Mansfield plant showed that it could.

We were also confronted with the problem of what to do about cemented doublets. If we applied what seemed to us the best method, the Navy Yard process, the heat would uncement the doublet. That caused us to turn more and more to the policy of having the coating done in the plant producing the optical elements, where the manufacturing process could be so controlled as to permit coating first and then cementing.

When it became evident that it was practical to produce optics in quantity production treated with these efficient non-reflective coatings, we proceeded to the general policy that optics for all optical instruments such as tank telescopes, binoculars and telescopes of all types for the field artillery sights and guns, be coated by that process. As we got into this business, our policy crystallized into something which was more or less conflicting. The first was the requirement that the film have maximum efficiency, the second that it have maximum durability. But durability seemed to decrease with efficiency. As so frequently happens in engineering matters these two requirements contradict one another to a certain extent, but the compromise as worked out by the Navy Yard is a very satisfactory one. We have therefore adopted as first preference, what we call the Navy hard-bake process, although you may know it by other names. That has not prevented us, however, when necessity drives us, to deviate from that first choice for other less effective coatings. I should emphasize that we deviate only when it is necessary to have something which we can't get at the time we must have it. In pursuance of these policies, we now have working for Army Ordnance a capacity of around six hundred thousand square inches of surface for an eight hour day with at least fifty percent already in immediate production. There are still loose ends to be tightened to make truly effective the production possibilities of this equipment, on organization, scheduling and more effective use from a technical

standpoint.

At this meeting today and tomorrow, we have brought together people who build and use all known types of apparatus for producing the preferred type of film. We hope to bring out and lay on the table all the troubles and difficulties and to bring to their attention any system, as well as all the things that any of you have found.

Mr. Peterson: The employment of vacuum procedure has beBausch & Lomb come more than a university curiosity. The
equipment in use at the university laboratory
was in most cases small and a long period of time was required
to pump out a system. With the advent of the high speed oil
diffusion pump however, this picture has changed. This new pump
when coupled with the mechanical forepump is capable of evacuating relatively large chambers to a working vacuum in short
periods of time. Various companies undertook the study of
vacuum work from the standpoint of the sale of these films. These
companies discussed their problems with the equipment manufacturers many times during the course of their work.

In our case, we were able to call upon Distillation Products. They were in Rochester and they gave us appreciated cooperation. I am sure that that is the way is has been with the others who have collaborated with the equipment manufacturers. We have the result of these efforts displayed before us today, the basis for equipment design being the experiences of shop practice.

**Dr. Lyon:** I believe the Navy was the first to really push this idea to any extent. It seems that except for oral discussions which might have taken place early in 1940, the initial impetus came from Dr. Hurlburt of the Naval Research Laboratory who attended a meeting of the American Physical Society and there listened to a couple of papers which were presented by Dr. Cartwright on the subject of low-

Naval Research Laboratory who attended a meeting of the American Physical Society and there listened to a couple of papers which were presented by Dr. Cartwright on the subject of low-reflecting films. He reported to the Bureau that this phenomenon should be investigated. After some general discussion, a directive appeared at the Naval Research Laboratory and Dr. J.A. Sanderson was assigned to work on this problem. Dr. Sanderson's report simply confirmed the fact that natural phenomena which had been observed to occur within a university laboratory would also occur in a government laboratory. This did start production people thinking and Captain Brown who was then Production Officer of the Navy Yard thought that there was a possibility of applying it to instruments on a large scale. In January 1941, I went to the Navy Yard through the efforts of one who was ac-

quainted with my work at M.I.T. on thin films. It appeared to be my job to put into production, something which hadn't been fully developed. A few coated binoculars had been sent out by the Naval Research Laboratory for test under service conditions and a report had just been received that they were satisfactory. They wanted all the binoculars treated as rapidly as possible. My first efforts, of course, were directed to making the films more durable, and I stumbled upon that old idea of heating the elements in a vacuum. From that time, and as we treated more binoculars, the word went around through the Bureau of the advantages which resulted from coating.

When I started on this job in January 1941, I made trips to National Research Corporation, Bausch & Lomb and Eastman Kodak. They were then in the process of putting on low reflective coatings but I recognized that more development was needed in order to obtain a hard film. The present equipment which we designed at the Naval Gun Factory follows closely the major features of the Bausch & Lomb, Eastman Kodak and National Research Corp. equipment, which were in use at that time, but with the added feature of a coil for heating the elements in the vacuum. The little crucible which some of you have seen at the Naval Gun Factory for holding the fluoride was an idea we borrowed from the Bausch & Lomb Co. At times we have been given credit but it should go to someone at Bausch & Lomb.

This rather modest beginning—there were only four companies in the business at that time—in conjunction with the aggressive advertising of the Navy after we had developed a durable film has culminated in this gathering today which encompasses fifty or more manufacturers, now engaged in this work.

Mr. R. S. Morse: It is now over three years since National ReNational Research Corp. search Corp. search started producing lowreflection coatings. During that period we have, in addition designed and built various types of coating equipment for our own use and for optical concerns. Although the equipment and techniques employed today are improved as compared with those common some five years ago when coatings were being made in the laboratory, there is still much to be learned. Coating on a production basis may still present many difficulties. Importance in design of equipment should be stressed. The problem before optical manufacturers today is that of producing low-reflection films on a production scale. Such a problem can be solved only by the use of equipment designed from a production man's point of view.

The possibility of operating continuous vacuum processes has

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been sufficiently explored to prove their practicability. At the present time, however, it appears that coating will probably be undertaken as a unit process. The most economical capacity per evaporating unit must in each instance be determined on a cost basis. As a compromise it is believed that an area equivalent to a diameter of 20" should be used.

In considering the design of equipment for a plant engaged in coating work the apparatus should be considered as a whole from an engineering point of view rather than in terms of a single unit. Assuming apparatus having comparable maintenance costs, increase in production can be obtained most easily by the reduction in time cycles of coating. It would seem that the greatest benefit along these lines could be obtained by reducing the present 30–60 minute cycle which is common with the single unit machines to perhaps ten minutes or less.

On the basis of our experience the shortest possible time cycle can be obtained through the use of "master roughing line type" of evaporation systems. A large mechanical pump, 100 c.f.m. or larger, is employed with suitable valves to rapidly "rough down" each of a series of evaporation systems. By such a system a pressure of less than 50 microns can easily be obtained in less than one to two minutes—if each individual unit is supplied with high speed oil diffusion pumps with appropriate valves between the diffusion pump and the evaporation system. The diffusion pump thus operates constantly with a low forepressure which is maintained by its own individual mechanical pump. Systems of this type have been successfully used for many other purposes, and not only permit much faster exhaust cycles, but eliminate many of the problems of maintenance particularly of the diffusion pump.

High vacuum technique has, until recently, been limited to the laboratory. Many of the ideas with regard to design of equipment have unfortunately suffered. It is high time that vacuum engineering should be dealt with from an industrial point of view and the ideas of wax joints, rubber tubes, glass stop cocks, etc., be eliminated. This problem of operating vacuum equipment 24 hours a day on the production line is an entirely different one from that encountered by the experimental physicist. Entire units should be of metal construction. Glass to metal seals, glass gauges, and all other such fragile items should be eliminated.

The production man has no time to look for leaks. It isn't enough to say that the apparatus will be designed simply so that the leaks may be found.. Leaks should never occur at all. The question of both the time cycle and the ultimate vacuum obtained is, of course, very largely dependent upon outgasing of the equip-

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ment. This is not only a function of cleanliness of the system from a maintenance point of view, but also the type of materials exposed to the low pressure. Lens coating equipment is not entirely fool-proof by any means and many things can render it inoperative particularly in the hands of inexperienced personnel. Through the use of proper controls many of the difficulties encountered in production can be eliminated. Apparatus should be supplied as completely instrumentated as possible to eliminate such difficulties as lack of cooling water in the diffusion pump, over-heating of pump oil, burning out of the ionization gages, etc.

# Vacuum Pumps



Mr. Behrndt:

Before discussing forepumps, I would like Distillation Products, Inc. to say that the evaporation method of coating is simple in its mechanism and applica-

tion. Only a relatively small amount of equipment is necessary to obtain successful coatings. The vacuum equipment manufacturer must put into the hands of the coating artist or technician an enclosed vessel which can be held at vacuums between 10<sup>-4</sup> and 10<sup>-5</sup> mm of Mercury. This degree of vacuum was arrived at because within these limits the mean free path of the evaporated molecules attain values which are greater than the dimensions of a coating unit. The term "mean free path" may be described as the average distance traveled by a molecule before collision with another molecule. Some idea of the magnitude of this term can be obtained from the data on the mean free path of Nitrogen at 0° C. versus variations in pressure.

Pressure in mm (Mercury)	Mean Free Path
760	$8.5 \times 10^{-6} \text{ cm}$
1	$6.5 \times 10^{-3} \text{ cm}$
10-3	6.5 cm
$10^{-4}$	65 cm
10-5	650 cm
$10^{-6}$	65 m
10-9	65000 m

From this data it can be seen that optimum operating pressures lie somewhere between 10-4 and 10-5. The method of expressing low pressures in mm of Mercury by the negative power of 10 is easy to understand if one considers that 10<sup>-1</sup> power is equivalent to 1 mm. Likewise, 10<sup>-3</sup> equals 1/1000 or .001 mm. It should be pointed out here that another term is used for expressing thousands of a mm, that term being the micron.

Because I have said that the evaporation method of applying coatings to optics or other surfaces is simple, you may have wondered why it has only been recently, that it has come into use. Its widespread use was delayed because of a lack of:

- 1. Heating technique such as use of the bare tungsten heater.
- 2. The adaptability of the process to non-metals.
- 3. And most important, the development of the high speed vacuum pumps.

In reference to the development of "1." of the heating technique, it might be mentioned that a type of filament must be used which itself will not evaporate.

Because pressures in the range of  $10^{-3}$  to  $10^{-5}$  mm are necessary for successful coatings, two types of vacuum pumps are required for attaining such pressures. One of the types of pumps which is necessary is known as the forepump, the roughing pump, or the mechanical pump. I shall try to describe the principle of operation of this pump by means of a diagram. In the #1 position, the plunger in the pump is starting its cycle which is counterclockwise. It can be seen that the port connected to the system to be evacuated is closed and the pump cylinder is full of gases from the previous cycle. In the second diagram, the plunger has reached the half-way mark in its cycle. The gases which were originally in the pump have been partially forced out, whereas gases from the system to be evacuated have entered the pump chamber because of the low pressure. In #3 the cycle is carried a little further and as the plunger completes its cycle, as in #4, the gases originally present in the pump have been completely eliminated and the pump cylinder is full of gases from the vacuum chamber. The discharge port is closed and the cycle is ready to start over again. The efficiency of the mechanical pump is maintained by oil seals around the pump cylinder and plunger and necessarily, of course, by the close machining of the parts.

There are many different types and kinds of mechanical pumps, but if the principle of operation just described is understood, the working of most pumps on the market today will be intelligible. The mechanical pump operates successfully over a pressure range from one atmosphere to as low as one tenth of a mm of Mercury. and the speed of mechanical pumps range from a fraction of a cubic foot to 1000 or more per minute. The speed depends upon the dimensions of the pumping chamber, and the operational speed and efficiency of a mechanical pump decreases sharply as the pressure drops. It is this lack of pumping speed in the lower pressures which necessitates the use of other kinds of pumps such as the diffusion pump. One other reason for making use of diffusion pumps is the fact that pressures lower than a few tenths of a mm of Mercury are not obtainable with the mechanical pump alone because of the vapor pressure of the oil used in the mechanical pump for the oil seal. A mechanical pump which is commonly used on vacuum coaters has a capacity of approximately twelve cubic feet per minute. This pump enables the operator to evacuate a leak-free system down to 25 to 100 microns in approximately five minutes. A faster pump could be used, but because the system is a closed one, a larger forepump is not deemed advisable or necessary.

**Mr. R. S. Morse:** The proper forepump to be used with an *National Research Corp.* evaporator is, of course, dependent upon the design of the equipment and the type of dif-

fusion pump employed. From an economic point of view I believe that generally speaking most of us have tended to use mechanical pumps of too small a size. In the case of units operated without a master roughing system where exhaust cycles of approximately 30 minutes are common, the size of the mechanical pump is not so critical. It is common practice to use a Cenco Hypervac 20 or a Kinney 5 x 5 pump. In the selection of a mechanical pump perhaps it is well to emphasize the fact that there is no real relationship whatsoever between the size of the mechanical pump and the pressure which will be theoretically obtained in a vacuum system of a given volume. In other words, the question of out-gasing of the diffusion pump and the evaporation system itself, particularly at the start of the evaporation cycle, is of far greater importance than merely the removal of given volume of air as determined by the physical dimension of the equipment.

Generally speaking, the cost of the mechanical pump is relatively small as compared with the rest of the equipment used in a lens coating plant. If by spending \$500 more for a mechanical pump the operating time cycle can be halved the investment is obviously worth while.

The question of maintenance of the mechanical pump is an important one and should be recognized in the beginning. If a diffusion pump is to operate most effectively in the high vacuum range, it is essential that the forepressure be maintained below 50 microns. Although the best mechanical pumps will blank off with a McLeod gage reading at 1 to 5 microns such pressures can never be obtained after a pump has been operating on an evaporator over a period of time. The pump oil becomes contaminated with water, decomposition products and dust, and gradually the exhaust cycle will increase. In our plant in Boston, for example, we have a central oil purification system which has long since paid for itself by the resulting savings in oil used by a large number of high capacity mechanical pumps. Although a complete vacuum system with a series of units using a master roughing line is recommended for production work, each evapo-

rator with its attendant holding pump should also be considered as a unit. Provisions should be made for easily removing each individual evaporator with its mechanical pump to a central maintenance room. In terms of production time, a rapid exhaust cycle must be maintained by proper care of equipment. The maintenance of vacuum apparatus is a necessary expense which should be recognized at the very start of operations.

**Mr. Wilson:** I would like to call attention to the fact that for the most successful engineering layout of a lens coating department, we find it very

desirable in the Perfex Corp. to use a roughing pump which directly enters the jar between the backing pump and the diffusion pump. For this purpose we have used as the roughing pump a four hundred cubic feet per minute Stokes pump, which includes the centrifugal oil purifiers and has so far operated three months without giving any trouble whatsoever. We are using the standard equipment, the Cenco vacuum pump and the diffusion pump of the type which is illustrated on the machines we have before us. I would like to add that we have placed a valve in the system, above the diffusion pumps. This valve makes it possible to close off the diffusion pump from the bell jar and allows the diffusion pump to operate continuously without interruption. It has proved very successful, to mount all of the pumps in a separate room. In our arrangement in the laboratory, we have placed a wall behind the lens coating assembly and behind this wall in a separate room which is ventilated by its own flues, we have placed the pumps which carry off any oil vapor which might be diffused into the atmosphere of that room from the operation of the pumps. The cycle of operation is greatly speeded up when the operator closes the bell jar, leaving the valve above the diffusion pump closed and turning on the roughing pump so that the pump goes into the jar directly. When the pressure has been brought down to the best possible value by the large roughing pump, which will occur within a very few minutes, then we operate the valve which is above the diffusion pump and allows the diffusion pump to take hold in the cycle of operations. In that way, we have shortened considerably, the operating time cycle. We owe to the research facilities of RCA a great measure of the success of this system, because we have used the design of valve above the diffusion pump which was developed by that engineering group. I wish to emphasize that when the diffusion pump oil seems to show an indication of being contaminated by a general slowing down of the process, by all means change the oil and if necessary, clean the system. (Editor's note: Mr. Wilson at this time was working with soft coating.)

**Mr. Behrndt:** If we now assume that the coating system un-Distillation Products der question has been brought down to 50 microns by the use of the forepump just described,

we can lower the pressure further by the operation of the diffusion pump. The principle of operation is relatively simple. Vapor from a heated boiler fluid is ejected from the jet and forms a partition between the intake chamber and the pump wall. The vapor coming in contact with the cool wall condenses and returns to the boiler as a liquid by gravity. Gas molecules in the intake chamber diffuse into the vapor partition and having but small chance of returning to the intake chamber, they are carried along by the vapor stream into the pump proper. Here the gases are compressed by molecular bombardment to pressures, sufficiently high to enable the forepump to remove them to atmosphere. Diffusion pumps operate over relatively small pressure ranges. They can compress gases from 10<sup>-6</sup> or 10<sup>-7</sup> to 10<sup>-2</sup> or 10<sup>-1</sup>. Speeds are attainable in diffusion pumps range, depending upon the size and construction, from a fraction of a liter to 10 to 15 thousand liters per second. It should be pointed out here that these speeds are calculated with the pressure attained with the pump and do not mean liters of atmospheric air (1 cc at atmosphere expands to 760 liters when the pressure is reduced 1 micron).

There are perhaps, hundreds of different types of diffusion pumps. Some are made to operate with mercury which was the first boiler fluid used. Mercury has been replaced by the newer fluids which, in general, are oily materials with the following properties:

- 1. Low vapor pressure
- 2. Stable to heat
- 3. Chemically inactive
- 4. Good boiling point
- 5. Having high molecular weight
- 6. Quick recovery cycle

By this I last property we mean that the boiler fluid is able to rid itself of the absorbed gases after it has been subjected to the atmospheric pressure. One type of diffusion pump used on the coater units is the Distillation Products' type MC-500 which has a speed of approximately 500 liters per second at  $10^{-4}$  and an ultimate vacuum of  $10^{-5}$  with pressure as low as  $4 \times 10^{-6}$  having been obtained without the use of cold traps. Another type of pump used is the Distillation Products' type MC-275 with a speed of approximately 275 liters per second at  $10^{-4}$  mm of Mercury and an ultimate vacuum of  $5 \times 10^{-6}$  at  $25^{\circ}$  C. when Octoil is used as a boiler fluid.

#### DISCUSSION

**Dr. Williams:** Perhaps some of you know that I have had familiarity with the production of thin films by evaporation procedures for the last nine or ten years. There are two or three points in connection with the use of pumps that I think haven't been adequately mentioned.

In the first place, I think we can all get the clear distinction between a forepump and a diffusion pump. A fast forepump is without question beneficial. The problem of outgasing, I think, should not be discussed particularly in connection with a forepump, because the pressure of fifty to a hundred microns is not a problem on a forepump. When one comes to a diffusion pump, one should realize that the name diffusion is a name which aptly describes a diffusion pump. A diffusion pump will pump only as fast as the molecules of air and other gaseous materials in the vacuum system, elect to come over to the pump and let themselves be pumped. You can't hurry up nature in connection with a diffusion pump. If the molecules won't go there, you can't pump them out. Therefore you want a pump which in general (I think these remarks are quite correct) is in close proximity to the vacuum chamber. You want the opening from the vacuum chamber to the pump to be as large as the opening in the pump or even larger if possible, and in general you want a straight line to exist between the vacuum chamber and the actual inside of pump.

Now, as far as pumping down the chamber from the pressure produced by the forepump to evaporating pressure is concerned. a fast diffusion pump is doubtless desirable on the whole, though certainly the diffusion pumps made by Distillation Products, as has been pointed out, are amply fast to take care of the air as it diffuses from the vacuum chamber into the pump. The pump can take it out as fast as it comes in at these low pressures. However, that would indicate that perhaps there is no particular advantage in having let's say, a really fast diffusion pump as compared to a slow pump. I am thinking in terms of one aspect which wasn't mentioned on the diffusion pump, and that is, that as soon as you start evaporating, you are evaporating molecules from the magnesium fluoride to the wall of the chamber. These residual gases are outgased at the wall of the chamber and your pressure will instantly and fortunately momentarily increase, but during this time when it is increased, one can obtain soft coatings of magnesium fluoride. Therefore you want to pump that gas fast in order to keep the pressure low at that critical time when the magnesium fluoride is fast distilling on to the walls of the chamber.

Another point I think I might mention is this: the worst enemy of high vacuum work is, without question, water. Water vapor is the villain. If we are to have a system that will pump out in a minimum of time and cause minimum trouble when the magnesium fluoride is evaporating, we must keep it operating as much as possible, and above all things, keep it as dry as possible. If it is to set idle for a week or so, put a drying agent in it, and keep it dry. But it is best to keep it in use. If you can't keep it in use for a couple of hours, keep it pumped out. It has been my experience over a number of years, that a vacuum chamber when set aside for as little a time as three or four hours, will not produce a film comparable in quality to that of a vacuum chamber used ten minutes ago, and that, in my experience, is the most important point of all. They must be kept dry and in use.

#### Vacuum Valves



Mr. Morse:

In connection with the design of proper high National Research Corp. vacuum valves we believe that here again equipment should be designed from an in-

dustrial point of view. Laboratory gadgets such as stop cocks, pinch clamps, etc., are impractical and not of the rugged construction necessary for plant operation. There are two general types of valves: (a) Valves for use in forepressure lines with high vacuum on both sides and (b) Valves for use between atmospheric pressure and extremely high vacuum. Where extremely low pressures are not encountered and where leakage through the valve is secondary in importance to leakage into the valve from the outside, a number of standard types are available. In the magnesium industry, for example, Merco Nordstrom lubricated plug valves have been quite successful in pipe size up to 6". Several rubber diaphragm valves of the Saunders type are also manufactured. The rubber diaphragm valves are employed both to seal the valve from atmospheric pressure and to make the valve seat. Although valves of this general type, where rubber is used under tension, are not recommended for extremely high vacuum work, they are quite satisfactory for use in forelines.

Where valves are to be used under extremely high vacuum, that is pressures in the range of 10<sup>-4</sup> to 10<sup>-7</sup> mm some type of packless or bellows valve with a synthetic rubber type seat is preferred. Through the use of flexible bellows, motion may be introduced into the valve in a variety of ways thus insuring the complete absence of atmospheric air leakage. Through the use of appropriate seal materials the question of line leakage can be eliminated. In the case of 16" valves for example, combined outgasing and leakage rates of less than 10 microns per hour are common with one side of the valve exposed to atmospheric pressure. Properly designed packed stem or rubber seat slide valves are perfectly satisfactory for high vacuum work providing the outgasing problem is solved by the use of appropriate materials. Such valves are somewhat simpler to construct than the bellows type unit and can be serviced readily.

Mr. Behrndt: The valves used on the system are few. There Distillation Products is a solenoid valve in the oil line on the mechanical pump. Its presence prevents the sealing oil from flooding the chamber and thus making the pump difficult or impossible to start. It also minimizes the danger of flooding the diffusion pumps with the mechanical oil.

Another valve used on the system is the forepressure valve located between the forepump and the rest of the system. Of the two types of valves used, one affects the seal by means of a single rubber diaphragm and the other employs a flexible bellows to seal the valve stem to the atmosphere and a conventional seal for closure. The purpose of the forepressure valve is two-fold:

- 1. It enables the operator to test the efficiency of the forepump alone.
- 2. It enables the operator to test the tightness of the system. The forepump can be shut off, and the pressure rise in the system can be observed.

Mr. Pettus: Mr. Wilson made reference to a particular valve which we at RCA Victor have employed for the control of the oil diffusion pump. I would

like to discuss that more in detail. In order to do that I would like to refer to an appropriate diagram showing the nucleus of a lens coating vacuum system. At the upper portion of this diagram we see the vacuum chamber composed of a bell jar setting on a header plate or steel base, and the first seal becomes evident at the junction of the bell jar and base plate. At a section of the header plate we see a valve mounted above the oil diffusion pump port which has been referred to previously. It is shown in the open position. The diameter of the valve plate is 5½" and opens 1" above the opening of the header plate. Although this does not permit a mean free path to the oil pump, it does permit a large area for exhausting the gases. Probably the most important part of such a valve is that of mechanical linkage to the external control. Sylphon bellows of the metal type were originally used, but maintenance problems arose with increased production due to work hardening of the bellows where such operation requires motion in two directions. To combat problem an investigation was made for a suitable rubber bellows or diaphragm, and the use of a Lord shock mount was found satisfactory. Let us go into detail as to how this seal is formed. Between the header plate and the high-vacuum flange of the oil diffusion pump an additional cylinder has been placed having external flanges at each end for attaching to the header plate and oil diffusion pump. On the side of this cylinder, another flanged bushing is attached

which mounts the rubber shock with a rubber gasket between the shock flange and the bushing. Linkage to the valve plate is made, thru the metal insert of the shock mounting and a seal for the linkage is obtained by using a cup washer, somewhat larger in diameter than the insert, soldered to the shaft on the inner side and second cup washer secured to the shaft with a nut on the outside. The seal is obtained by allowing the cups to squeeze against the rubber diaphragm. Additional external linkage is coupled to the shaft and terminated on the control panel. Such a diaphragm permits good flexibility with sufficient motion for operating the valve mechanism. I am happy to report that more than a year's service has been given with all of our seals without a single leak.

In the operation of such a system the poppet valve would normally be closed when the oil diffusion pump is not in use, however the pump is kept ready for use at all times and brought into operation by opening the poppet valve. Our system employs the use of a large roughing pump (50 cubic feet Stokes) for the initial pumping cycle which allows lowering the pressure of an 18" jar 21" high to approximately 18 to 20 microns in 3½ minutes. At this point the valve in the roughing line is closed and the oil diffusion and hi-vacuum mechanical pump put into operation by opening the poppet valve. This allows a total pumping time of approximately 15 minutes to a pressure of 0.2 microns.

# Arrangement of Optics Within the Vacuum Chamber



**Mr. Mattern:** *Minneapolis-Honeywell* 

Dr. Robley Williams at the University of Michigan and we at Minneapolis, have deviated from the usual method of holding the

glass either in a horizontal position in the upper or the lower part of the vacuum chamber. We have designed our holding equipment in the form of a cylindrically shaped supporting rack approximately  $16^{\prime\prime}$  in diameter by  $18^{\prime\prime}$  high. Around its outer perimeter this rack supports vertically 12 lens holding plates. These holder plates are made of polished chromium steel and accurately locate the glass surfaces during the filming process. The glass is placed in these holder plates (which have an active glass holding area of about 50 square inches each) the plates hung on the holder rack, the bell jar lowered onto the base plate and the pressure reduced to approximately  $5 \times 10^{-4}$  mm. As you will note, these holders have been so designed as to hold the two components of a doublet in the same plane and adjacent to each other, thus avoiding mixing them during filming.

The glass is then heated in the bell jar previous to and during the filming operation using electrically heated coiled tungsten elements. These heaters are supported vertically at each end by ceramic insulators spaced uniformly at 12 points around the inner perimeter of the holder rack. They are approximately 17'' long by  $\frac{1}{4}''$  diameter and are spaced inward from the vertical edges of the holder plates, about  $1\frac{1}{4}''$ . The temperature of the heaters is maintained at approximately  $1200^{\circ}$  F. throughout the filming operation.

By this means the temperature of the glass to be coated is brought to  $450^{\circ}$  F. as measured by an iron-constant thermocouple imbedded in a ceramic placed near the floor of the base plate. Obviously this thermocouple does not register the actual temperature of the glass but is calibrated through its ratio factor against a thermocouple which had previously been placed in contact with the heated glass.

The fluoride evaporator filaments consist of conically shaped, helically wound coils of .020'' diameter tungsten wire cut to a length of 12'' and having a cold resistance of .091 ohms. Three of these evaporator coils are used for each bell jar. They are placed

in a parallel electrical circuit, making it important that their individual resistances be held as uniform as possible. They are accurately spaced on the vertical axis of the lens holder rack and held in place by clamps attached to copper rods. The lower end of the bottom coil is placed exactly  $2^{\prime\prime}$  above the base plate, the middle coil  $9\frac{1}{2}$  above the base plate, and the upper coil  $17^{\prime\prime}$  above the base plate.

Into these conically shaped coils are placed the press formed fluoride pellets using a specially designed pellet loader that deposits the proper pellet in each coil. These pellets are formed by dry pressing accurately weighed quantities of very finely powdered fluoride, in a hardened steel die. After molding, the pellets are carefully placed in the pellet loader, care being exercised to avoid loss of powder. During the filming operation these pellets are completely evaporated. Obviously, it is very important to have as pure a magnesium fluoride as it is possible to produce. If the fluoride has an impurity of either magnesium oxide or magnesium oxyfluoride it is detectable by a selective evaporation. This exhibits itself by a portion of the fluoride evaporating and then, due to the higher evaporation temperature of magnesium oxide, requiring that the temperature of the filament be raised considerably in order to completely evaporate all of the material.

In order to provide a uniform distribution of fluoride over the glass surfaces held in the lens holder it is necessary to accurately calculate the proper weight of fluoride for each filament. In calculating the individual fluoride pellet weights advantage is taken of the fact that propagated fluoride vapor in a vacuum follows generally the laws of light. Therefore the equation used for these calculations is:

$$Tp = \frac{M}{4 \pi DR^{2}} Cos \Theta \cdot N_{f.}$$

Tp = Optical thickness of the film in millimeters at any selected point on the lens holders.

M =the weight of the fluoride in milligrams.

D =the density of the evaporated film.

R = the distance from the evaporation filament to the point P in millimeters.

Q = the angle between a line normal to the coated surface at point P and a line between the evaporation filament and point P.

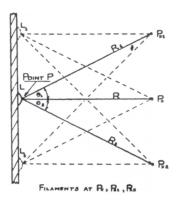
 $N_f$  = the index of refraction of the coated film.

In using this equation I have found that for the pressure and temperature we maintain in the vacuum chamber, a density of 2.7 and an index of refraction of 1.35 are about right for the fluoride film.

As we use three evaporation filaments, it follows that in calcu-

lating the film thickness for any point on the lens holder, this will be the sum of optical thicknesses produced by the individual fluoride pellets, held in the three filaments. To illustrate I shall

show a distribution diagram: In this diagram, points L represent TP in the equation, while P represents the weight of the individual pellets or M in the equation. In actual practice the weight of the lower pellets needs to be somewhat greater than the upper pellet. This is doubtless due to the filament supporting this pellet being directly over the throat of the pump, so that during the evaporating period some of the fluoride is robbed by the pump and does not reach the glass surfaces.



Actually the weights of the individual pellets for an optical thickness of film about 115 millimicrons (which is about right for a minimum reflectance in the near blue) are .082 gram for the upper pellet, .047 gram the center, and .089 for the lower pellet.

In order to assure success with this process it is necessary to control pressure not merely as low as it is possible to go, as I do not think this is necessary, but to as low as is necessary to obtain a satisfactorily hard coat and at the same time not sacrifice film efficiency by evaporating at too low a pressure. By this I mean to hold the density of the evaporated fluoride as low as possible and still have sufficient hardness to meet all of the specifications that have been set up for these films. We have found that by holding the pressure between 3 and 5 x  $10^{-4}$ , and maintaining the temperature of the glass at  $450^{\circ}$  F. it is possible to meet all the requirements, such as distilled water test, salt atmosphere, hardness and others with which you are all familiar.

Another important requirement in this process is to follow a uniform evaporation cycle. During the evacuating and glass heating period, when the glass has reached 380° F. (indicated by the thermocouple), the evaporating filaments are brought to a dull red heat and held at this temperature until the glass has attained a temperature of 430° F. This baking period is important in order to drive off any last trace of water vapor from the fluoride and eliminate any danger of spattering.

By following this procedure I can say that we do not experience any trouble due to spattering of fluoride. The temperature of the filaments during this preliminary baking period is controlled by a large Variac and an ammeter holding the current at 20 am-

peres. When the temperature of the glass reaches 450° F. the filament current is raised to 37.5 amperes which brings the temperature to the melting point of the fluoride. This molten fluoride flows to the lower portions of the conically shaped filaments. At this time several of the upper turns of the filament become incandescent due to their not being in contact with the molten fluoride. The majority of the evaporation takes place at these upper turns, the time required being about three minutes. Finally to assure complete evaporation of the fluoride pellets the current is raised to 50 amperes and held for 1 minute. The filaments are then individually observed, using a pair of dark blue goggles, to make certain that all of the fluoride is evaporated.

Following the evaporation, the heaters and pumps are turned off, the temperature of the glass allowed to drop to  $280^{\circ}$  F. and then filtered air is allowed to slowly enter the vacuum chamber.

#### DISCUSSION

**Mr. Morse:** First of all, I believe we would like to say from our experience in the use of the heater element we have discovered that when a tungsten wire is used as a source of radiant heat for bringing the lenses to temperature within the jar is left in its high metallic luster, the distribution of temperature over the elements is sometimes poor. That distribution will improve when the actual heater element is very blackened, either due to deposits on the surface of magnesium fluoride, or due to a natural process evolved by the element after being heated for a long time. This is a very important thing to bring at this time to the attention of everyone. If you do not have even distribution of heat on the lens surfaces within the jar, you might do well to attempt to make several evaporation cycles until the heating elements have been coated uniformly with the magnesium deposit, which eventually occurs upon them.

**Mr. Mattern:** We use about ten evaporation cycles before we put the jar into actual service. The proper condition of the heaters can be determined by a reduction of the voltage required for a given current density in the heating element. In other words, 132 volts are required to obtain 25 ampere current density in a bright tungsten heater. However, after the tungsten heater has been dulled or fluoridized with a film of magnesium fluoride only 110-115 volts are required for the same current density.

**Mr. Kahn:** I have a problem with my filament and perhaps one of you gentlemen may have the answer for me. I have been advised by some to place the filament above the fluoride and others to

place the fluoride on the filament. When the fluoride was placed on the filament the filament burned out. Can anyone advise me as to the correct position of the filament with respect to the fluoride?

**Dr. Lyon:** The only reason for putting the filament above the fluoride was our experience that little particles of fluoride would at times, melt around the wire and the filaments would burn out just as you say. By placing it above the surface, it did not strike the fluoride when it was heated and the filament would last much longer. That was our experience at the Naval Gun Factory. There are people here who have actually imbedded the tungsten wire in the fluoride. I don't know what the results have been. Our filaments very seldom burn out. They are usually destroyed because someone hits them, causing breakage. I don't see any reason why tungsten filament should not last a long time, provided care is taken to prevent somebody from hitting it, since tungsten becomes very brittle after heating to a high temperature.

As regards holding fixtures, I wish to bring out that before any manufacturer decides to adopt any particular method of holding the elements, he should know definitely whether he is to coat a large quantity of a limited number of sizes or whether he must deal with a relatively small number of many different sizes. In the latter case, it will be advisable for him to use a holder which is versatile and which can be adapted quickly and conveniently to the many different sizes with which he must work. A dome shaped holder divided into segments lends itself admirably to this end.

**Dr. Williams:** I wanted to mention the reason for using the M-H<sup>1</sup> system. I don't think Mr. Mattern made that clear enough. Number one, you heat from the front and that is just where you want the heat. It's the front of the glass you coat. If you heat it from the back you must draw the heat along to the front. You can see a difference in heating it from the back and from the front. As he mentioned, no one looks at the coating while it is being coated. Weighing the fluoride on the balance before it is ever used determines the film thickness so no one has to observe whether the color of the optic is purple or red. The question of relative areas is simply this: if you take the perimeter of such a bell jar and check the area, you have about 600 to 650 square inches available for coating, if your arrangement is vertical. If you take the area of the dome, it comes out around 250 square inches. If you want to put your lenses on the dome, that is alright. If you want to make jigs and hang them on the wall, that is alright. In any event, you have to punch holes, of course, and mount your lenses in some sort of a jig outfit. Those are three primary advantages of the method.

**Mr. Mattern:** That obviously would have to be worked out by <sup>1</sup>Minneapolis-Honeywell Reg. Co.

each individual for his particular requirement.

**Mr. Denton:** I would like to ask you this question. The thickness of coating depends on all the fluoride which you put in the jar getting to the glass surfaces and you brought out the fact that the vacuum must be carefully maintained. In addition to that, I imagine that you could also lose the fluoride from a spiral coil of this sort by the fluoride spilling out between the wires. That would be a way to lose fluoride which will never reach the surface. Have you ever experienced any such difficulty?

**Mr. Mattern:** One of the reasons we include the bake period was to avoid that very thing. Most of the loss of fluoride from the filament is due to absorbed moisture, causing an explosion of the pellet. If the fluoride is melted rapidly this will cause spatter, and we are all familiar with that. The reason we are using: the bake method is to avoid spatter, and we actually have very little of it. Perhaps over a period of two or three days, a small quantity of fluoride is detectable on the top of the pump cover, but not enough to affect the film thickness noticeably.

**Mr. Denton:** How long have you been doing production in that way?

**Mr. Mattern:** With this method—about seven to eight months.

**Mr. Denton:** And you have been able, in the majority of cases, to control the thickness tolerance during that period?

**Mr. Mattern:** Oh yes. If for instance, instead of the minimum reflectance at 4600 angstroms a minimum is required at 4800, about five percent more fluoride is used for each pellet.

**Mr. Berlant:** May I ask Mr. Mattern if he has any idea of what his average rejections are for poor color?

**Mr. Mattern:** Well, I haven't any figures with me, but I would say for poor color it is less than 3 percent. Most of the coatings we are producing we attempt to hold the minimum reflectance between 4600 and 5000 angstroms. We have been asked by one of the military groups to hold it at the low end of the spectrum, and we are doing it.

Our system is not limited to this minimum. We can coat to any mean minimum desired by varying the weight of the fluoride in the pellets.

**Mr. Heacock:** At our plant, we have a different method of holding the glass in the fixtures. Dr. Lyon puts them in the position

of a sphere. Mr. Mattern as he explains, has them around the wall. We ourselves take a perfectly flat plate; we mount the lenses In that and coat from below one side of the lens, then reverse the whole plate with all of the lenses in it and without changing our vacuum, we coat the reverse side of the lens. That seems to be basically different from either of the other methods that have been described. Up to the present time, most of our production has been on what we call low temperature bake. We bake at  $45^{\circ}$ C. after the glass has been removed from the bell jar. At the present time, we are changing over our equipment so that we will be able to supply what we all refer to as high temperature bake—magnesium fluoride, but we still plan to use a flat plate. If we use high temperature bake magnesium fluoride we contemplate taking both of the elements of the doublet and put them one above the other and coat one side, turn it over and coat the other, although up to the present time, that is only a thought, because we have not done that in production with high temperature bake. (Editor's note: Special equipment is needed where the flat plate is used.)

**Mr. Liebman:** I would like to ask both gentlemen one question. Did you find a variation in the coloring from one end of the plate to another on the flat plate or on the side running up?

**Mr. Mattern:** Yes we do, but the variation is slight. We experience very little variation in the thickness from top to bottom of the holder. I cannot answer your second question specifically, unless I know the type of lens being coated. If it has a severe curvature, some difference in color will be found between the top of the curve and the edge. Obviously we have all experienced that on an individual lens. (Directed to Mr. Heacock) I would say that except for the compensation we have made by putting in three sources of fluoride as against one on your flat plate method, we are both doing about the same thing. You are coating from a single source to a horizontally held flat plate, and from our experience and from what Dr. Lyon has just said—you must get a variation in color between the glass in the center of that plate and the outer edge. You certainly must do that in your case.

**Mr. Heacock:** That is correct, except we have a compensating plate which is rotatively driven between the source of fluoride and the surfaces to be coated to correct that. Suppose we are making a seven inch diameter stock coating all on one piece of glass, if you have a point source here without the compensating plate your statement is correct. We would get too much fluoride in the center and not enough at the edge, so we have introduced this magneti-

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cally driven compensating plate which takes care of that with the result that we do get a uniform color from center to edge. Without this plate we would encounter the difficulties mentioned.

**Mr. Mattern:** We did it a little differently. We do get a uniform color from end to end of the holder. That requires careful control of fluoride weights, but we restrict the active width of the glass area of each holder plate to  $2\sqrt[5]{8}$ . I think this system has a much greater area. If you calculate it you will find that for  $7\sqrt[4]{2}$  distance between the evaporation filament and glass, the difference between the edge and the center is about one-tenths of an inch, and with that small difference (getting back to our equation), there is not enough difference to give any trouble at all. We do not experience any trouble from this source.

**Mr. Liebman:** I think I should bring out one point. In our system you can eliminate all that by using the dome.

**Capt. Heroman:** I think Mr. Denton should describe the racks used at the Arsenal.

**Mr. Denton:** Well, I don't think our method is anything unique. We have plagiarized almost everything we use from other manufacturers. The men at RCA were kind enough to show us how to use their compensator with a flat rack. The flat rack is useful because it can be made adjustable. The Arsenal probably has more different sizes of lenses per unit of production than any optical shop represented here; probably 300 different lens sizes are made at the Arsenal. Our rack consists of a ring about 16" in diameter and 1" wide which has two slots in it. Each slot is 1/4" wide and covers a 120° angle and the slots are diametrically opposite to each other. Stringers are used to cross the ring parallel to one another. There are shelves on the stringer so that the lenses can be mounted in railroad fashion. Each stringer has slotted ends so that it may be bolted to the ring at each end; by using four different lengths of stringers, we can cover the whole range of the 120° slot, so we can put in a bunch of two or three inch lenses at one time and the next time in the same rack, put in a bunch of 34" lenses or for that matter, put both sizes in simultaneously. If anyone is interested, I will be glad to send them drawings. This type of rack is mainly useful because of its flexibility and comes in handy with our experimental instruments—where we don't want to wait for the machine work necessary to make special jigs.

# Vacuum Gages



Mr. Wilson:

Perfex Corporation

There are several types of gages that can be considered of possible use, but there are some types which would be a waste of time

to make. If one uses the auxiliary Kinney pump and auxiliary roughing pump on a system which works independently and completely through its own individual manifold, it desirable to check occasionally the pressure which that pump is creating. We have found that it is very valuable to place a thermocouple gage made by Central Scientific Co. or the Stokes Manufacturing Co. for the occasional check of the auxiliary roughing pump in the high vacuum system. There are usually two types of gages used—one of them must be very rugged and designed so that it does not burn out if used at times when the pressure within the jar is high enough to cause oxidation and subsequent burning out. Two types of gages which seem to be robust enough to stand this are the Pirani gage and the thermocouple gage. We have found that both of these gages, manufactured by the two facilities which we have mentioned here are dependable and if we desire to check their performance at any time, we maintain a separate reference ionization gage which has been calibrated for us by the General Electric X-Ray Corp. in Chicago. For the determination of very low pressures within the jar, a determination which is made when the operator is satisfied that the pressure within the jar is low, and that there is no danger of burning out—the gage which is most often used for that is the ionization gage. We have two types of ionization gages represented on the instrument we see here, and we have had experience in our plant with both of them. With a careful check as to calibration, we found that both gages used here are dependable. One of the difficulties encountered in this ionization gage is the burning out of the ionization elements. The ideal type of gage is that which is completely assembled within the jar consisting of all metal parts. Eventually we can look forward to the possibility of using that type gage. Most of our ionization gages are enclosed in glass, and are attached to the vacuum system by means of some auxiliary tube which extends from the system itself. The location of the gage is of tremendous importance as is the aperture of the gage. The degasing of the gage will be in accordance with the degasing of the jar. One possible practical idea would be to have the gage element locked within the jar, by constructing an over-lying table within the jar for supporting the mechanism and placing the gage element below this table so that it is removed from mechanical danger. That would be the ideal situation. We have experienced this in our plant, and at the present time are looking forward to the addition of a gage which will be all metal, directly included underneath the supporting place within the vacuum jar.

**Mr. Mueller:** We have found that the safest place for the ionization gage in the two way valve system we are using, is right in the diffusion line, so that it is valved off from the atmosphere at all times. There is no chance of turning on a gage then to the atmosphere or burning out the filament.

**Mr. Wilson:** May I say that is also possible, I believe Mr. Mueller must use a system whereby you shut off the diffusion pump from the jar when you have opened the jar. You are using an auxiliary system. It is only possible when the jar is operated independently of the diffusion pump by means of a valve inserted at this point here, or directly below it. The location is over the diffusion pump.

Mr. Behrndt: I would like to describe the gages used on our units. Two types are necessary because of the extent of the pressure range covered. The Pirani gage measures pressures between 750 microns and 1 micron. This gage, named after its inventor, consists of a fine filament sealed into a glass tube which can 'be connected to the system under test. A small current is passed through the filament tending to heat it. Gas present in the tube tends to cool the filament by conduction and is a function of the amount of gas present or the pressure. The lower the pressure, less heat is transported away by conduction, the higher the temperature of the filament, and the higher its resistance. Thus if a means is provided for measuring the resistance, a measure of the pressure is possible. A means is provided in the form of a bridge circuit. One refinement of this gage is the mounting of a similar tube in an adjacent arm of the bridge. The use of the auxiliary bulb serves to make the gage sensitive to variations in room temperature. The Pirani gage must initially be calibrated with another gage, the McLeod.

The other gage used on the system is an ionization gage which measures pressures from  $10^{-3}$  to  $10^{-8}$ . This gage is a three-element glass tube similar to a conventional radio tube. The elements are:

- 1. Filament which emits electrons when brought to a white heat.
- 2. The grid, a large open spiral on which a positive voltage is main-

- tained so that the electrons from the filament are attracted to it. In streaming toward the grid, the electrons collide with the residual gas molecules causing dissociation and the formation of positive ions.
- 3. The collector, kept at a negative voltage so the positive ions formed in the collisions are collected and passed on to the measuring device (the control circuit).

When conditions of the filament current, grid voltage, and collector voltage are constant, the number of positive ions formed in unit time is proportional to the amount of gas present (the pressure). A sensitive galvanometer for measuring the positive ion current will act as a pressure measuring device. The DP-100 gage manufactured by Distillation Products is such a device. The range of the DP-100 is  $10^{-3}$  to  $10^{-6}$ .

**Dr. Williams:** Has anyone ever tried making a Knudsen gage? Fundamentally the Knudsen gage should eliminate the difficulties, if you could make one that would be rugged. It is a type of gage that acts on the pressure exerted by molecules in a partial vacuum; and the air in there is put into motion by heat. It doesn't have a hot filament. It gives the direct reading on scale and is sensitive at low pressures. I made one years ago, but it wasn't rugged enough to consider under these conditions. I was going to suggest to the companies that they might, if are interested, like to look that up in the Scientific Instrument Review of a few weeks ago.

**Mr. Zack:** The Vard Co. of Pasadena, California is using them and produce themselves.

# Operation and Maintenance of Equipment



**Mr. Pettus:** *RCA* 

In all types of production work, the words operation and maintenance are very close blood relations. High volume production therefore

requires good maintenance on a schematic schedule. We have inaugurated a maintenance policy whereby the maintenance men come on duty thirty minutes prior to the regular shift. This permits starting the equipment and giving it a preliminary pumping cycle before going into production service. Since the first pumping cycle of an equipment left idle for a period of time is usually longer than normal, the preliminary pumping avoids a loss of operation time. A cleaning operation is also employed regularly. At the end of the first shift, the bell jars are removed and cleaned by washing, and as much of the vacuum chamber equipment as can be removed easily is also cleaned. This procedure allows the second shift to operate with approximately the same efficiency as the first shift. At the end of the second shift a complete cleaning of all vacuum systems is made and the equipment restored to normal for the following day. This plan works efficiently for a two shift per day operation, and any relaxation from this schedule usually shows a decrease in production. It is also necessary to provide some maintenance personnel on hand for all regular operating shifts if long breakdowns are to be avoided.

In such a system, ionic bombardment is used for outgasing the glass as well as a general clean up of the system. The ionization cycle is employed after the roughing has been completed. This permits a good discharge (using 3000 volts) without injury to the rubber gaskets on the electrical connectors which might result from ionizing at higher pressures and at the same time does not impose a heavy current drain on the high voltage transformer. The ionizing cycle is two to three minutes and is not generally carried to a dark vacuum. The electrodes for ionizing are made from magnesium strips and should be cleaned with steel wool daily.

I wish to make a few remarks regarding roughing pumps. In the use of the Stokes pump as we have employed them, a condition has been found that if the pump motor stops or the magnetic starting switch opens due to current interruptions and if the pump is connected to the vacuum chamber at the time, oil from the pump's crankcase may flow through the manifold and reach the vacuum chamber. This causes a serious repair and

cleaning operation, since all lines and associated equipment must be removed and cleaned thoroughly. To prevent such a mishap, we found that the oil distribution system on the Stokes pump could be provided with a solenoid valve operating normally open and connected to the motor starting switch. Should the circuit open, the solenoid closes and prohibits the oil flow from the crankcase through the system. To prevent a transfer of the small quantity of oil in the pump cylinder, it is best to provide an oil trap in the manifold line which can be drained occasionally.

A few words might be added regarding other vacuum tight valves. A rather simple valve may be made with a pinch-clamp on a section of vacuum rubber hose and has been employed for some time, but due to present day conditions, the quality of rubber hose is not satisfactory for the purpose. It has been found that repeated clamping caused a constriction to develop in the hose thereby reducing the overall efficiency. A good mechanical valve is now available on the market manufactured and is by the Kinney Pump Co. This valve uses a bellows for forming a flexible seal with the gate. Maintenance problems are not excessive with this device, and replacement of defective parts is not difficult.

There have been several references made to ionization gages and the subject is probably of great interest to us. Apparently the chief trouble with ionization gages is that of short life. We being radio tube manufacturers, believe the ionization gage is a useful instrument and like to use it since we have a suitable tube of a standard type as well. We have adapted the use of the type 210 triode which has a nickel plate and a sturdy tungsten filament. It is only necessary to use the element of the tube by removing the envelope, base, and attaching the element lead wires to suitable electrical connections at the base of the vacuum chamber. This gage is operated by supplying sufficient filament potential to cause the grid to draw 5 milliamperes and reading the plate current in microamperes which in turn are translated into microns. The gage and electrical circuit are previously calibrated from a McLeod gage. One of the most important factors in using an ionization gage is that of preventing the gage from being turned on when the vacuum chamber is at too high a pressure. One method of control is the use of the mechanical inter-locking switches operating from some part of the pumping equipment or on a time pumping cycle, but the latter is dependent upon consistent pumping speeds. It is well to point out that when using a gage of this type for the first time that it is necessary to operate it at above normal filament potential for 30 minutes or so in order to clean the filament and free it of gases. We have used such a device for several years and find the useful life for a single

gage to be several months of service.

In concluding these remarks, I would again like to repeat that good maintenance and good operation will always lead to maximum efficiency, and such a goal can best be reached when these factors are under control.

**Dr. Bateson:** Although the two speakers have covered the subject of pump valves completely, I want to add a few personal observations that we

have made in the two and a half years we have been performing evaporation work at Research Enterprise in Canada. I am in hearty agreement with Mr. Morse on the difference between the speed of a roughing pump under ideal conditions and over a period of time. One only finds the pitfalls by working with these pumps over a year or two.

The practical speed of a pump depends essentially upon the kind of pump. In a Kinney pump which uses oil as a sealing medium, you have probably noticed that on Monday morning, the pumps are very often slow and over a period of time your vacuum system instead of getting better, gradually gets worse. There are two kinds of vacuum systems; those which get progressively worse and those which grow progressively better. Those that grow progressively better present much less of a maintenance problem. We found that the vapor in the pump – the Kinney pump – is the chief cause of gradual increase in pressure, which normally would be expected to go down to 60 to 75 microns in about five minutes time. The pump, after a weekend or during a period of dampness, in spite of an air-conditioning system, would refuse to pump below 200 microns. We took the pump apart, drained out the oil, put some in a test tube and observed the emulsion. Then we put the oil under a vacuum pump and immediately the slight amount of water vapor was removed and the pump operated normally. We then went over to the method of treating the oil with heat and a vacuum, and in this way we were able to keep the pumps in operation. Meanwhile, we installed a complete air-conditioning unit which would maintain a relative humidity of about 30 percent and the Kinney pumps normally operate as well on Monday morning as they did at the close of production on Saturday. What actually happens is that the waste is drawn through the pump from the system and forms an emulsion which will not precipitate. I have mentioned this point in case anyone has run into the same trouble and wonders why their pumps are slowly getting worse.

The next point I would like to mention would be this question of ionic bombardment. We have used ionic bombardment in pre-

paring surfaces for both the metallic and fluoride coatings. We bombard during rough pumping after pressure has reached the strength of a hundred to 150 microns and if possible as long as we see a rise in pressure on the Pirani gage. There has been a good deal said for and against ionic bombardment. It certainly is not a cure-all, but it is an excellent insurance against poor pressure.

We have found the use of infra red lamp a great help in drying glass after cleaning. We have large baking lamps under which the glass is placed in the air conditioned rooms after cleaning. This seems to have the advantage of removing absorbed waste vapors on the surface of the glass, resulting in a better pumping speed in the bell jar. We find it fairly good practice.

I can't add anything on the question of valves. It has been covered very- well. I am in complete agreement with the men who are working on a continuous system. Were we able to redesign and build a lot of our equipment, I am sure it would be to convert them to the continuously running system. However, we have a war to win, and we have glass to get out, and we will have to do it with the equipment we have on hand. We have no time to revamp our equipment to make it continuously operating. Any new units going in are to be equipped, I hope, with those refinements.

The question of ionization gages is quite near to my heart. We break some of them. We use Distillation Products Sylvania ion gages and an ion control box. However, we have a little system for replacing gages in a hurry. The gage is located in a tube leading into the diffusion pump and mounted with a small rubber seal so arranged in conjunction with 2 brass inserts as to keep the exposed amount of rubber to a minimum. The whole assembly is held in place by a single nut and the seal maintained by keeping the rubber under pressure. When a gage burns out the operator can quickly unscrew the nut, take out the gage and insert another one which has previously been made up.

We haven't had any trouble with outgasing; it reads quite well and stands up over a period of time. The chief mortality in gages has been that the operator has turned the filaments on at high pressure, the gage has been broken during maintenance work, or the operator has knocked them off while behind the equipment.

We have standardized on our Pirani gage. If the normal Pirani gage does change its calibration over a period of time, and it is necessary to reset it to zero, that can best be done by the ionization gage. We have no comment whatever to make on the gages of Distillation Products, with the exception of one point. The sup-

ports on the older type gages were glass with two wire connectors supporting the grid structure and the output leads were insulated with glass beads. We found that tungsten evaporated from the filament, coated the beads, and provided a bridge between the leads which caused the gage to lose its sensitivity over a period of time. We stopped that by putting in a mica plate thereby increasing the life of the gage to six months from six weeks. We may have been unfortunate but that happened one after another. The last gage I received from Distillation Products was of a different design.

There is one more thing, beware of copper in the presence of octoil at high temperature. If anyone has a diffusion pump with a copper umbrella, silver plated, running in this equipment, they are likely to run into trouble, due to contamination caused by the break down of the octoil in the presence of copper. It is a high catalyst, and it releases a quantity of gas. I don't think it is the Distillation Products' policy to supply a copper umbrella, if you do happen to get one, with silver plating over copper, watch out for contamination.

#### DISCUSSION

**Mr. Berlant:** We have been in a position that is somewhat different from that of most of the speakers this afternoon, that while we are not in large scale production of coated surfaces, we have been in it for a matter of months, and we haven't evolved any considerable changes from standard equipment. At the present time we are running one hundred percent on standard equipment. I have some comments which might be helpful to those who are going into production with standard equipment on a basis somewhat similar to our own.

The question of starting up cycles in the morning, after a weekend is one for which we have found a very excellent solution. It is something that also helps get production out, and that is to operate seven days on a twenty-four hour schedule. You will get the maximum output. We have been doing just that for over three months now, and during that period of time we have trained girls – we have absolutely none of them from any other company who has done any of this work. What we do is select girls who know how to handle optics from our optical shop and they have an appreciation of the value of a coated surface and we can depend on them to give it the best handling possible. The next thing is to spend a lot of time with them and get a couple of smart boys who are willing to get down on their backs under these machines and let them learn vacuum technique. That is

the only way to keep these machines going over that period.

Gages, valves, seals and pumps are tied together in a way that makes it necessary from the point of view of production to deal with them as one unit. Gages are simply a measure of the condition of your pumps, and of your valves and seals. We have found that – standard equipment that has been supplied up until the present time did not have any shut-off valve between the diffusion pump and the forepump, and the only way it is possible to get a quick location of a leak, that is, a baffling leak, is to provide yourself with some special equipment. We found that once we acquired the habit of chasing leaks, we would spend a great deal of time doing just that, and while we are chasing leaks, we can't coat. To reduce the time to a minimum spent in that way, we use two pieces of equipment, one of them an electronic-multi tester which tells you very quickly what you very often suspect, that there is nothing wrong with the pumps, but that the gages are not in proper condition, due mainly to mechanical difficulties, vibration loosening connections at one place or another. That happens a great deal. The electronic-multi tester will very quickly isolate and spot the condition of a poor gage. In other words, we can start at the connection to the jar, and run right up the circuit and make sure that everything in that circuit is in proper operating condition. We can't believe there is a leak until that is shown. If the gage indicates that it is in proper condition, the next thing to do is to simply go over all of the flanges and make sure the connections are tight, that none of the bolts in the course of operation have loosened up enough to permit a leak. Hunting for leaks by the use of acetone on the gages; ff you put a little acetone or ether around the edge of your gasket or near the leak, you will see a quick result on your gage. While that is often true, just as often, it is not. Many a time we have gone over and over the equipment, being unable to find a leak and have followed by a very careful check of all things that would cause leaks making sure that everything was cleaned up, and we still weren't able to isolate the leak. We would then resort to the use of soda straws. It is a good policy to keep a few of them on hand. By blowing smoke through the straw, large leaks are very quickly found; more leaks found that way than with acetone. We can find leaks that we can't pick up on the gages, and it seems to be contradictory to what a lot of other people found.

Most important, however, is cleanliness. We insist over and over that the bell jar be cleaned regularly. As soon as we start getting into any of these maintenance troubles, we stop everything and give a first class cleaning to the apparatus. If we get to a leak that we can't trace within an hour or two hours of work,

we simply clean the inside of the bell jar, drain the oil, and wash out the inside of the diffusion pump – wash down the jets with acetone, reassemble everything, tighten up things and check the level of our oil in mechanical pumps. Nine times out of ten, it brings us back into good operating condition. We have found on the whole, that if we keep equipment clean and if we keep our glass dry, that is, the glass is thoroughly dry before it goes into the apparatus, our maintenance problems are greatly simplified.

# Practical Modifications To Present Filming Machines



**Ens. Storms:**Naval Gun Factoru

In our work in training operators for the Navy, we train them in the operation of three kinds of machines. The two that you see here and our

own at the Naval Gun Factory. Of the three, only the two that you see are used afloat. The Gun Factory machine is being confined to a shore base activity. I have had very good luck with the National Research<sup>1</sup> machine and with the with the DPI<sup>2</sup> machine as far as that is concerned, but we will confine ourselves to the National Research machine.

My first suggestion would be the base plate. I think it might well be cadmium plated, to get best results because in its present steel state it picks up fingerprints too easily, which results in rust spots, creating quite an outgasing problem.

Second I think I would like to see a Kinney valve installed between the mechanical pump and the diffusion pump. I think we are all agree that valves are very useful in leak finding and general machine maintenance. That problem would be simplified if a Kinney valve were incorporated, and I say Kinney valve because it has been our experience that that valve is the most satisfactory valve for high vacuum work. We have only just begun to get into other types of valves. We have not been in a position to say whether the type that is on the DPI at the moment is as good as the Kinney valve or not. I am inclined to think no, offhand. I am completely sold on the Kinney valve for purpose. We have trouble with the sharpened edge cutting through the rubber gasket. Another very important thing in my opinion is to take off this double switch that they have here on the filament voltage, and use two switches so that you can heat while depositing your fluoride, rather than turning off the heat and depositing the fluoride merely under the residual heat that you have in the optics. This would be easily taken care of. The gages I think, are very good. I like the thermocouple gage because it is simple and rarely gives you any trouble and if necessary you can install a new one at negligible expense. We have had our National Research machine approximately four months and I have vet to see any soft coatings come out of it: it is an excellent machine. It does very good work and very rapid work. I think they have completely given up the idea of the 40 mil tungsten fila-

<sup>&</sup>lt;sup>1</sup>National Research Corporation <sup>2</sup>Distillation Products, Inc.

ment, isn't that right?

**Mr. Jewett:** We have both filaments available so that people may use either.

**Ens. Storms:** I like this new heater. It is the first time I have seen it. That is not their fault, but because of the bell jar, they had to put a much smaller heater in there.

I want to criticize the little toggle switches that operate the ionization gage, as it is possible to turn the ionization gage on when the pressure is too high resulting in the burning out of the ionization gage. I think a small shield welded to it would help the operator. And the same criticism for the push button switch, and the main mechanical pump switch. By modifying the filament and heater switches you would also get around another potential difficulty when using inexperienced operators, as invariably many of us are. They forget to turn this Variac back to zero before switching on the switch for the filament, whereupon they get a large amount of sputtering in the fluoride because of the sudden rise in 4 current rather than a steady outgasing which is necessary before you bring the fluoride up to full temperature. I still wish the gages were made direct reading instead of just indicators. I see that the thermocouple has been changed over to that, but the ionization gage still remains in its original state. I like the idea of being able to say your vacuum is three times ten to the minus five, without referring to the McLeod gage to find out. That is probably a minor consideration because of the fact that you are actually getting good films out of this machine. I think the maintenance problems are probably simpler in this machine than any we have come in contact with. For some reason, they just seem to stay together.

About the DPI machine, I had one of the earlier models of the DPI and I must say I had a little trouble getting into operation. Probably more my fault than DPI. They have now undermined my criticism simply by changing everything I would change on the machine, with maybe one or two exceptions. I think they can do a little better job with their machine here to get a smoother finish all over the base plate, thereby cutting down the possibility of absorbed gases. The new terminal system that they have is a vast improvement over the old rubber stopper method of maintaining the base plate seal. I think everything else is about the same. DPI is a highly satisfactory machine, I believe, and we get excellent results with it. Good hard coatings – this heater is very well designed, and I like the simplicity of the thermocouple on there to indicate your temperature prior to coating.

**Dr. Lyon:** I wish to add what I know about the DPI machine. The shield surrounding the lamp for viewing the films, should have some little slits and crosses in it so you can view small elements and elements of different curvatures. If you depend on the open end only, it isn't sufficient for all conditions.

Another thing is that this well down here collects oil. The rightangle bend is supposed to act as a baffle, but it is imperfect and the oil will collect in the bottom of that well.

Furthermore, I don't see the need for two pumps. It is alright and will work, but I have had considerable experience with baffles, welded joints and some of these metal pumps, which are furnished by DPI and I must say that the welded joints have been very leaky. Apparently they have not been tested. I took that matter up with DPI at the time we had trouble, and they assured me that these were tested at  $10^{-5}$  millimeters pressure, yet we found holes in baffles so large that a Kinney pump (type VSD 556) wouldn't pull down below 200 microns. That has been one of my chief criticisms of the metal pumps and metal baffles. Apparently they are painted without having been tested and the paint seals the leaks somewhat and makes it difficult to find the precise location of that leak because the leak actually appears one or two inches away from the real hole in the weld.

I don't know what vacuum is obtainable or how long it will take to get down to the evaporating pressure in this machine – perhaps 20 to 25 minutes – but that is just about the time it takes with a single pump working on a Naval Gun Factory machine. It seems to me unnecessary to go to larger and larger diffusion pumps. If the leaks in these machines are effectively closed, a single pump with a speed of 275 liters per second will do the job adequately.

I have also noticed that considerable discussion has centered around the ionization gage. The emphasis has been on the troubles which have been encountered. Burn outs and breakage have been frequent, and the troubles in attaching the glass tubulation to the metal parts of the machines have been discussed. Why not eliminate all these troubles by discarding the gage? The Naval Gun Factory machines do not have ionization gages. Eleven machines are in operation at the Gun Factory and others at the Navy Yard. None of them have ionization gages. To the best of my knowledge, these machines are still producing a large number of coatings which are the equal of those produced by anybody else, without encountering any troubles from ionization gages.

**Mr. Berlant:** I would like to deal for a moment with ionization gages; to find out the troubles involved with untrained personnel. To date we have burned out about four or five ionization gages of which two were burned out accidentally - we never knew how it happened. They are really burned out when the switch is turned on without vacuum. Another case happened accidentally, when a switch was turned on and off again through error. A beginner got flustered; it was her first run at the machine. She was told to turn on the thermocouple gage, and before she could be stopped, the damage was done. The reason for that is this we use the ionization gage to verify the fact that we are ready to coat. We depend on the thermocouple gage which is furnished in the National Research machine; it is very dependable and rugged. We take all preliminary readings on the thermocouple gage and when a proper vacuum is indicated we turn on the ionization gage for a check. A record is made of the reading just for the sake of maintenance and for the sake of reassurance that we are not going to coat without a proper vacuum. We have never had any basic trouble; when we did have any trouble, it was usually in getting very weird readings on the gage. Then we found that due to the passage of time, the filament of the ionization gage had begun to sag with the heat and expansion. When that happens, we replace the gage. On the whole, we have not had excessive trouble. We have used the ionization gage only to verify pressures before coating.

### DISCUSSION

**Mr. Been:** With reference to the spattering caused by the rapid heating of the fluoride, the Navy Yard is using a shield placed directly over the filament on the fluoride, so mounted that it can be turned from the outside of the jar with a magnet. During the preliminary heating period the shield is interposed between the filament and the surfaces to be coated. After all possibility of spattering has passed we withdraw the shield and go on with our coating. We in the Navy Yard have had no spattering for quite a while. Nevertheless it is a good idea for anybody who would care to use it; and possibly we can help them on it, with blueprints or any other information we can supply.

**Mr. Behrndt:** I would like to explain something about our technique of pump testing. I know that our pumps leak occasionally, but I also know that when the pumps are shipped out at Distillation Products, that the possibility and probability is that they do not leak, because we give each of our pumps a severe test, and

several checks. The usual procedure is to subject them to a positive pressure as high as 40 to 60 pounds. After that, it must come down to  $10^{-5}$  mm or the pump does not go out. And I realize that you are going to say: well if that is true, why do we have leaks in our pumps? We are rather fortunate in this instance in being able to say that with the lens coaters, somebody else checks the pumps besides Distillation Products' employees; a Navy signs his name to the testing procedure, and when we say that this machine is brought down to an elemental vacuum and the vacuum pumps are turned off, and that the leak rate is only three microns in three hours, the Navy man signs his name, not us.

Well, we shipped that pump, and it arrived at its destination. Two days later, the customer turns on the vacuum pump and finds that the forepump comes down to about 300 microns. Then we begin to wonder what has happened during the shipment of the pump. We don't want to blame all our troubles on the carrier, but I think that is where some of the difficulty lies at the present time. These machines even though quite large, are delicate. The main thing to do is to get some help in setting the machine up, when you first get it. I hope I haven't stressed the point too much that we at Distillation Products have never knowingly sent out an inferior product.

**Mr. Zook:** Another thing would be to put the panel in a little more convenient place on the NRC machine. In the NRC machine you have to stoop over to operate any of the controls. I think the gages and all equipment should be put where the operator can reach quickly from his work to the instrument panel. Panels should be arranged in a logical order within easy sight of the operator.

**Mr. Wallace:** It would be advantageous for the manufacturers of these machines to have available a ready supply of spare parts for different kinds of Variacs, little switches, transformers, fuses, gages and so forth, because many of the people find that after they have operated these parts for a period of a few months on a continuous twenty or twenty-four hour basis, that failures occur. Adequate maintenance and an availability of such a supply of these parts would be a great help to the people who are coating.

### **Future Filming Machines**



**Mr. Behrndt:**Distillation Products

I think that probably most of the changes that will be put into the machines will come directly from you men who use them, be-

cause we do not do any coating. You must come to us and say "we don't like this" or "we don't like that" or "your pump has leaks," and tell us what you want. We don't ordinarily furnish the heating element, because we don't know what you people want. If you let us know what you want, we will do our best to see that you get it. I might add that we do not have any spare parts sitting around in our factory. If we did have any spare parts, we would put them into a complete unit without delay.

There are always certain things that the Engineering Department at DPI has been working on, as a result of the suggestions being made continuously by our customers. Many people have suggested that it would be very convenient to have a valve between the diffusion pump and the bell jar, so that the diffusion pump will operate continuously but I don't want to give the impression that these valves are going to be available tomorrow or next week. Therefore don't hesitate to order any equipment in the hopes that you will get one with a valve in that position by waiting. I have been assured by the Engineering Dept. that they have a valve which will do two things; it will enable the operator to isolate the two pumps from the bell jar, and furthermore, it will act as a baffle. That may solve some of the difficulties of operating on faster cycles.

There has also been some very just criticisms about the cost of ionization gages. What we are trying to do now is to make a better ionization gage, make one that can be purchased for two dollars, one that is more rugged. I can't promise that for next week either. As a matter of fact I don't want to make any promises.

Next, is the question of the Pirani gage. We realize that the Pirani gage does not maintain an accurate zero point, so that you may be getting pressure readings on it of one or two microns, when the actual pressure in your system is below  $10^{-3}$ . That is caused by the disintegration of the filament, impurities on the filament and baking them on. Those things have to be overcome, but I think that perhaps the most important thing is to get the basic equipment to you as soon as we can and as best as we can, so that you people will be able to turn out as much coating as the Ordnance people would like to have.

**Mr. Jewett:**National Research

At National Research Corp. we are trying to develop industrial equipment for high vacuum processes and we are trying to incorporate into

that equipment certain philosophies that we have about what industrial high vacuum equipment should look like. You had a pretty good example this morning from the discussion that went on concerning the origin of the design of our machine. The machine started as a university research laboratory set-up, and although we have made certain minor improvements in the way the parts are put together, it still remains the same basic idea of a single pumping system and a bell jar for the volume in which you place the lens, and it has certain other features on it which have been retained from its university days.

Sometime ago, Optical Research, Inc. came to us with a problem that was somewhat different from any that we have run into. in regards to coating machinery. Our own shop for instance, as well as most optical shops up to the time we got in touch with Optical Research, had been dealing with fairly small quantities of lenses. As a matter of fact, when I first started running our coating department, business was a little hard to get and I wasn't too unhappy when the machines did take eight hours to pump down. If they came down in an hour, it would mean sitting around for seven hours with nothing to do. When that company stated they wanted something to coat 25,000 lenses or some such figure per day, we sat down with them and in cooperation with them worked out a design of a machine which incorporated their ideas and our ideas of what a production machine should have. Now, I think neither of us believes that this is the ultimate in production equipment. I hope that I will get from people here suggestions as to how we can further improve this machine. I have some pictures here which I will pass around. We had hoped that we would be able to have one of the machines down here. but as a matter of fact, we are lucky to have pictures of it and even they were not quite dry when they arrived.

I will skip some of the items. In the first place, we want to have the machine on one level so we don't have to climb on a platform to look thru the top of the bell jar. We want to incorporate the ideas that have been discussed this morning for a continuous pumping manifold. As soon as people began to coat on a large scale and could afford to have maintenance crews, we then could discard the principal that everything on this machine including electrical and gage circuits must be kept as simple as possible. In the machine that we are now building, we have incorporated various safety features for oil temperatures as well as interlocking relays and things of that sort, so

that gages can't be turned on before valves are opened or pressure reaches the correct value. We think that in industrial vacuum equipment glass has no place. However, we have to use glass for sighting purposes, so you can look in and see how your lenses are being coated, however the glass that is in this machine is a replaceable element. We are sorry that the ionization gages are also glass but as a matter of fact, the only two glass elements in this machine are the sight glass and the ionization gage. Everybody is talking about the development of a metal ionization gage, and we are too, but we don't have one yet. In getting the machine down to one level, we have built the machine with a lid in place of the bell jar which was to be raised and lowered. We look at the problem as a problem of a vacuum chemical process where you would have a tank or tub or something and lift the lid off and there are the lenses right in front of you in a workable type of a jig, which can be removed and another jig inserted. We have tried to cut down the number of operations necessary between evaporations. In our present machine we have to raise the bell jar, remove the heater and in order to remove the heater, we have to disconnect the filament and the heater leads. Then we have to remove the jig, place a new jig on, put the heater back on, tighten up the leads, and finally lower the bell jar. In the new machine we have put the heater in another lead. We have put on some additional gages such as a thermocouple to record lens temperatures, and a meter to record filament temperature. The lens heater has been designed to be merely a holding heater to maintain the temperature of the lenses previously heated in an oven before being put into the machine. The pump cycle is too short to permit heating the lenses in the jar. The handle which you see on the right hand side is one which actuates the vacuum valve which closes the diffusion pump off when the system is open to air, and when the system is being roughed out. That valve works through a Wilson seal and is just a straight lever action. Some of the additional safety features on the machine are by means of switches in the lid; if the lid is first cracked, the high tension in the heater is automatically cut off, so that the operators are not exposed to the danger of coming in contact with hot terminal in the machine.

I think it is important to point out that this machine was designed for a specific purpose. It is not the type of machine that anyone would want in an experimental laboratory, or in a shop where different operations such as silvering or special coating are being done. For that type of thing I think the older types of machines are more flexible and more tractible. This machine was

designed up to the present point, primarily as a tool for large scale production. It is operated on a manifold production line, which can be extended to include any number of machines that you wish to put on the line. The line that we are working on for Optical Research is at present designed to accommodate six machines and is designed so that two more machines can be added to the line before we run into trouble in the manifold pipe size which would slow down our roughing machine. We are now building our first batch of these machines. Before we start the next batch, we want to find out if there is anything that should be changed to make it fit into the production problems with which we are not ourselves familiar. I would appreciate any comments that any of you may have on the machine. We want to know about as much of that material as possible, because we want to standardize on a new unit which will be universally applicable so that we may possibly be able to cease building a special unit for every different company that orders machines. That slows down our production very greatly.

One other point that I think is important in the machine, is that we are attempting to eliminate all possible joints. The tanks have welded steel construction. Incidentally, to answer one of the questions that was brought up earlier this morning, the plate around the top of the different leads that opens up is plated and polished. so that fingerprints can be easily removed and it won't rust. The interior of the tank is plated. Regarding the welded construction, there has been some cause to doubt the advisability of welded construction, and that doubt is very justifiable, because just anybody can't make vacuum welds. As a matter of fact, we have been doing it on a lot of specialized vacuum equipment, so if the thing has to pump down to ten to the minus seven in a very short time, and has to maintain a rate of leak of less than one micron an hour, we have only found one place that knows how to do welding of that sort, although we have tried several different people. It is obvious that other people can do it, but it is a very difficult problem to get proper vacuum welding. However, up to the present time, we have not encountered serious difficulties in that field.

**Dr. Lyon:** I believe most of my points have been pretty well covered. I jotted down here what might be called "fool-proof control," and I think Mr. Jewett has already covered that. Most of the production problems are personal failures—failure to follow a proper sequence or turning the wrong valve or switch at the wrong time. If a system could be worked out using interlocking electrical controls, microswitches attached to valves so that nothing could be spoiled no matter what sequence followed then that

would be a distinct advantage. Simplicity in the design of this equipment should be stressed. I think that the fewer the controls, the fewer joints there are to leak, and any method which may be incorporated to isolate quickly any leaks which may develop in a joint, is also very desirable.

Coating is undoubtedly in its infancy. During my short time with the Navy, I can see many applications for films and all sorts of films are wanted for various purposes. Uses for multiple layer films will probably be developed because these films have interesting properties as selective filters which reflect one color and transmit the complementary color. The production of multiple layer films requires accurate control of the thicknesses of the component layers and I believe that some photoelectric methods of control will be necessary. This is in the future and no one needs worry about it for a year or more. Although simplicity and elimination of superfluous parts on coating machines is a desirable goal, photoelectric control may become one complication which will be dictated by necessity sometime in the future.

### Training of Personnel



**Ens. Storms:** About all I can tell you is that I am in the somewhat embarrassing position of trying to tell you industrialists how to train people, who have

been doing it for twenty years. However I have a little different problem than you. I take men and train them to be operators and maintenance men out in the middle of the Pacific where they are the only ones on shipboard who know anything about vacuum system. So it is necessary to figure out a way to train both of them in the best way, and that is pretty tough.

I thought we might say something about supervisors first, and all I can do on that subject is to tell you what we do at the Naval Gun Factory. We do not definitely train supervisors or operators for coating or for any other purpose. They are taken from training groups and for this purpose we try to choose men of above average intelligence and educational background. It is impossible to get anybody who has a background of actual high vacuum work, or at least very unusual. If you should get such a person, about all you can do is instill in him the principle of foremanship, rather than let him learn anything about the actual operation of the machine. Perhaps Mr. Quin can give you more information about that than I. Much the same problem occurs from the inspector angle. You can take an inspector and show her what you want her to look for, and show her examples of failure and then from that time, she must learn to inspect by inspecting. I think you all agree with that. Now, in our training course for Navy enlisted personnel, we find that they not only want to know how, but they want to know essentially why, and it helps a great deal in knowing how if they did know why they are doing certain things, so we spend a good deal of time in seemingly poor theoretical background.

I might run over briefly our training schedule for these men, first telling you their background. They are all enlisted personnel who are graduates of class A machinery school, and they are all non-rated men. At the present time, they are from the ages of 18 to 25, a cross-section of American boyhood with perhaps a little more mechanical aptitude than the average, or they wouldn't have been sent to machinery school in the first place. They are brought in for a two months' course, and we spend the first week in giving them the introduction to filming, or coating—why we coat optics, the advantages of coating, and bring it home to them

quite clearly by letting them observe two telescopes, one of which is coated and the other not coated. We do spend a little time on lens cementing technique, so they can realize what the optical repair shop or the optical shop is up against in dealing with coated optics. We feel that that is worthwhile because it instills in them the desire to put on good hard coatings, so there will be no difficulty in handling the coated optic. We go into some of the elementary principles of light wave mechanics, and the theory of one-fourth wavelength film, including a discussion of the special troubles which enable them to understand why it takes a pink purple coating at the proper place to stop the filming operation. About that time, we are ready to start on the machine. As I said a moment ago, we have to teach them how to operate three different types of machines. However, if they learn the principles of high vacuum, they can apply that to all machines of course. We consider the machines as units, or take up separate units of the machines rather, starting with the mechanical pump, working from the mechanical pump up to the top of the bell jar unit. They learn complete disassembly, and maintenance of three different types of machine pumps, the Kinney Cenco Hypervac and the Welsh pump. We have one machine that is the equivalent of a Welsh pump for that purpose, to explain the operation of the Welsh pump. It has been brought out pretty well today that the problem of maintenance of the particular Kinney pump is not severe but if you go out where you are going to need one, you better know how to take it apart. Therefore they completely strip the mechanical pump and we found that by making drawings of the machine in various positions and cycles, they will get a better understanding of how a Kinney pump works and how to put it together. We devote a couple of days to high vacuum system in general, in which we include what material is permissible and what isn't permissible on high vacuum systems, and we also include in that a discussion of various materials that can be used in maintenance on these machines as well as the various waxes and cements that you are all familiar with. We teach them how to install a sylphon bellows in a Kinney valve, or in the make-up of the machine, either with flanges or by welding as the case may be. We spend time in "Strong's Procedures in Experimental Physics." The principles of various types of gages that are used—the thermocouple, Pirani, and the ionization gage: hoping to instill in them the desire to take care of the gages and handle them with the care that is required and it seems to pay off. We don't have much trouble. The only ionization gage which has been burned out was burned out by me. So I can't complain about burning out ionization gages. They learn to completely disassemble the machine and reassemble it, testing it at each

joint as they go along, by a system of blanking off each joint and testing it with a Pirani gage at that point, and to observe leak rates at the point of blanking off. If the leak rate is satisfactory, they move up to the next place—we try to get them on to electricity, just enough so they are able to make minor electrical repairs. If major electrical repairs are necessary, such as the ionization gage, they are instructed to call in a radio electrician of higher skill than they are able to attain in a two months' course. We spend a week on each machine in learning its cycle of operation. We find that in time they learn to handle all the gadgets on the machines, and to do acceptable coating in that time.

With the theoretical background that they have obtained in five weeks, they are easily able to recognize the correct color of a film. We have made up a series of discs, varying from an uncoated lens to one that is very thoroughly over-coated in small hues of color—about twelve discs in a set. From these they learn to match to the correct color and what to do about it should they run over. This is the actual operation of any of these machines which is not very difficult to learn as soon as you learn the sequence of steps. After that, it is merely practice and teaching them to get the lenses clean enough for the job, before they are put into the machine. They spend a good deal of time cleaning lenses, and everything is inspected by the instructor before they are put into the machine. We find that a good job of cleaning plus plenty of heat before coating is attempted, covers a multitude of sins in coating work, and if you have high enough vacuum and still have trouble, you can trace it to improper cleaning or lack of sufficient heat before coating is applied. We give them some work on aluminizing and silvering in case they need to do that. At the time, this instruction is not necessary, but we are preparing for the future on that subject, because they feel that it is something that will be thoroughly worked out in a short time.

# **Mr. Quin:**Eastman Kodak

Ens. Storms' approach to this is different than ours in that he is speaking of an allaround training. Ours is not. It is more spe-

cific, specialized job of operation training, not including the maintenance. I was a little at sea as to how to approach this. I have found from previous experience that it is hard to train anyone for optical filming and the skill required for the production of optical elements tends to draw a definite line of distinction between this training. But I propose in view of the shortness of time, to try a little hop, skip and jump here, to first pick up the training of supervisors and then proceed to the training of employees without differentiating between operators and inspec-

tors. I would like to note here that the procedures are a matter of evolution. They are not what we started with at the Hawk-Eye plants, but those in charge have changed the procedure and we are running on it now. Nevertheless, there will probably be more changes for you must bear in mind that ours is more maximum production.

When we speak of training groups we have 50 to 100 in a group. To take up the matter of supervisors, we will refer only to male supervisors because to date, we have very few females in supervisory capacity. The present supervision program is broken up into two parts; general supervision and special supervision and the general supervision is aimed at company policy and procedure., in regard to wage and salary administrations, the maintaining of an effective working force, and of course cost and quality control.

In the general group, we had approximately 145 supervisors from various departments and they attended 13 weekly meetings. These meetings ran from about an hour to an hour and a half. As a basis for these discussions, we use the slide sound films that were produced by Voco-Film Corp. in conjunction with the National Association of Manufacturers and we tried to apply these films to Eastman Kodak's policy. These covered methods of starting the new employee off, methods of correcting the operators and correcting conditions. the method of organization and how to organize themselves, the way to reprimand an operator, method of training, the effective employee attitude on production, the handling of grievances and problems. and of course production schedules and costs. Most of these films ran for about twenty minutes, after which the group leader, who previously had obtained some special example from various departments in the plant, would try to bring out the discussion on that and make the men talk about their own departments. At the end of the session, he would try to bring all the discussion to a focal point, and have these discussions covered in the minutes that are made up and distributed to each foreman. That is a pretty general picture of it.

The second part of the program is aimed at special supervisory skills, and I want to say in this first group of supervisors I just mentioned, you would have men from machine shop departments, optical departments, sealing and polishing and glass making—that is just a general cross-section. In the second part, special supervision skills when working in the job instruction program, the job methods training took groups of supervisors from within a department to cover special jobs. In doing that we drew up a department organization chart—a big one—put it on the board and made copies of it and gave it to each supervisor in the de-

partment. Now some of these departments would have as much as 45 supervisors, and his name was shown right where he stood in the organization, and from that basis we took all of one shift as a unit and discussed their part in reference to these various policies, and then after having done that with the three shifts, we got people from three shifts who are connected with polishing operations and we got them to gather in a meeting. All these people had discussions about polishing procedure in an effort to standardize processes among three groups that were working on different shifts. That worked out very well. It was a more recent development, within the last six months, and we have very good results from it. It brought out interesting differences of opinion and did help to standardize those personal things that come up in manufacturing. For example, some of our polishing department people think you have to chew a certain kind of tobacco and spit on the polish to get a better polish; another is that people put a little vinegar on it, that is what you meet up with.

In operator training, we used two general types, the first was the vestigial training and the second was what we call odd job training. Now this vestigial training—most of our people in the optical coating department are women—this applies to training women. The applicant is first given the Minnesota Test and the experimental form of Otis Test. The first is designed to indicate mechanical aptitude, and the second just general experience, and the third test was the old pin board test, to test finger dexterity, and ability to follow directions, speed of eye, mental alertness, etc. Applicants were scored on these different tests and divided into four groups—that was a rather arbitrary grouping. The first group was not hired. The three other groups were called above average, average and below average. We have had to lower the way you normally call an average score; for instance in the Minnesota Test you have to do that because of the general quality of people whom you are able to hire in the last year. We have a training school for these applicants—we originally ran a two weeks' course, and these applicants were brought in—worked regular factory hours and were paid regular rates as though they were not going into a training school. They work six days.

At first we ran that school for two weeks and lately we cut it down to one week, and made the training more intensive. We developed a regular curriculum for each day; printed it and a copy was given to each one. I have one of these here if any of you are interested in seeing it afterward, because all of the optical departments have various skills in common, it was supposed to give the trainees in this school, a foundation which would start them in any of these departments. The general object of that

week's training was to first make the employee feel at home at the Hawk-Eye Works, and then to explain the part that the Kodak Co. in general and the Hawk-Eye plants in particular had to do with the war effort, to develop an interest in optical parts manufacture by showing the relation of the optic job to the various jobs as a whole, to develop an appreciation of quality required and the effort involved in the manufacture of optical parts and the care necessary in their handling. We tried to instill good working habits and a real appreciation of what is expected of a person in a production department. We wanted to develop a familiarity with and some skill in cleaning of optics, inspecting gages, and the mounting of optical parts. We wanted to provide the operator, or trainee with opportunity to observe actual working conditions. The high-light of the week's training were the lectures and slides showing Kodak's products in the war effort. We showed them a picture of the product as it is delivered from the plant, in some cases pictures of the sub-assemblies and then we showed them pictures of the products as they are used in the field, the gun sight or panoramic sight on a gun carriage or a gun. That arouses quite a little interest. Every day a field trip is scheduled. and they are taken to the various departments in the plant in small groups, with someone to explain the work to them so that by the end of the week, they pass through all of the optical departments and they do form an idea of where they would like to be, and they are asked that question at the end of the week. That includes the molding, grinding, and all those operations which go into lens production. We gave them demonstrations and lectures and practice in the use of a loop, and for cleaning and inspecting optics, instructions in cleaning lenses, instructions in varieties of defects. We have some charts we made—a standard chart for various defects such as scratches and bunches. on polish, and all the various things that go into optic manufacture. Those in the Arsenal have made copies of these and it is expected to be issued to all the optical manufacturers throughout the country. In connection with that, we developed some of the so-called boxes which gave us for instance, the quality of the lens. We found them so convenient that they have just been distributed through the factory. When the blueprint calls for a certain scratch, at least a certain passable defect, the inspector finds it very useful to keep them on the beam. We teach them blueprint reading and we have large slide-rules about two by fours for instruction in that art. We have a large model of a micrometer to show them how to use a mike. Then, towards the end, when we have some of our finished instruments, some of our gun sights, they are allowed to look through these gun sights

at a target at the end of the building, some 400 feet away, and in that set-up we are able to show them the need for cleanliness in assembly and what affect these defects have on a reticle, etc. and to differentiate between those things that are essential and non-essential in defects. Each one of these trainees are marked daily, just as they would be in school, and at the end of the week they are marked by the training school instructor. He goes to see the foreman, shows him the girl's record and allots one to a department.

We later have a follow-up at the end of three, six or ten weeks, and in three months, we have a man who goes to these foremen and gets the trainee's records which are brought back to headquarters and correlated with their training school records. After having gone through more than 2500 trainees, we have been able to establish a pretty close correlation between the student training record and the job he should have. If we pick out a girl who shows high in those tests and is put in our high grade operations, she generally does a pretty good job. At first that was not so, but it works pretty well now. Also by following that through, the woman who interviews applicants, at the end of these three, six weeks' and three months' periods, asks them how they like the job and what is wrong, etc., and we are able to reduce turnover by 50 per cent as against those girls who are hired directly through the employment department and put on a job. We were doing it both ways, we don't train for some jobs. Here, I am speaking of those optical departments.

Now, what is probably going through your mind is that this sounds like a very formidable program and your reaction is, in a week you can only hit the high spots. However, we have proved to our own satisfaction, that this saves the foreman a lot of time, and in that week, we can do a better job than he could probably do in two or three months by the old method. That is particularly essential now, as the foremen have very many other things to do.

The second part is on the job training and someone here gave you the answer to that, when he spoke about people getting down on their backs under a machine to see how it worked. That is the way we have selected them in the plant—in all the jobs you have a mental ability and skill in operation factor. I doubt now whether you can see this, but the man in charge of this training has listed here the various jobs in the plant and in the optical departments. Here he has the job evaluation for maximum training skill. Here he has the average minimum. If you wanted to take the operations in coating here is one called fine inspection—it would require 140, and you try to get a girl with a 38 average in pin test and about a 40 average in the Otis Test. There are not many of those girls floating around, but we try to get

them; the foreman in charge tries to place them according to their skill.

Now in the coating, they are broken down into cleaning and inspection of coating and final inspection. We have developed charts for these girls to try to teach them hard coating processes. There are tests that are supposed to be followed, that are on the process sheet. That is what we call the Hawk-Eye process. We have similar sheets for cleaning of lenses for inspection, etc. and in that way we try to put down all the essentials that a girl should follow in her job.

### Cleaning Methods



**Dr. Lyon:**Naval Gun Factory

Much of the success which you will obtain in coating is going to be due to the cleanliness of the surface prior to the coating operation. You

won't go very far before you will learn that merely picking up a piece of glass, breathing on it, then wiping it on your coat sleeve, may give you a very hard coating. You might do that many times. Do not be misled by such tests. On the basis of such rough tests, you may conclude that it isn't important to clean the surface. Experience has shown that you should take as great care in cleaning the surface as you would if you were silvering.

Silvering requires a very clean surface. A magnesium fluoride film, since it is a transparent film will not show up the imperfections readily as will a surface which has been silvered, but it is important, nevertheless, that the surfaces be absolutely clean. All experience demonstrates that the cleaner the surface the more tenacious will the film stick to the surface of the glass. The tests which will show up such uncleanliness will be some of the severer ones such as the salt spray, vigorous abrasion, or boiling in water. If you wish to determine by experiment what is meant by a clean surface, possibly one of the best methods of doing it is to take a thick piece of plate glass, about one-half inch thick, scratch it with a glass cutter and break it; then without touching the cleavage surface, coat that surface and see what a difference there is between that really clean surface and one which is unclean. You can also estimate the cleanliness of a surface by the way water will wet it. A clean surface of glass will be "wet" by water. That is, the water will not roll around on the surface like a globule of mercury. As you roll a drop of water over a clean surface, it will leave a track of water which clings directly to the glass. If the water does not cling to the glass you do not have a clean surface. Similarly, when you wash a lens the water should drain off in a continuous film leaving at the edges as it drains off—if you hold it vertically—little interference patterns or colors of the rainbow as the water recedes. If it breaks up into groups of rivulets, and blotches which coalesce into droplets the surface is not clean. Experience has also demonstrated that there is no best method of cleaning. There are several variations of a standard technique, which achieve the desired result. I am going to describe the method which is used at the Gun Factory.

The lens is washed in Aerosol and water—Aerosol being a strong wetting agent. It is believed that on new glass with which most of you will be dealing, the only impurities will be organic matter such as wax, grease and finger marks. Hence organic solvents and strong wetting agents should remove them. Inorganic materials will be either imbedded right in the glass, where they cannot be removed by scrubbing or they will simply be resting on the surface and can be removed by scrubbing. Consequently, the lens is washed in Aerosol just as you would wash a dish by rubbing with cloth. We feel this treatment will remove all the inorganic particles. The Aerosol being a strong wetting agent, will also make the water wet the surface thoroughly. It will clean off grease and finger marks. After this washing, which takes less than a minute, the lens is then placed in a pan of running tap water to rinse off the Aerosol. The lens is then stood up in a little rack vertically and a stream of hot distilled water is played upon it. This removes the tap water which may have impurities. If the tap water is allowed to dry on the lens, the impurities may be deposited leaving a dirty surface. This spray or stream of hot distilled water not only washes away the impurities but also heats the lens. This water is held to about 95 degrees Centigrade. Boiling water will not do any harm. We have never broken any lenses or prisms by the direct application of a stream of hot distilled water upon them. Before the distilled water has been allowed to dry or the lens to cool, a rinse of redistilled acetone is squirted on the optic. This has the function of removing any slight traces of grease which may remain, because even distilled water may have traces of sediment or grease in it. The hot lens, of course drys quickly because the acetone is so volatile. Acetone boils at 56 degrees Centigrade and the heated lens will be dried very quickly, because the acetone evaporates so rapidly. Now, the lens is picked up by the operator with the fingers. Rubber gloves are not necessary for this operation. It has been found that if the fingers do not touch any part of the surface to be coated, the lens can be picked up by the edges and placed into the lens holder where it is then ready to go into the machine.

Now, I can't stress the importance too much of thorough cleaning. Results of improper cleaning will show up as pin holes or blotches or small irregular patterns which will be removed by people in the factory as the lens goes through assembly operations. The lens receives a scrubbing a number of times in the assembly operations and if the coating has been deposited upon unclean surfaces there will be spots removed from the coating. Notice that this method does not use cloth, and nothing but liquids touch the lens after it is removed from the first solution.

The surfaces to be coated are not touched thereafter by any cloth. paper or fingers. You will probably hear variations of this method in which some cloth will be used or there will be subsequent rubbing. It may be merely prejudice on my part but I feel that any cloth which is used will ultimately become contaminated with finger grease, and with particles and grease which are present on table tops. These will be transferred to the lenses and sooner or later you may run into trouble, because one never knows just when to renew the cloth. The method which I have described eliminates all those possibilities. Those of you who have visited the gun factory may have been a little discouraged upon observing this method in operation and with some justification, because the handling of a large number of many different sizes has not been worked out fully. It is somewhat in the experimental stage. We don't know how to handle a large quantity of large prisms and then again small lenses. It is a question of mechanics—a mechanical operation of handling these lenses rather than the method.

Mr. Denton: Gentlemen, the first thing I want to say about cleaning is that as I said vesterday. Frankford Frankford Arsenal Arsenal got into the coating game much later than many of the other companies, and unquestionably we would be a lot further behind than we are if we had not gone around to the companies which are represented here and obtained information from them. Particularly National Research, RCA and Bausch & Lomb, Eastman Kodak, the Naval Gun Factory and several others. Some of these people here may say that we got this information from them. It is quite true, I did, and want to give you credit for it. What I will try to do is to give a general talk on cleaning, because I feel that there are probably several systems all of which work properly, and those of you who have not already set up your cleaning plants and have them working to maximum efficiency for your type of production will probably pick the one which seems best suited to you.

Now, I think Dr. Lyon stressed enough the importance of cleaning. If you don't have a clean surface your coating won't stick and adherence seems to be, as Dr. Lyon discovered, the most important factor contributing to durability.

In running our production at the Arsenal we put a lens which had been rejected for something else in a convenient position to control our color. Literally many times we spit on it and wipe it off on any cloth handy. The cloth may be dirty or oily in some cases and a surprising number of times we have then tested the test lens and the coat seems to be at least as good as we get upon the glass which we have very carefully tried to clean. Now, you

will undoubtedly hear plenty of stories about that sort of thing. It can be true in isolated cases, but what you want to do is to get 99.9% of your production through, and I am sure a method such as that, although it may work in isolated cases will not work on large production orders.

In cleaning for non-reflective coatings or mirrors I think one thing should be stressed. You must get the element clean and dry. It is comparatively easy to clean glass. There are a lot of things that will take most dirt or grease off the lens and leave it setting in distilled water or acid or alkali or a solvent. But, to get that lens surface dry and ready to coat is probably the hardest problem, and connected with that problem is this: when you are cleaning any optic you must be sure and get your cleaning agent off whether it's soap or detergent or alkali or a mild abrasive or just water. If you don't get the cleaning agent off properly, you will probably be worse off than when you started. That is something to bear in mind.

I have tried to divide the different types of materials according to the different processes which will take them off into four categories. The first category will be oil, grease or dirt which adheres to the lens. There may not be a lot of grease or in some cases there may be more, but at any rate you have got to give 100% cleaning for that type of material because it seems to be the experience of most manufacturers that there is plenty of grime and dirt on the optics. The next, and what I think the most important part of, cleaning is the drying. You must get the cleaner off and get it off perfectly or else you are just as far behind as when you started. The next thing you could call "delinting" or "dedusting." That would be dust which would get on the optic after you have presumably cleaned off most of the grease and dirt. Even in the cleanest place you can imagine you are bound to get some dust. The final would be the cleaning in the vacuum to get off the mono-molecular layers of anything which may be left on the glass. There are two common methods of doing this. One is the heating which we are using almost universally and the other is the electronic discharge.

Now in starting off you have to perform your preliminary cleaning. You have to get off most of the dirt, junk or wax or whatever else is on there. The common materials used to do that in the optical industry are inorganic reagents such as alkali or acid, and in some cases solvents, detergents and mild abrasives. I have heard people theorize upon the fact that you can probably get a completely automatic cleaning method in which you just dip through a bunch of solutions. Frankly, on the average run of optics which you are coating today, I don't think that will prove to

be entirely practical. It has been our experience and the experience of several of these manufacturers that mechanical action is necessary. Alkali is known to take off grease and so forth, but if it is stuck on there good and tight a little mechanical action to rub it off seems to help and I would advise that somewhere in your cleaning process you actually rub on the lens with some kind of cleaner which will not damage it. You may have to use clean cloths and whatever mild abrasive you use has to be non-gritty. These together with mechanical action seem very important.

The next step is the drying after you have either washed it in alkali or soap or possibly rubbed it with mild abrasives such as precipitated chalk. You want to get rid of that wetness. There are three methods of drying. One is the solvent method of drying which Dr. Lyon just described—his very good method using double distilled acetone. Another method of doing the same thing which I think was first developed for optics at Bausch & Lomb was the use of the vapor of refluxing isopropanol. The optics are dipped in the vapor. This is really a method of getting double distilled isopropanol. Another method of using a solvent is to put it on, then take a cloth and wipe it off. That method I wouldn't recommend. Although some personnel can wipe a lens clean that way, they have to be pretty careful and pretty skillful in order to do it. Now, still another method and perhaps possibly the best method, but one that doesn't seem to be easy for production, that is, after you have finished your lens cleaning, to wash it, rinse it thoroughly in distilled water and then get rid of that distilled water without using any other material. Such a method is used by one company. You can take a stream of clean compressed air and chase it off by proper technique and if you chase it off fast enough, you get a good clean surface. Another method for lenses, at least, is just to put them in a spinning device and spin at maybe one or two thousand rpm, shoot your distilled water in a stream on the lens. The spinning will shoot it off so fast that you will get a very clean surface. Still another method which sounds very poor, but which we have found to work fairly well, is to rub the lens with a mild abrasive suspended in water and then actually let it dry on there and wipe it off. We are using at present a product called "Wet-Me-Wet" which is precipitated chalk held in a cotton flannel bag. We are indebted to Bausch & Lomb for introducing us to that method and have found that it works very well. We put it on and wipe it off with two clean cloths. Part of it is wiped off with just an ordinary piece of cheesecloth and that is followed by wiping with a lintless cloth. Acetate rayon is a very good lintless material and the charge that is set up on the chalk dust seems to repel that on the glass so it doesn't stick the way that lint

sticks when you try to brush it off. One manufacturer actually uses Bon-Ami, which most of us would consider too abrasive.

Here is the main thing I want to put across. Mr. Boydston has tested at the Arsenal recently a large number of coatings from different companies, and I think all of these methods were represented in the coatings tested. We have tested several of them in our production. All of these methods of drying do work if used properly and good coatings can be produced by any of these methods. Now, your exact production needs and your own references will determine which method you want to use yourself and it may be that you can get a shade better adherence by using one method rather than another. At any rate coatings can pass perfectly well when the surface is dried by any of these methods.

As far as de-linting is concerned, the first thing is to get your atmosphere as clean as possible. An air-conditioning system using a Precipitron is very helpful. There are several methods which may be employed after the rack has been put in place and the bell jar dropped down over it. One of these is to direct a stream of clean low pressure air over the surface of the glass. This seems to work very well. Another method is to brush the surface of the glass with a fine brush surrounding a vacuum nozzle. This also is successful. A third method which is not to be recommended is merely to brush the surface of the glass with a fine brush. Another method which frankly we haven't done anything on, but several persons have mentioned it to me, is the possibility of actually putting the element through some kind of little precipitation or electrostatic gadget. I think that's a good idea. However, whether or not it can be worked out practically I don't know.

The last thing that we have to take off is whatever we can't take off before, which is the mono-molecular or several molecular thicknesses of adsorbed molecules. It may be water, soap or various other things. The heating method at present seems to have the priority as being the best method of getting such material off, and finally getting the surface ready for the material that you are going to evaporate so that it will stick. The electronic bombardment which you heard described yesterday may do about the same thing, but up to the present we have no results to show that it is as good.

I think that unless somebody has data to prove otherwise that you are going to have to do your cleaning, at least a great deal of it, by hand where each element gets individual attention, by using one of the above methods. I do not know of anyone who has developed a mechanical method which has worked properly yet. The most important part in your cleaning no matter which method you use or which variation of these procedures, is to keep after

your help all of the time. Try to tell them how to handle the glass without breaking it of course, and without getting more dirt on than they take off, and continually, keep the quality of this work in front of them by some kind of a reject list for poor cleaning or something similar and post it in the room where they are working. Keep them on the ball as it were, all of the time, so that they will try to keep a high quality of cleaning. I feel that is most important because I know there are defects in all of the methods of drying. For example, in any solvent drying method or any method where you are spinning or blowing off distilled water, if you let just a little bit too much of any liquid dry on that surface unequally, you are going to get a mark or stain which is going to show up in your coating and very often make it non-adherent At that point, and in any method where you're taking an actual abrasive or mild abrasive and letting it dry on the surface and wiping it off, you run into the dust problem. Even if the optics are cleaned reasonably well and they don't use this cloth right in wiping the dust off you get into trouble.

One or two other things that I might bring up here are these: it is very often a good idea to have the optic actually cleaned mechanically twice. At present, at the Arsenal we first just use a wash with detergent. Aerosol is good, Dreft is similar, also Orvus. All will work properly. After rinsing the detergent off we wipe it with the "Wet-Me-Wet," let it dry and then wipe off. Now, if the chalk is going to do a good job you might object to using the detergent method also, and that is probably a valid objection. I think a good cleaning with either agent really gets it clean enough in most cases. However, the fact that you have cleaned it twice and actually gone over the surface twice is important, because by going over it twice, the chances of the operator missing a place twice in a row is brought down much nearer to a minimum, and in that way you have double insurance against poor cleaning. I think that's about all I have to say. Thank you.

Dr. Lyon:

I am glad to see that even though Mr. Denton Naval Gun Factory came in after I had started my talk a, at least we didn't disagree. He stressed the idea of mechanical action or scrubbing. I think that is very important. He also spoke of a non-abrasive material. He means materials such as precipitated chalk or calcined magnesium oxide. One point I didn't cover was that it is not necessary to use any corrosive liquids. Sometimes sulphuric, hydrochloric or nitric acids and strong alkalis are used for cleaning. It isn't necessary to use any of these corrosive liquids. Just use a simple detergent such as Aerosol or the "Wet-Me-Wet" method and you will come out alright.

For blowing off lint, we use hand operated rubber atomizer bulbs and they seem to be quite effective because we have had rather sad experience trying to purify air. Although the air is passed through large silica gel filters, the tendency is to wait until trouble is encountered before the silica gel is renewed. It is also very difficult to get really clean air, even with the silica gel. Some particles come though and will show up. It may not be quite so bad on magnesium fluoride because the specks that are present may not be easily visible but we have found that compressed air does give specks when we try to silver and we conclude that the specks are there even when we are applying reflection reducing films.

**Mr. Peterson:** I am in full agreement with the principle of the need for clean surfaces because it has been our experience as well as Dr. Lyon's that without a

clean surface to start with there is no such thing as a good film. I might elaborate more fully for you our methods of cleaning. The two methods referred to above as the degreasing and "Wet-Me-Wet" are in use at our plant. The degreasing method has the advantage and it eliminates handling of the clean glass with the hands thus avoiding the spread of thin films of body greases over the glass surface. The degreasing method is not complicated. We have built our own degreaser but I believe that any of the commercial types can be adapted to this work. A stainless steel tank was obtained in the bottom of which were fitted two Calrod electric heaters. Copper tubing was run around the top of the tank to provide cooling for the alcohol vapors. A quite thick vapor layer of alcohol results which is kept in the tank by the cooling coils. The glass to be cleaned is washed first in a wetting agent solution. This can be 1% solution of Orvus or Aerosol. The glass is then allowed to rinse in a running stream of tap water for a few minutes to remove the wetting agent from the surfaces. The glass is then placed in a suitable holder and rinsed by dipping several times in two pans of hot distilled water. It is then dipped into a pan of isopropyl alcohol. The holder and glass is then suspended in a "degreaser" tank. The lens remains in the tank until no further condensation occurs on the surface. They are then removed in a hot dry condition and placed in the filming rack.

Am alternate method of cleaning is one that we have chosen to call the "Wet-Me-Wet" method. In this process the preliminary cleaning in wetting agent solutions is as described before. After the lens has been washed, it is merely rinsed in water to remove some of the wetting agent solution. Then a finely milled calcium

carbonate powder is spread over the surface in a thin film and allowed to dry. The drying can be hastened by the assistance of a blast of warm air from a hair dryer playing across the surfaces.

When the thin film of powder has dried it is removed by wiping it off with Kleenex tissue. The lens is then inspected for cleanliness and placed in the filming rack.

The calcium carbonate powder can be purchased under the trade name of "Wet-Me-Wet." It comes in a small flannel bag that is convenient to use. The whole bag is moistened after which the powder oozes out of the bag and can be spread evenly on the glass surface in a thin film. There is no need for scrubbing the glass with the "Wet-Me-Wet." The surface is wiped gently. We have no evidence that the glass surfaces are scratched by this method

**Mr. Pettus:** *RCA* 

We feel that this subject is probably one of the most important phases primarily due to the infancy of the lens coating field and the nature of

the reflection reducing films. Although we have been conducting research along these lines for several years and have been actively engaged in coating optics, we find we are often confronted with new and different problems. I would like to recall a little of our history in this field prior to the war, when we were engaged in coating of optics to be used in camera, projection machines and other such devices which came to us from the field. Quite often we might expect to find anything in the way of tarnish or stains upon those surfaces. Some of these deposits could be removed in ordinary washing solutions while others could not. Experimental work showed us that some could be removed in alkalized solutions while others required acid solutions. Very often that was not the answer, because acid will attack some glasses and alkali others. We finally arrived at a policy such that if the glass were stained beyond the point where a neutral detergent or a proven cleaner would remove that stain, we were forced to return it to its owner asking him to have the surface repolished. I only cite that because that was a common problem with us prior to the war. With the advent of the war these problems were greatly reduced because today we are working with new stock that has not had a chance to accumulate tarnish or stains.

However, even since the war, there are a few obstacles which we have found still to exist. To list some of those in their order of importance I would class them as rouge and emery deposits, oil and grease, pitch, finger marks and even water marks. Since it is quite difficult to determine just what might be present on a particular lens and at the same time determine just which sol-

vents or solution should be applied, it is quite evident that a large percentage of rejects can be attributed to the preparation cycle. It is also difficult to employ an individual inspection operation when you consider that it means the handling of thousands of pieces of glass each day.

As we have previously heard there are many different procedures used for the preparation of these surfaces and I would like to describe the method which we at RCA have employed with the best results up to the present time. The first step consists of immersing the glass in a warm solution fifty degrees Centigrade, containing one to two percent of Calgonite and a small amount of tri-sodium phosphate in which we perform the mechanical scouring action, which is guite similar to that used with the Aerosol and other wetting agents or detergents. Then we follow by simply rinsing in warm tap water again at 50 degrees Centigrade, followed by a distilled water rinse. Then dip in a filtered solution containing fifteen grams of Bon-Ami per 100 cc's of distilled water. This film is allowed to dry and then is removed with carefully selected linen cloths. This operation is done by two operators. The first one wears rubber gloves primarily for the protection of the operator's hands which are constantly in water. The second operator wears lightweight cotton gloves and performs the wiping or polishing step, and in turn places the work directly in the coating mount. Upon completion of the loading, the mount is kept protected from dust and atmosphere by placing in a suitable storage cabinet where it remains until ready to be received at the coating machine. This practice of long storage is somewhat frowned upon and we endeavor to time our cleaning cycle with that of the coating cycle thereby permitting the work to flow continuously from one position to the other.

Before the work is placed in the evaporating position we consider it the duty of the coating operator to make certain that all of the glass in the particular mount is cleaned and dust-free. I wonder how many of you have used the method of exhaling on a glass which is clean and observing interference patterns. We consider that rather a good test for the inability to produce fringe or interference pattern is an indication that the glass is not cleaned and should be rewashed. The removal of dust particles is accomplished with the hand bellows and as has been pointed out, the use of camels hair brushes is not to be considered a choice of the two methods.

Now, I would like to recall just one of our blue Mondays. We, of course, are producers of reflection reducing films only and receive glass from a number of various optical manufacturers. In one instance we received from a new manufacturer a large ship-

ment of glass and started to process it. At the end of the first day's work we found ourselves having about 27% rejections for soft coatings. Because we employ the identification principle to all glass so that we can return such glass to its rightful owner, we were able to trace every one of those rejections back to that manufacturer. Then we examined the glass which was still to be coated and found that almost every surface had some sort of film on it which we had not been able to remove in our normal cleaning operation. By going back and using Mr. Denton's method of a double mechanical scouring we were able to accomplish a good cleaning action. This instance is cited only to show that all of us must be on the alert if we are to avoid such pitfalls. Therefore, we as producers of non-reflection films appeal to all of the optical manufacturers to use every precaution to prevent the contamination of the surface of elements which are to be coated with foreign substances. Your help in eliminating these problems is urgently requested and will be greatly appreciated.

I would like to make one more defense in the use of Bon-Ami. I think there is a close parallel to that of "Wet-Me-Wet." I don't know the direct composition of either of them. I am not giving a testimonial for makers of Bon-Am, but we find that their little slogan "Has never scratched yet" has been pretty practical. We do add a filtering when we make up such a solution. Personally I can not say that it's worth the effort, because we have never found any traces of scratches or damaged surface due to the fact that we have been using Bon-Ami. It was also mentioned a little while ago about the possibilities of electro-static discharge. Some of our research men went into that in a small fashion some time back and it has been reported that the results are not what might be expected or, at least our company has not gone into the thing fully. We found that with the electrostatic field you might have a particle of dust and when the discharge hits it, it might fly off there and return some other place. We employed that thing for the use of cleaning glass plates when applying acetate film for making instantaneous recording blanks where the cleaning of glass is also somewhat of a major problem. Possibly there are other methods in which ionic discharge can be employed and which we did investigate. It is something to look forward to in the near future.

### DISCUSSON

**Dr. Williams:** I would like to correct might have been a misconception from one statement of Mr. Denton's. I don't believe he intended it the way it sounded. I think It would be agreed that ionic bombardment in the chamber is not the equivalent of heating the glass to high temperature on the final result. I think his statement implied that it was. To be sure both mechanisms have a similar action in removing the water vapor that might be adherent to the glass in that intense bombardment is the same as heating, but heating provides a further action having to do with the tenacity of the film.

I believe most of us found that new glass has stains and burns which cannot be removed by any ordinary method of cleaning. Now, I have heard of a de-stainer. Does anyone here know more specifically where it is or what it is?

**Mr. Berlant:** The de-stainer is a product of U. S. Instrument Co. I think that quite a few of the gentlemen have had samples of or have purchased quantities of this material from us in the past. It is a combination of detergents and cleaning agents that we had to work up ourselves when we encountered a bad stain problem on prisms. We passed it around and quite a few of you gentlemen have used it, and it is available to anybody who wants it.

**Mr. Wallace:** We have followed the Gun Factory method for the last ten months and have met with a great deal of success. We have one modification where we are cleaning fairly large lenses, that is lenses of about  $3\frac{1}{2}$  to  $4^{\prime\prime}$  in diameter, about  $3\frac{1}{4}$  of an inch thick. We place that type of lens in an electric oven at 75 degrees Centigrade and upon removal from the oven we immerse them in the hot Aerosol and then scrub it with a swab of degreased cotton and follow that with boiling, double distilled water and with re-distilled acetone. It is very important to use re-distilled acetone. Commercial or chemically pure acetone is good.

**Dr. Williams:** In the matter of cleaning we are all working in the dark, and there is no one who has yet devised a simple test which will not dirty the glass when the lens is tested to see whether or not it is clean. Now, I am perhaps asking for Utopia but I would say if someone wants to do a little research that would probably be the most fruitful field in which he could possibly work. It is too late to find out after it has been coated.

**Mr. Gilmour:** During the discussion two ideas have occurred to me which I should like to leave with you. We have found that the degreaser for drying lenses previous to coating has worked very well. It is more satisfactory to dip the lenses in ethyl alcohol previous to degreasing them to use an isopropyl dip. The reason for this is not readily apparent. Experience has shown us that it is necessary to clean a degreaser thoroughly before using since we have found that it is generally contaminated with small particles that tend to adhere to the lens. With regard to the general subject of cleaning glass surfaces. I am reminded of the old-fashioned process of sputtering platinum which has been done for many years. In this process I have often noticed that glass which has been sputtered once and cleaned off and sputtered a second time, tends to retain the platinum film much more firmly upon the second application than upon the first application. This indicates that it might be possible to satisfactorily clean a glass surface preliminary to evaporation by replacing the usual platinum plate with an aluminum plate so that the actual sputtering does not take place, and then proceeding as in the sputtering operation. Another point that I believe should be emphasized is the importance of preliminary processes of polishing, blocking, and unblocking to the final nature of the surface to be coated. Refrigeration provides an excellent means of unblocking without affecting the surfaces of the glass. However, the blocking pitch must be so constituted that it may be used satisfactorily in the refrigeration procedure.

With reference to testing hard coatings, one of the tests which we find effective occurs when the instruments in which our lenses are used are chilled to  $-40^{\circ}$  F. As they are returned to room temperature, the dew point is passed and if this moisture can be satisfactorily wiped off the coating, it constitutes an excellent test for adherence. If the same procedure were followed before coating single lenses, it should constitute a good test for cleanliness since it simulates the breath pattern test which time has proved effective.

I should like to ask one question regarding production, that is, in what proportion do other manufacturers find it necessary to divide their people between coating and cleaning in order to obtain maximum efficiency in their department? I find that our cleaning problem is greatest and if we are coating 70 lenses to a bell jar, our coaters are standing around looking for work. It isn't because we don't have space enough. It is because of the slow procedure in cleaning. I just wonder what someone else's proportion of cleaners to coaters is. Ours is 3 to 1.

**Mr. Berlant:** We have approximately three cleaners for each bell jar in operation and the only reason we keep three cleaners to

each bell jar is because we need the cleaners for future use and we can't have trained cleaners on hand. We believe  $1\frac{1}{2}$  to 2 people is all that is necessary to keep the bell jar of the conventional type of one hour cycle filled with the cleaning methods we're using.

**Mr. Denton:** The proportion at the Arsenal is exactly the same—3 cleaners to 1 operator.

**Mr. Gilmour:** The next question is the use of isopropanol degreaser. In the use of distilled acetone for film drying is it necessary to have an exhaust system?

**Mr. Berlant:** I can answer on our experience. We have a commercial degreaser for the use of isopropyl alcohol. The degreaser is electric and was made for our needs. It was necessary to add a suction hood to carry out fumes. If we had had fewer bell jars and less equipment we might have been able to get away with it.

I would like to point out a word of warning in regard to commercial degreasers. We experienced trouble when we first used a commercial degreaser. We discovered whatever compound had been used in brazing the joints of the degreaser was not resistant to the degreasing agent.

**Mr. Denton:** I do not recommend the use of a suction hood, first, because generally the vapors are heavier than air and second, because any lighter vapors will be drawn out. Nevertheless if something must be used to eliminate fire hazard and obnoxious odors, I think possibly the most effective system is to encircle the top of the degreaser with a continuous suction nozzle. Any vapor then attempting to escape over the side, will be drawn off immediately without unnecessary loss of the lighter vapors.

**Dr. Lyon:** In using the acetone method at the Gun Factory we do carry out the operation under a hood because the hot lenses evaporate the acetone so rapidly that a fume hood is required.

**Mr. Been:** We at Brooklyn Navy Yard, since the problem of cleaning is one of time, try to eliminate as many steps as possible. Degreasing is one of those eliminated. The Naval Gun Factory method has proved entirely successful, therefore why not standardize on it?

**Mr. Berlant:** We try to avoid building our own equipment whenever possible. We are busy coating glass and we do have a com-

mercial source of supply for a satisfactory electrical degreaser which can be obtained by any of you for use with isopropyl alcohol. You will have to have added to it as a suction hood similar to that described by Mr. Denton—a type of hood that we found was satisfactory. Anyone wanting the name can obtain it from me.

**Mr. Jewett:** We have tried several times to develop some sort of production technique for cleaning lenses as well as jigs to eliminate handling. So far we haven't been successful primarily because the liquid solvent we are using refuses to dry without leaving some sort of stain on the lenses. I wonder if anybody tried or found some successful method?

**Mr. Gilmour:** We investigated several kinds of holding fixtures. Of course, we still have to individually take each lens and place it on the holding rack. I can see from recent discussions it is a decided disadvantage. However, we did go to brass racks for holding the lenses after the initial scrubbing. In other words, we merely scrub the lens with any of the things that have been described and then we set these lenses in a rack either under water or under alcohol and carry the rack thru the various rinses and out of the degreaser and then ready for dry transfer from that rack. These racks that we make hold about 12 of the largest lenses which is adequate for our purpose. I think we hold about 20 to 25 lenses on the smaller rack.

**Capt. Dawson:** In the polishing and inspection rooms of most shops they use these red wax pencils very extensively to mark out defects. Mr. Denton and others tell me that once you get that wax marked on the polished lens surface, it is extremely difficult to remove, so I would suggest that we give consideration to some other method of marking during polishing.

## Preparation of Pure Magnesium Fluoride



**Mr. Peterson:**American Cystoscope

About a year ago when the Bureau of Ordnance issued a directive to many of the producers of Naval optical fire control instru-

ments that it would be necessary to coat the optical elements, we looked around and investigated the various equipment available and had the opportunity and pleasure of investigating many of the units that were then in operation. We were advised that fluoride would probably be very difficult to obtain; that the facilities have the only available source, that the Naval Gun Factory was very crowded and due to the demand of the various service facilities for the fluoride, outside contractors might have considerable difficulty in obtaining a supply. For that reason, we provided facilities for the production of fluoride following the procedure developed by Dr. Lyon at the Gun Factory. We have now been manufacturing it for our own use for several months. Later we had a capacity for more than we required and were requested by the Bureau of Ordnance to make that excess capacity available to other people who were short of fluoride. Since that time, we have been doing just that.

In the preparation of fluoride we start with the chemically pure magnesium chloride having six molecules of water of crystallization and chemically pure hydrofluoric acid. Hydrofluoric acid which is about 40% HF is placed in a platinum dish of about 500 cc capacity. Approximately 198 grams of magnesium chloride is dissolved in a minimum amount of pure distilled water. That is added to the hydrofluoric acid and the mass is constantly stirred with a platinum rod. That mass is then heated very gently over a low flame for a period of about one hour with constant stirring. The temperature is then raised. The mass, of course, becomes very gelatinous and stirring has to be more or less continuous in order to avoid spattering. The temperature is then further increased and the vapors are tested until no acid is detected in the vapor coming off the dish. Concurrent with those tests, tests are made for the presence of free chloride by a sample of the magnesium fluoride with diluted silver nitrate solution At the end of that period the fluoride is transferred into shallow platinum dishes and it is placed in an electric furnace at 850 degrees Centigrade. It is left in the furnace for a period of one hour and is then transferred to sealed containers. Now, having a Coating Division, we are in the fortunate position of being able to test our own product and to use it on the optics.

A good fluoride should be a soft, fluffy heterogeneous mass having no solid crystals. Now, of course, any chemically pure raw material such as magnesium chloride or hydrofluoric acid will have a certain amount of impurity. The impurities are relatively small. Chemically pure hydrofluoric acid will contain about fifteen thousands of one percent of iron. The magnesium chloride itself will contain a trace of phosphates a very small amount of sulphate which is lost in the heating operation, a very small amount of calcium which is calcium fluoride, a very small amount of sodium, about .004%, which forms traces of sodium fluoride. It contains a small amount of ammonia and a small amount of nitrate which are lost in the heating operation. Of course, all of these impurities that are not lost remain residual in the finished fluoride. Yield is relatively low. It is about 20% of the chemicals used. The operation is rather slow and tedious. It requires a lot of the platinum, a reasonably good laboratory and good exhaust system for getting rid of the hydrochloric acid. We use one mol of magnesium chloride to 2 mols of hydrofluoric acid plus an excess of about 15% so as to always insure an excess of the acid.

There is no critical temperature in the operation. It is a question of first evaporation of the mixture that is present in such a way that an excess of spattering will not occur. Then you will get a very gelatinous mass in the dish and apply heat at which time you are very apt to get severe spattering. The temperature is equivalent to starting temperature of a low Bunsen flame and the highest temperature is the temperature of a full Bunsen flame and is determined by observation by the eye. The final temperature in an electric furnace is very carefully controlled at 850 degrees Centigrade.

Dr. Lvon: What I wish to stress is the necessity for using Naval Gun Factory pure fluoride. When I first started on this work only the commercial samples were available. Invariably they resulted in poor film. They simply did not give the reduction in reflection which a good film should have. Now, we didn't make a complete survey throughout the country of all possible suppliers of fluoride and it is possible that some of you may locate a suitable source of commercial fluoride. We guickly found out, however, that if we were to have an unfailing supply whose characteristics we could be certain of, we would have to develop a method of making it ourselves. Mr. Wallace has just described that method to you. Therefore, I earnestly beg you not to bother searching the country for commercial fluoride. Either make it yourself or purchase it from someone who is already making the

pure fluoride. You will save yourself much grief and probably a lot of unnecessary work.

The instructions for preparing this material were first written in October 1942 and approximately 50 copies have been distributed. These are being revised to include a few refinements in the test which have been found advisable since the first writing. If anyone is interested in obtaining copies of those instructions, I will be glad to send them to you if you will give me your name and address on a piece of paper at the end of the meeting.

**Mr. Mattern:** *Minneapolis-Honeywell* 

When we decided to go into the coating of optics we made a few decisions and one of those decisions, arrived at after our inabil-

ity to secure magnesium fluoride of such purity as can be used for filming, was to manufacture our own. We have investigated several methods of producing fluoride but I shall discuss only two of them. There is a third method, that appeals very much that I will mention but I will also mention the pitfalls of that method. It is a method in which you do not have to use hydrof-luoric acid.

The first method that we investigated used magnesium oxide as a starting point. I think that method is being used by some of the manufacturers of fluoride, and it is rather a simple process of boiling the magnesium oxide with an excess of distilled water to produce the hydrate  $\mathrm{MGOH}_2$ . This hydrate is then treated with hydrofluoric acid to form magnesium fluoride. All that is necessary if your chemicals are pure to start with is then to evaporate to dryness and follow by ignition, as has been brought out, at high temperature in order to produce the finished product.

The second method and one that I mentioned a few minutes ago is based upon the combination of magnesium chloride and sodium fluoride. Both are soluble in water and this product can be made in glass—not platinum. For those whom are interested in equations, I feel I should put this on the board. This material is easily filterable and therefore the sodium chloride can be easily washed out of the filter. I wouldn't advise taking too much of this because I will show you a side reaction that is rather interesting, but defeats this process. On the face of it, it looks very feasible and appeals because you get away from the handling of hydrofluoric acid and the necessity for a hood. It can be made in most any clean atmosphere. I shall endeavor to show you a little bit of what does happen though—on a side reaction. (Mr. Mattern will supply the chemistry upon request.) This balances the equation. This is the salt that is actually produced and although it has quite excellent filming efficiency, it is unfortunately too soluble

in water. Therefore this process, although it did appeal, was abandoned and we finally adopted the same process that is used by Dr. Lyon. We use Baker's special crystal reagent magnesium chloride and then we crystallize it to make sure that we have as pure a product as we can commercially produce. The hydrofluoric acid is Baker's special hydrofluoric acid reagent. We have scoured the country and feel that this is the purest hydrofluoric acid that we can obtain. After producing the gelatinous mass in the platinum dish we have deviated a little from Dr. Lyon's process in that we dry carefully at about 110° C. To accomplish this, we place it in an oven at the drying temperature for several hours, never allowing the temperature to rise much over 125° C. There is a definite reason for that. We have found that when the fluoride is energetically heated and by that I mean to about 1000° C., it actually breaks down chemically. This may be determined by noting blue litmus turn red when held in vapors given off while the fluoride is kept at this temperature. We have seen this happen for as long as 4, 5, or 6 hours of heating. Muffle heating must be carefully done or the result is a mixture of magnesium oxide and magnesium fluoride. Therefore the last heating—the high temperature heating, to remove the last traces of occluded water vapor—is performed in a vacuum chamber, the pressure of which is held to about ½ micron while the temperature of the fluoride is raised to about 1500° F. We have found that by this method we are able to produce fluoride that not only is free of magnesium oxide but fluoride that is dry. In the discussion yesterday it was brought out that some of the sputtering or spattering was caused by adsorbed water. By following this method the fluoride is completely dehydrated and spattering eliminated.

Before I close this discussion I want to bring out a point that possibly was missed yesterday in the discussion that I headed on our method of coating and holding glass. That was that the fluoride was molded into pellets, the pellets varying in length from about 1/8" to 1/4" and a diameter of 1/8".

#### DISCUSSION

**Mr. Wallace:** You mentioned the decomposition at 1100 degrees Centigrade. That's a little bit difficult to understand because the melting point of fluoride is 1376 degrees Centigrade under atmospheric conditions and it's possible that the evolution of hydrochloric acid or chlorine or fluorine that you might have observed could possibly have been caused by a lack of heating prior to that last baked cycle. That's a possibility. I don't think you would get any decomposition before it got to the melting point.

Now, the question of adsorbed moisture in relation to sputtering. There are many ways which sputtering can be avoided. Some were discussed yesterday. One gentleman, I think from the New York Navy Yard, discussed the use of a shield. That has been used by a number of people and has been very satisfactory. Other people use the molybdenum filaments. That is the method we have found to be the best. Use a relatively wide filament of molybdenum about eight thousands of an inch thick. We get a very thin layer of fluoride that melts and diffuses quite rapidly, and films very rapidly without sputtering.

**Dr. Lyon:** Mr. Mattern spoke about preparing fluoride from the oxide. That method was the first one which we tried. It failed because you start with an insoluble product, magnesium oxide, and the magnesium fluoride which is formed as insoluble in hydrofluoric acid. Consequently, there is a perfect opportunity for superficial reaction to occur in which a little particle of magnesium oxide becomes surrounded by a thin protection layer of magnesium fluoride. Then action will cease. The product may produce beautiful films two or three times but after that, it begins to fail, and finally no coating whatever can be formed except possibly a dull, gray deposit made up perhaps of some impurities. Nothing but oxide remains in the crucible. That is why we discarded the method of making it with the oxide. We did not try boiling in water to change it to hydroxide first but started with pure oxide.

Mr. Mattern spoke of decomposition when the fluoride was melted. We have held a pound of magnesium fluoride at 2700 degrees F. i.e. 1500 degrees Centigrade, in an induction furnace for about one hour. It is a colorless liquid at this temperature. After cooling, it was ground to a powder and used with perfect success. It never gave any spattering. There may be a little decomposition to magnesium oxide at this temperature but the oxide will not evaporate at any temperature less than 2000 or 2200 degrees Centigrade which is far below the evaporating temperature of magnesium fluoride.

**Mr. Mattern:** I possibly failed to state that the reason we have not adopted the magnesium oxide method was just as Dr. Lyon has brought out, that even though we were successful in producing the fluoride and have acceptable fluoride by the method of hydrating the oxide by boiling, the danger of some of the oxide not being hydrated was enough to cause us to discard that method. We went to the hydrofluoric acid method. Dr. Lyon has brought out in his last remarks a very important point with us. that is that oxidation of the fluoride by chemical breakdown first forms magnesium oxyfluoride and finally by further oxidation the magnesium oxide. Whichever occurs it will be detected upon evaporation of the fluoride leaving the oxide behind. That I agree heartily with, but by the mass method of coating that we are using no magnesium oxide is permissible for the simple reason that it throws our calculations entirely out. We must have pure magnesium fluoride. We are not willing to take the chance of producing it any other way than the one we have described. We are quite sure then that we have pure fluoride and by the way, we do not have a good method of determining the amount of magnesium oxide in the presence of magnesium fluoride. I would like suggestions for making this determination.

**Mr. Tyler:** I would like to say that we have definitely shown the presence of magnesium oxide in high temperature baked magnesium fluoride by X-ray methods, something like 10 to 20 percent, and which may be due to our baking technique. I would like to add my own observation. I think, Dr. Lyon, the magnesium oxide doesn't evaporate. I would also like to bring this thought in, that if it is true why do we use platinum in order to manufacture magnesium fluoride, because the only determining agent would be silicon dioxide, an end product which is also quite difficult to evaporate.

**Mr. Denton:** We had some fluoride which when analyzed showed about 5% of silicate when analyzed after operation the silicate wasn't there. I don't know where it went to. It definitely didn't show afterwards. Another thing that analysis showed you—you shouldn't get brass too close to fluoride when it is going to be hot in your jar. We had those things in a very appreciable quantity—half percent of zinc afterwards. In reference to chloride I think there is some latitude in the types of CP magnesium chloride you should choose. Every time we have tried to get away from CP we got into trouble. No matter how small a coating plant you have, from a standpoint of cost, you want to test each batch of fluoride and to make larger batches. If you can make five times as much

fluoride per batch you will save money, because labor cost is the highest cost of producing it.

**Mr. Been:** I just wanted to confirm Mr. Denton's observation. We had a batch of fluoride we had absolutely no bad results from. Then we ran out of that. We had another batch of fluoride which gave us some spattering. We had that analyzed and found a high percentage of silicates in it.

**Mr. Wilson:** I would like to ask for discussion of members of the panel considering proper methods of storage of fluoride in the coating laboratory of the various lens producers. I think that is very important and pertinent to the success of the coating operation. I would also like to make a suggestion that those people who are manufacturing fluoride send to those of us who are buying fluoride already prepared, ampoules of the fluoride of approximately 25 grams each. They would be sealed off and under reduced pressure to prevent contamination by adsorbing moisture from the atmosphere. I believe that the additional cost effected by such a means of storing this product would be well warranted by the success. I would like to suggest that for discussion on the floor.

**Dr. Lyon:** I didn't know there was any problem of storage. We usually put the fluoride into a bottle which holds approximately 450 grams or about I pound. It has an ordinary bakelite screw cap cover, and is kept in the laboratory or in the coating room. When more fluoride is required, it is poured directly from the bottle into the crucibles. I don't know how much importance to attach to the idea of adsorbed gas or water. It seems to me that adsorption is a phenomenon which will occur almost instantaneously. As soon as the fluoride is removed from a vacuum jar, and comes in contact with atmospheric air, and the air has a sufficient time to diffuse through it, water will adsorb on the surface of the fluoride. Magnesium fluoride is not a hygroscopic material. It doesn't dissolve in its own water of crystallization which it picks up from the atmosphere, so I am not convinced that spattering is caused by adsorbed water.

**Mr. Wilson:** I agree with you that the logic as you have just illustrated is probably correct. However, we have made very careful check in Milwaukee. It may have to do with our climate but our data has shown on days of relatively high humidity we seem to encounter a great deal more trouble with the spattering in the jar, and we find also that any supply of fluoride which we pur-

chase and leave unstoppered for a period of time will eventually give more and more trouble as that quantity remains on our shelves being stoppered and unstoppered as it is being used.

**Ensign Storms:** I have exposed fluoride to synthetic sea water at elevated temperature for as long as a week and found perfect success with it—spread it out in a thin film, so apparently that's not your trouble. It's something else. It is understood that others have stored it in open containers at humidity of 70, 80 or 90% and have found no spattering. Mr. Mattern, can you give me an idea of the extent of that side reaction?

**Mr. Mattern:** Unfortunately, I cannot give you that for the simple reason we were pressed for time to produce fluoride and therefore we discovered that due to, no doubt, the almost same solubility of the two salts the reaction could very easily take place. We abandoned the method immediately because the solubility of the salt is about 2.6%, so that it would be a very unsuccessful material to use in a film requiring a high resistance to water.

**Ens. Storms:** The possibility occurred to me while sitting here although it may be an entirely unfeasible method of course, that you might get rid of your soluble compounds electrically.

**Mr. Mattern:** We discussed all of those points. It was agreed that possibly that could be licked but we didn't carry it down to perfection. It would result in a very nice efficient material for coating.

**Ens. Storms:** You have to get rid of your sodium compound.

**Mr. Mattern:** Yes, you do because of solubility.

**Capt. Dawson:** If you will examine Mr. Mattern's jar up here you will find the outlet has been attacked by hydrofluoric acid. In his jar it is evident that he is successful in drawing off hydrofluoric acid from his fluoride thereby eliminating what gives a great deal of trouble to the other methods. He has a very good fluoride which is difficult to produce. Mr. Been brings out the use of shields. We use good shields and good operators. We have not been able to avoid dangerous spattering when we have had bad fluoride and by dangerous spattering I mean every now and then we get fairly heavy rejections in a jar where we have critical items. We don't think shields are the answer. We believe as Mr. Mattern said, it is very well justified in making good fluoride and we would

like to see one or two agencies set up to make fluoride by these very careful methods regardless of the expense.

**Mr. Snyder:** On the subject of oxide in the coating material, in some experiments we have produced oxide not because we wanted it, but because that was the thing that came out. We have actually been able to get an oxide coating, so that being the case, if your temperature in the bell jar is sufficiently high, you will get some mixture of oxide on your coating. On the subject of moisture, I think that the trouble from the spattering there, is probably more an entrapment of moisture than adsorption from the atmosphere, so that the holding of the material in an ampoule or something of that type won't be of particular advantage. If the fluoride is properly prepared so that you don't have entrapped moisture, you won't have spattering from it.

### Inferior Films and Their Causes



**Dr. Lyon:** Most of the causes of poor coatings were dis-Naval Gun Factory cussed in a general way this morning. (1) the use of poor fluoride. Impure fluoride will not give a reflection reducing film which is efficient. (2) Improper cleaning or insufficient cleaning will result in blotches from which the coating is easily removed in subsequent cleaning operations during final assembly. Finally (3) poor vacuum. I think these are the three main causes for poor coating. Spattering, of course,

results in a poor coating in that it produces specks. I do not consider spattering in the same category as a poor coating.

A good indication of the vacuum in a machine in addition to the gages is the condition of the fluoride. If you find that the fluoride develops a very black deposit on the surface, it is usually caused by one of two things—(1) Poor vacuum—that is leaks or (2) Burned oil. Both have been found to give a real thick black deposit on the surface of the fluoride. The fluoride should remain almost white. It may develop a slight grayish tint on the top which is unimportant. If the oil is burned out it will manifest itself in two ways. If you happen to be using a glass pump you can see it very readily, because the oil will give off phthalic anhydride which crystallizes like small chestnut burrs or little white specks about one-sixteenth of an inch to one-eighth of an inch in diameter. In a metal pump you can not see these crystals and you have to depend upon smell. Burned oil will develop a very bad smell. The vacuum system will also become sluggish in operation. You will not be able to get the vacuum up to that required for coating in a reasonable time. It is possible to use burned oil provided it hasn't been burned too much. The final criterion for determining whether oil should be changed or not is whether successful coatings are being produced in normal coating time. Cleaning, I think, has been thoroughly discussed already.

Mr. Pettus: It seems that of all the discussions which we have entered upon here, this one is the one to stick your neck out the farthest, and I think it is one in which we are vitally interested. I have been trying to think of a definition for poor coating and these words sort of strike me as being useable. Poor coating is one which does not support itself to the end use of the product to which it has been applied.

Now, I would like to break the poor coating down into two classifications. One—poor adhesion and the other one—poor physical construction. Please note that I use poor adhesion since I can not see the word "softness" or "hardness" applied to microscopic films directly. I think that is just a common usage which we all make and at the same time understand. There appear to be at least three good reasons why a film cannot be produced having good adhesion. Namely, poor or improper cleaning of the glass surface, incorrect vacuum range for evaporation and just plain bad material. It is obvious that we as engineers or process men would not expect to paint a piece of metal which was covered with an oily film and expect the finished appearance of that paint job to be good. Secondly, we would not expect to do a firstclass painting job if our spray apparatus was not functioning in the proper manner, and third, we would not expect to do a good job again if we didn't have the right kind of paint. Those are simple phrases which I think can be applied to this case. No doubt film thickness is probably one which causes considerable rejections of the finished product, and it is quite apparent that some type of exact control must be employed if variations in film thickness are not to exist. Possibly, we can eliminate a number of these controlled methods and settle upon either photo-electric measurements, fixed time or fixed quantity of evaporation or the visual control. Probably the latter one is the one which is most commonly used. Although we at RCA have firmly set our sights towards a range whereby we expect to see fully automatic equipment in the not too distant future, there is also the question of uniformity and rejections from non-uniformity which contributes along many of these problems. In applying a film necessarily some method must be used to produce a uniform field of evaporation and as we have seen demonstrated here previously, there is in use various types of jigs as well as the compensator which we have developed.

The application of a film over another film which might be tarnished or stained is also a very common cause of rejection. Although we know that some small off-color regions of the coat on the lens does not necessarily make it a complete failure it should not be taken just as an excuse and the blame placed on that beyond our control. In view of these reasons for poor coating, I believe that most of our problems are to be found in our own backyard, and can easily be solved by rolling up our sleeves and digging in.

**Mr. Wilson:**Perfex Corporation

The two speakers before me have summed up the typical causes of poor coating and I have a few additional remarks to make.

I would like to direct our attention to the study and consideration of stains. We have discovered in our plant that certain of our optical elements will come from the Polishing Department showing slight stains which are almost invisible before coating and which will be very perceptible and marked after the coating has been laid down upon the glass. This type of stain has proved to have its origin in the polishing and is usually the result of water creeping in between the pitch and the glass when certain areas of the lens surface become separated from the pitch. We have eliminated this stain by carefully controlling the Ph value of the water and also improving our pitch composition in such manner as to prevent it from breaking loose from the glass in spots. The control of temperature in the room where the polishing operation is carried on is also important in preventing the glass separating from the pitch. A careful check of the physical characteristics of the pitch by means of an interferometer which we have constructed in our laboratory has eliminated a great deal of this trouble.

Some of the causes for poor tenacity of optical coating are chiefly eliminated when the pumping system is kept in perfect operating condition. The use of the roughing pump to assist in rapidly reducing the pressure within the jar has proved of great value in high production of coated optics. The roughing pump eliminates the passage of moisture laden air through the diffusion pumps and in this way preserves the low vapor pressure quality of the diffusion pump oil. A careful check should be given the pumping system at least once each working day and if the cycles of operation show a tendency to slow down, an immediate check should be made to determine the cause of the slowing down of the process and this cause should be eliminated.

Those of us, such as RCA, the Perfex Corp., and others who are using the low temperature process without heating in the jar, feel that we have eliminated the causes for poor tenacity when we have consistently coated optics at pressures within the jar ranging from 3 to 5 x  $10^{-5}$ . If the low temperature process is used, it is absolutely essential to use electronic bombardment within the jar in order to completely degas the surface of the glass.

I wish to call attention to the degasing of the fluoride before the evaporation process is carried on. Several satisfactory ways of doing this have been developed. Perhaps the safest method is to employ a shutter device which may be allowed to cover the fluoride boat or filament during the time of the pre-heating of the fluoride which is necessary to bring it up to evaporation temperature. The operator will open the shutter and expose the evaporating fluoride to the lenses only during the moment at which the coating is being done. The pre-heating of the fluoride is very essential and some samples of the fluoride may exhibit a tendency to evolve gases and spatter with even more violence than will be exhibited by other samples. The manufacturer will have to depend upon the judgment of the operator to give the fluoride a proper pre-heating treatment.

During this conference we have used the term "sputtering" to describe the violent reaction which fluoride often exhibits during the heating treatment. I believe this term is incorrect because the term "sputtering" has been usually used to describe a method of transposing metals from an electrode to some other surface under conditions of reduced pressure. I believe we would do well to use the word "spattering" in describing the fluoride reaction to heat.

Our Inspection Department has called our attention to certain rejected lenses which show a certain unevenness of coating and we have determined that the causes of this unevenness of appearance has been due to a sputtering action of the high potential electrodes which are used in the ionic bombardment of the glass lenses during the pumping cycle. If any of you are to use the electronic discharge method for cleaning up the jar, it is advisable to employ electrodes made of pure magnesium metal and these electrodes should be cleaned once a day with steel wool. An accumulation of copper and fluoride will occur upon the high potential discharge electrodes and a sputtering action will cause these deposits to be transferred to the lenses. On some occasions, we have noted clearly the shadow of certain pieces of equipment in the jar to be shown upon the lens holders and the outline of the shadow will be clearly marked by a deposit of the sputtered material.

Tin plating or cadmium plating of parts within the jar will greatly facilitate the cleaning of the jar and aid in the constant and rapid reduction of pressure.

Many of us will have occasion from time to time to recoat cemented doublets which have been rejected because of poor coating. The process of separating the doublets often leaves a residue of balsam or cementing plastic material upon the ground edges of the lens. This material is often not removed by the regular cleaning processes which are applied to the lenses in preparing them for the evaporation jar. It has been proved in our laboratory that it is absolutely essential to remove all traces of balsam or plastic material from the lenses. This can best be done by

actually scrubbing the edge of the lens with a piece of cotton which has been saturated with a suitable solvent material to remove the balsam or plastic. If a particle of balsam or plastic is allowed to enter the jar, the pumping time will be considerably increased and the coating laid down upon the lens is most likely to be lacking in a sufficient tenacity and will, therefore, not pass inspection. One lens which is contaminated with balsam may actually ruin the coating laid down upon a large group of lenses that would be located in its neighborhood. May I again stress the importance of prolonged mechanical scrubbing of the rim of the lens with a suitable solvent in order to remove the last traces of cementing compounds. If the cold process of lens coating is used, cemented doublets should also be cleaned carefully to remove traces of residue balsam or plastic compound from the rim. It is essential, however, that in the cleaning process the solvents used do not enter into the cementing compound which lies between the lenses because when this occurrence takes place, the splitting of the lenses will be noted to occur within a few days.

#### DISCUSSION

**Mr. Raines:** Frankford Arsenal found in making mirrors rather than films that some days when you start your pumps you get a cloud in your chamber. When that occurs we have never been able to make a decent mirror. The solution is to start pumping more slowly.

**Mr. Wilson:** In answer to the statement just made, I have never seen our pumps turned into the jar without the production of a Wilson cloud. I will agree on some days it is worse than others and particularly if we are to employ an auxiliary roughing pump which will very quickly reduce the pressure in a large jar of the size illustrated upon these units. We find sometimes the cloud will be dense enough to form water that you can see condensing upon the glass. I believe, however, in most cases the evaporation of that material at least in our experience has proved to be completely satisfactory without really encountering any difficulty in the final result. Does that agree with your experience, Dr. Lyon?

**Dr. Lyon:** Yes, exactly. A large pump will always produce this condensation of the moisture after the first few revolutions but it disappears quickly and we have been successful in coating all optics with low reflection films as well as aluminizing without any difficulty from that source.

I also wish to corroborate what Mr. Wilson has said about re-

moving the balsam from the lenses which have been un-cemented. There is a tendency to leave a thin film of balsam over the surface. This balsam film is usually invisible, but a fluoride film will not adhere to such a surface. Our procedure in cleaning such a lens is to wipe it with a small piece of cloth wet with acetone. This cloth is then discarded and the process is repeated using a clean cloth. This procedure is repeated four times, after which the lens is subjected to the regular cleaning technique which has already been described.

I also wish to say a few words about the use of the glow discharge for cleaning the optics in the vacuum. A glow discharge is of no benefit whatsoever when the optics are heated in the vacuum by the high temperature process. It can well be discarded in this case.

## Decoating



**Dr. Lyon:** Decoating has been a problem from the early days. At first we wondered how to make the films stick to the glass. Now, we wonder how to

remove them when an error has been made! Our results thus far can be summed up by saving that we know of no positive sure-fire method of removing a good hard film except by repolishing. The lenses have been boiled in dilute sulphuric acid, nitric acid, hydrochloric acid, a mixture of sodium hydroxide and sodium hydrosulphide and many others. To be sure, some of these will remove the films, but it also removes some glass, leaving the surface mottled and rough. I am speaking now of real hard films. People have tried a great many things and word goes around the country to the effect that a certain material will remove the coatings. We get this information at the Gun Factory and try it, and find that in some cases it does work. One of the most successful, I believe, has been boric acid, but Ens. Storms can tell you about boiling optics in boric acid for the better part of four days without having much effect on good hard films. This is a problem which is quite important because there are many times when it is desired to remove a film. If any of you have any suggestions which are positive in their action, I am sure it will benefit everybody.

**Mr. Tyler:** Well, I am sorry I can't give you a sure-fire method for removing magnesium fluoride films. All I am going to try to do is to present to you a

report on a process that so far has been incompletely investigated and probably not ready yet for production. Of course, before the introduction of the new high temperature baked or super-hard films, decoating was a fairly easy problem. A little calcium or rouge or cerium oxide or anything almost would remove the coating. I can remember some that you could even blow off.

With the introduction of so-called high temperature bake films these methods become inadequate. Our first experiments in decoating were very misleading. We tried two or three lenses in any one solution and would have excellent results on these two lenses. With our hopes high, we would go to a larger batch of lenses only to find that the two we originally tried to decoat were apparently soft and the larger batch contained a greater percentage of hard lenses. I imagine that a great many others have

had this experience too. We have tried four different groups or classes of materials. First group I call salts. Sodium chloride at 15% cold solution gave no action. We used a saturated, hot solution with very erratic results. We tried potassium cyanide ten percent solution and obtained etched lenses. We also tried potassium cyanide saturated with ammonium chloride, ammonium sulphide, ammonium nitrate, and ammonium carbonate with incomplete or unsatisfactory results. Our work with the strong acids, that is sulphuric, nitric, and hydrochloric, check the results of Dr. Lyon. Nitric acid five, ten or twenty-five percent solution gave practically no action cold, whereas fifty percent or concentrated hot gave too much action. Hydrochloric acid is particularly bad in that it attacks the lenses and produces whitening of the surface. Sulphuric acid gave the same results both cold and hot—very poor in action. We followed this by mixing both acids and salts. We have tried, for example, sulphuric acid with ammonium sulphate nitric acid with ammonium nitrate and hydrochloric acid with ammonium chloride with equally unsatisfactory results. We finally tried the weak acids which are phosphoric acid, boric acid and acetic acid, and we have had. I am happy to say, reasonably good results with these acids.

The technique we have developed for using phosphoric acid looked particularly promising. In using this acid there are several more factors. We take 85% phosphoric acid which is commercially available and mix it 50-50 in distilled water and then add a small amount of ammonium phosphate. The presence of the ammonium phosphate is not mandatory but it does seem to help a little bit. Very weak phosphoric acid tends to produce etched films such as RCA has been working on in the past. Stronger acid solutions are simply wasteful. Actions are relatively slow and we use boric acid if necessary. The temperature of the solution is extremely important. We found that 80° Centigrade is easy temperature to obtain. We put the phosphoric acid in a water bath and heat the water. If the temperature drops much below this, the action becomes very prolonged disproportionately with the drop in temperature. Circulation of the solution around the lenses is also important for two reasons. One to speed up the action and secondly, to prevent possibility of etching which sometimes occurs. In removing the lenses from this hot solution the obvious precautions must be taken to prevent heat cracking or heat straining. We transfer our lenses from hot phosphoric acid to hot distilled water and where necessary to colder solutions and finally to alcohol. The drying, of course, comes next and we found we can dry simply by wiping with Kleenex or a clean rag.

The alcohol step is something I would like to elaborate upon.

We found if you simply decoat in phosphoric acid and then rinse in distilled water, perhaps two rinses, and then attempt to dry immediately, you get a pronounced water stain which is difficult to remove after it is once formed. We also found in a great many cases we can prevent this water staining by removing the water with an alcohol bath ahead of time. I was going to say it requires about an hour decoating in phosphoric acid solution, but that is going to give the wrong impression because some lenses will decoat in five or ten minutes and some won't decoat for two days.

Recovery, as nearly as we can estimate at the present time, is about 75% and the rejects are principally for stain. We have had a few examples of etching.

In using boric acid the same general factors apply. The concentration, I believe, we have used and I think most everybody else has used, is saturated solution. The temperature again should be around 80° Centigrade with boric acid. Drying does not seem to be quite so much of a problem. We haven't at the present time used any alcohol in drying operation. Again, decoating requires about an hour and recovery is 75% or thereabouts, and the rejections are principally for stain.

As far as acetic acid is concerned, we have only done preliminary work. We have used concentrated phosphoric and boric because of the ease with which they can be handled, and because they are solid acids. Any of these weak acids will tend to etch and precautions must be taken to prevent this if good results are to be expected. Circulation, I have already pointed out, is an important factor and there is one other factor that may be of interest if any metal part of the lens holding jig comes in contact.

Before closing I would like to make a few remarks about recoating decoated optics. After all, the final test of any decoating process is whether you can recoat those lenses to advantage. We have recoated only a few, proportionately speaking, of the lenses we have decoated, but we found that decoated lenses will coat to the same color as new lenses within very close tolerances provided no faint brown stain is visible on the decoated surface. If this faint brown stain is visible and sometimes it is very hard to detect and very uniform, the lens will coat to a slightly darker or bluer color. That is to say a new lens would be red-purple; whereas a decoated lens would be blue hardness.

I think I should also say that so far we have only done decoating on two kinds of glass. I am passing this process on to you in hope that you will do something with it.

#### DISCUSSION

**Mr. Denton:** We tried many solutions but failed to get them off. We did find that by taking a very fine rouge and rubbing with clean absorbent cloth, if you do it properly, you can rub the stain off. The one caution is not to rub on the stain but to rub over the whole surface; to cover the whole surface every minute or two, and that does not sound like good procedure but it does work. The only other way is to do it mechanically and rub them off by hand.

## Durability and Hardness of Films



**Dr. Lyon:**Naval Gun Factory

The discussion which has just closed showed the differences which can arise because of surface conditions. The effect of heating has been

shown to produce a cleaning action on the glass, but this is not the only effect. By heating the glass to approximately 200° Centigrade, which is about the limit required to produce good films—most of the water which is adsorbed on the glass surface is driven off. The final molecular layer is not driven off until 350° Centigrade to 400° Centigrade has been reached, however. This cleaning action probably accounts for some of the increased hardness which is noticed with a baked film because it adheres better to the surface.

There is another effect of heating, however, which is much more important. It is a sintering effect on the film itself, which will explain why films heated in a vacuum are invariably harder than those which are heated to the same temperature in the open air. It has been established that these films are not single homogeneous crystals but that some materials, for example, calcium fluoride, will deposit as individual microcrystals tightly packed together but with definite spaces between them. These microcrystals have the shape of laminae or thin plates like mica. As soon as such a film is removed from a vacuum chamber, water adsorbs on the surface and proceeds to work its way down between the crystals. Now, if such a film which it covered with adsorbed water is heated in an oven in the open air, the adsorbed water reacts with the MgF atom and releases hydrofluoric acid. The OH radical remains attached to the magnesium atom. Every minute space between the crystals is therefore lined on both sides with OH radicals. This combination of atoms is very stable to heat and so the laminae cannot coalesce to form more perfect crystals of fluoride because they are separated by the OH radical. If. however, a film is heated in the vacuum before water has had a chance to adsorb in all the millions of minute interstices among the crystals, then the laminae can coalesce to form more perfect crystals of magnesium fluoride. The film is therefore harder. This explains, I believe, why heating in a vacuum results in much harder films than heating a film in the open air in an oven.

**Mr. Jewett:**National Research

Heating lenses to a high temperature before and during coating unquestionably results in the production of hard coats. How-

ever, I am not exactly sure what the mechanism of the process is. One theory states that water vapor is driven off during heating, but if lenses are heated to the required temperature and then allowed to cool in the bell jar under vacuum—thus preventing the acculturation of water vapor on the surface—a thin film applied to such lenses will not be hard.

**Dr. McRae:**Eastman Kodak

The first thing that occurs to me is that Dr. Lyon's remarks about the beneficial effects of heating when the lenses are still in

vacuum, applies to baking after the coating has been put on. I would still like to know what the beneficial effects are before the coating is put on. We have also noticed a very interesting effect in that some of these coated optics coated with real hard coatings apparently have a greater hardness. Some of these coated surfaces on some glass will endure 100 grams, and we have gone as high as 400 grams on a base that has been coated with hard coatings. There is, of course, a great, difference in different types of glass.

We tried a lot of schemes for hardening coatings and among others, we tried baking after coating. We took them out and put them in an oven and baked them and found some success. But I would like to confirm a statement that has been made here a few times this morning. I think cleaning is the biggest thing. I think that is the biggest step forward in the whole process. We have noticed one other effect on hardening, and that is we feel we have some evidence that the rate of evaporation of magnesium fluoride has some effect on the hardness of the film. Some people are using 15 minutes for evaporation. We use about two minutes, and we find that seems to be better than the longer period. That requires that the magnesium be well degased.

**Dr. Lyon:**Naval Gun Factory

I wish to make some comments on the last remarks by Dr. McRae. We have tried heating the glass and then allowing it to cool to room tem-

perature before applying the coating. The coating was then applied cold. The film so produced is so inferior in hardness to one which is deposited while the glass is hot that they are in completely different classes of films. Hence there must be something besides the cleaning action of the heat on the surface of the glass which will account for the extra hardness of the baked film. It is my belief that the extra hardness results from the sin-

tering action of the heat on the film.

I also wish to comment on the effect of the rate of evaporation on the hardness. I believe you stated that your experiments indicated a harder film with a rapid rate of fluoride evaporation. The theory, as well as experiments with calcium fluoride, lead to the conclusion that the slower the evaporation, the harder the film. It will undoubtedly be impossible to determine this effect on hard baked films because no test would be sufficiently sensitive to separate it from all the other variable factors. It might be done, however, on unbaked films.

**Mr. Denton:** There was some discussion by Dr. McRae and Dr. Lyon about the hardness of coats varying due to the rate of evaporation, and I think there is a little confusion there. If you want to increase the rate of evaporation there are two ways to do it; one way is to increase the heat through the same filament and thereby put more heat into the fluoride, and the other is to increase the heating surface. I think Dr. McRae was talking about increasing the heat through the filament, so that he has a higher temperature filament and should get a harder coat. If he had merely increased his heating surface, he could actually have had a lower temperature and not obtained a harder coat.

**Mr. Quin:** I wonder if Dr. Lyon would care to comment on the question of tenacity and hardness? We know that in the cold coating of calcium fluoride you can get a hard surface, but if you add some moisture to it, the thing wipes right off. Therefore, the question of tenacity does in a way bear a relation to some of the remarks just made.

**Dr. Lyon:** I don't know how to separate tenacity and hardness when talking about a film which is only four one millionths of an inch thick. Undoubtedly both effects are present. The explanation for the observed effects of heating may be incorrect, but it certainly does fit the facts more closely than any other. The work of Dr. DeBoer of Holland shows that by heating evaporated films, more homogeneous crystal structures are formed which have fewer crevices among the individual crystals. This can be interpreted as an increase in hardness. It does not explain whether the adherence of film to glass is increased. Since heating has the effect of cleaning the surface, and it is common knowledge that a film adheres better to a clean surface than to a dirty one, it can be concluded that heating not only increases the hardness of the film itself but also increases the adhesiveness of the film to the glass. Both effects are so interdependent in such thin films, how-

ever, that it is probably impossible to separate them.

**Mr. Mueller:** We did find definite indications of extra hardness or durability by the use of an oven heating. We heat our lenses for about an hour at 500° F. after we are through coating them, and we have found definite indications that this does add to the durability of the coating.

Mr. Berlant: I have a suggestion that may help in getting a line on the effectiveness of heat. A very large percentage of those units which we coat are cemented doubtless, which of course, means coating without heating either in the vacuum or after the vacuum. They come out with a degree of hardness that is comparable on early handlings with those that are produced by high heat. You had the opportunity of inspecting some of them vesterday, and I think you can verify that. But we have made this observation that on exposure to unusually high humidity condition those coatings will degenerate to the point where they can be readily wiped off. We have found means of checking that, so that we are able to, by giving them conditioning, prevent them from softening to a point where they are not workable by the assemblers. There is no question in our minds but that the exposure to humidity in the period of several hours following the removal from the vacuum works a tremendous difference in their tenacity or hardness, and we think that if somebody will carry that investigation out, they may find a lead some place as to the effect of heat in the vacuum.

Dr. Lyon: Mr. Berlant has referred to the differences between a hard baked magnesium fluoride and one which has not been baked. It is a fact that a film which has been deposited without baking will appear to be very hard as long as it is kept dry. But the difference between the two coatings will show up where they are subjected to a humid atmosphere or are soaked in water. The unbaked film will soften and the reason for this behavior falls in line with the explanation already given. You will observe that when an unbaked film has been soaked in water, or subjected to high humidity over a period of time, the film becomes bluer, indicating an apparent increase in the thickness. For example, a drop of water which has been allowed to evaporate on an unbaked coating will either remove the film or leave a permanent bluish stain which indicates a thickening of the film. The explanation is that water adsorbs quickly on the outside surface but produces no apparent effect, but when sufficient time has elapsed for the water to diffuse into the small interstices among the crystals, a swelling of the film takes place as the water pushes down into the crevices. The crystals of fluoride have been separated and a permanent stain results because the swelling has increased the effective thickness. The film has now become much softer and can be removed with ease because the crystals comprising the films are no longer closely interlocked.

Dr. McRae: Dr. Lyon and one of the other gentlemen in the audience mentioned the fact that if you get back-coatings of the lenses that it indicates poor vacuum. We have had evidence of such coatings when we have our vacuum down to what is ordinarily very adequate, but we don't find that the coatings are soft or have poor adherence on the other side. There is a particular case I have in mind which is some of the large Navy prisms that we have to coat with magnesium fluoride on two sides and then silver the hypotenuse. We find when we get around to silvering that even though our vacuum has apparently been of the best, something has gotten on that hypotenuse, and must be polished off with rouge before the silver can be made to stick. Now lest we be accused of having poor technique I would like to point out that they are having exactly the same trouble down at Naval Gun Factory. We found them spending ten minutes polishing that off before they could silver. I was wondering if anything further could be said on that.

**Dr. Lyon:** That puts me on the spot, because frankly, I didn't know anything about it. I didn't know they were polishing after the prisms had been coated.

I ran the Coating Department at the Naval Gun Factory about three and a half months after we had gone into full production, and I left that department last February, so I am unacquainted with the exact details of what they do now. During that time, we coated about 600 prisms which required silver on one side. To my knowledge, they were never repolished before they were silvered. One trouble we had with large prisms was that the silvering operators used charcoal to remove the silver, which had run down upon the coated surfaces. The vigorous rubbing removed some of the film on the edges.

Another trouble which we encountered on large prisms were irregular blotches of variable size where the film would be removed after several cleanings. These blotches usually appeared near the apex. These blotches had all the appearances of being dirty glass prior to coating but nothing we did improved the situation. It may have had something to do with the heating of such a large mass of glass. None of these prisms were repolished be-

fore silvering and this back-coating is a difficulty which has been encountered since.

**Mr. Schroyer:** We also are working on prisms for the Navy Dept. and we have conducted several tests. We found that we have back surface coating regardless of the vacuum. We have run our bell jars as long as four hours before attempting to evaporate. We have checked our vacuum, found it adequate and analyzed the back surface coating. We had silvered the hypotenuse of the prism by chemical deposition. We found that where the glass had been silvered and the silver removed, it was easy to resilver but the section of the prism which had not been silvered had to be repolished before it could be silvered. This seemed to prove that there was back surface coating. We eliminated most of that by placing a masking plate on the hypotenuse side of the prism. Any fluoride which is deposited there could be very easily taken off with calcium carbonate and a felt polisher—just a few rubs would take off this film.

**Mr. Zook:** Dr. Lyon, I think we have noticed that a piece of glass which has been heated in a high vacuum, becomes hydrophobic and that may be why they have to repolish the surfaces in order to silver it; not because of the back-coating. That undoubtedly indicates that the surface of the glass has been altered by its exposure to high temperature and very low pressure.

**Dr. Lyon:** We haven't conducted any experiments on back-coating because it was never a problem. Experimentally, we have coated two sides of large prisms and then evaporated silver on the hypotenuse without encountering any trouble. There was no apparent back-coating previous to putting on the silver. We have noticed, as Mr. Zook just stated, that a glass surface which has been heated to a high temperature in the vacuum becomes hydrophobic or water-repellent.

**Mr. Wilson:** We have encountered no difficulty in production whatsoever. There is no indication of back-coating or of any interference affecting the coating.

**Mr. Zak:** We at Bausch & Lomb haven't had any back-coating but I wonder whether this back-coating you speak about is a partial film or an etched film formed during the grinding and polishing and work operations before they get into the filming operations. I think that's the basis of it.

**Mr. Tyler:** I can answer that briefly. You can scrape it off with a sharp pointed wire. Observe the effect under a magnifying glass.

**Mr. Mueller:** I think there is a little misunderstanding of what Mr. Tyler meant. In his back-coating he referred specifically to optics that are coated both sides and what he wanted to warn against was a soft coating being formed on the uncoated side at the time of coating the first side. With an insufficient vacuum there is not enough residual velocity left in the molecule to travel to the bell jar or to the heater unit, thereby impinging itself there in the form of dust. Therefore, we feel there may be a region of reversal in which the molecule comes back down and deposits very softly on the back side of the lens. Then, when back side soft coating is reversed and used as a base for a hard film it will not allow that hard film to cling tenaciously to the glass surface.

**Mr. Wilson:** If I might draw an analogy between the construction of an X-Ray tube and the interior of the jar with the hot cathode cooling type tube, we would be led to believe that that inclusion of electrons by a cathode would be at least shadowed by the other elements in the tube. We can prove that all parts in the X-Ray tube with all of the glass or metal are sources of radiation which must indicate that there is general travel and scatter throughout the tube of electrons removed from the hot filament. That analogy might be carried over to the radiation of magnesium fluoride in the film.

**Mr. Denton:** I want to acquaint you with other phenomenon we have observed. Just after the glass comes out of the vacuum, Mr. Scatchard who does our lens coating at the Arsenal, recently showed me what he could do to a lens at the time he lifted his bell jar up by just rubbing his hand over it. The reflected color would change towards the blue side of the spectrum indicating a thicker coat. Any kind of washing that we have done on such lenses would never bring the reflection thick to the same value as it was before. We have checked that by letting the lens cool down to make sure it wasn't just a heat effect.

When you finish coating, you have a film practically free from adsorbed matter. The minute that anything hits that film adsorption is going to take place and adsorption depends upon time. That is, adsorption is not instantaneous. Although air definitely hits the film first, if something is put on the films quickly while it is still warm and before air can completely adsorb, interesting adsorption effects will take place. For example, Mr. Scatchard tells me that for the past three weeks or more he has

been increasing the thickness of our light films by greasing them up so they would sneak up into the specified range. There seems to be no difficulty with that procedure. I would expect on the basis of the size of oily molecules that they could add practically one-tenth of the thickness of the whole film, whereas air molecules would be only one-hundredth of it and not increase it by any great extent. The difference in refractive indices would also help to explain. It may be, as Mr. Berlant stated, a good idea to put the right thing on the film while it is still warm. Inasmuch as the Army and Navy are both actively considering anti-fog agents for optics to reduce possible external or internal fogging moisture condensation it might be that the best thing to do would be apply the anti-fog agent promptly upon removing the optic from the vacuum jar. I think somebody should investigate that.

## Filming Cemented Optics



**Mr. Denton:**Frankford Arsenal

I will discuss the coating of cemented elements with high temperature bake process. We have done that successfully although I don't think

at present, that we were first ones to do this and we only did it a couple of days ago. We were able to deposit a good coat without harming the cement in any way. These elements were coated in connection with some tests on various plastic cements I have been making.

I found three cements which would stand  $210^{\circ}$  F. to  $-80^{\circ}$  F. indefinitely. They were subjected to  $210^{\circ}$  F. in a bell jar and two of them popped and the other seemed to work. I repeated that with perhaps thirty only the day before yesterday and over two-thirds of them came through practically untouched. With the others there was some difficulty. Many of those difficulties may be ascribed to the fact they were optics upon which I was experimenting—the surface not cleaned too well. At least they were old surfaces and hadn't been polished recently. We had no jigs with which to properly clamp the elements after cementing. Now, I have a few samples here which I am going to put on the table and you may look at them afterwards.

As must necessarily come out of this discussion the main thing about this is what is the cement and where can I get some, and is it any good or really not so good. As you say there may be other cements which will do this and we have at least one other cement manufacturer in the audience here who may know of something. Nevertheless, the only cement that I found that would do this among the ones I tested was one which is manufactured by Bausch & Lomb at present called PKR-15 by them. They make it from, I believe, resins obtained from Columbia Chemical Division of Pittsburgh Plate Glass Co. and I believe the information is readily available as to which one that is. Furthermore, I believe Bausch & Lomb are in a position to supply that, although I don't think at this time, anyone could guarantee that a consistent result would be obtained. For example, my test lenses must be supported in the rack absolutely horizontal. Inasmuch as we didn't get a hundred percent success in doing it, it is very likely that a lot of things will have to be worked out. Now, after I get through I guess Mr. Zak will answer any further questions on the cement itself. First, I'd like an explanation from the floor on this talk. Should any more work be done on this? Is anyone interested in coating cemented elements? Now, RCA probably is.

They get cemented elements and it's going to be difficult for them to coat them with the hot process.

Mr. Zak: We at Bausch & Lomb for quite some time wanted to do some work on cement, where we Bausch & Lomb could film after cementing and centering the optics. About two or three months ago we did some preliminary work along that line. We had indications that that could be done. I didn't know until I got down here yesterday morning that Frankford Arsenal was interested along the same line and they very probably have done more work and had more success than we had with the material. This developed as a result of the request of the Air Corps for cemented photographic optics to withstand -65° F. and up to 165° F. As far as this particular cement is concerned, we have carried on quite a few experiments, and learned that it will stand cold temperatures. It will also withstand elevated temperatures. We have had optics cemented and in temperature ovens at 100° C. for a period of one month, and so far as we could see there has been no deteriorations of the cement. We have had optics in bell iars at 400-450° F. that weren't entirely successful. They were not as successful as Frankford Arsenal has been. That is probably due to the state of polymerization. As far as the cement is concerned I don't know just exactly what the composition is. It's a glycol base material. We apply it cold in cementing. Then we true our optics optically and mechanically. We finish the polymerization of the cement at about 135° F. for a period of 12 to 20 hours, depending upon the size of the optic which you are cementing. We intend to do more work along that line and any more that we can find out we will be glad

We have been cementing lenses for the Air Corps Mr. Pittman: Eastman Kodak with low and high temperature cements for the past year and a half. Depending on the method that is used in the polymerization, these lenses will stand tests up to 400 or 500, and we even have tests that will take 600° F. to take the lenses apart. In normal production some mistakes are sometimes made, and it becomes necessary to take the elements apart, so therefore, we tailor-made our cements for certain temperature ranges, and I would agree with Mr. Zak that it would depend upon the polymerization then at these temperatures. That would be your guide in saying to what temperature you could raise them before they would fall apart. You would tailor-make your cement to stand 350, 375 or 400 and in that manner I think you would be able to coat your cemented lenses. I think

to contribute at later meetings.

probably most of the manufacturers are cementing them after either hard coatings or soft coating.

# Specifications and Standard Tests



**Mr. Raines:**Frankford Arsenal

It is our duty to insure that the Army will get coatings of suitable optical and physical characteristics. We must have films that will remain

suitable throughout wide ranges of temperatures, humidity, and stand mishandling either during assembly or by Army personnel after they are issued.

The increase in light transmission is perhaps the most important characteristic of the coating. The proposed Army Specifications, covering these coatings are here today, available for distribution to those who want a copy. This gives you in detail the transmission or increase in transmission to be expected from these coatings. It gives certain tests for tenacity and durability, which we use to determine if the coatings will retain their transmission after handling. It is not intended to burden the manufacturers with a multitude of tests on ordinary production optics, but it is considered advisable that you people know what the tests and requirements are, and be capable of making tests more or less as a check of your own quality.

On straight production optics it is the intent to reduce the measurements required merely to a measurement of color. This will be done by comparing the production optic with a set of standard samples, which will be prepared by Frankford Arsenal and distributed to the various Ordnance Districts concerned. We feel after having tested numerous coatings from many manufacturers, that if we could, we would want to insist upon the high temperature coating. You will notice that the specifications permit other types of coating to be used. The purpose of that is something of a compromise with necessity rather than any intention of accepting the coatings as the equal of the high temperature bake. Some of us at least hope that sooner or later will be able to eliminate that and stick to the high temperature bake entirely. I know that none of you have had a chance to comment on the specifications during those meetings last week. However, if after seeing the thing you have any specific questions to raise, Mr. Bechtold, Mr. Boydston or myself will be here the rest of this afternoon and we will take it up at that time.

**Dr. Lyon:** At present there is a movement afoot for the Naval Gun Factory Navy and Army to get together on a Federal Specification for reflection reducing films. This new specification will probably differ from the present Army Specification, but everybody is going to produce the same type of film. You are not going to use one technique or color for the Army and something else for the Navy. The Navy is going to revise its specifications, also, to bring it in line with experience which has accumulated since the first writing. It has been found that the color range which was originally specified is not the wave length range given. The color range was too narrow. That color range is going to be expanded. If and when a Federal specification is issued there will probably be more changes.

To those manufacturers who are just beginning, this continuous changing may be very perplexing, but if you will read over the specifications, you will see that it can be divided into three parts. (1) A section which deals with the optical properties, such as color and amount of reflection. Adherence to this section of the specification will be determined visually at the time the film is made by adjusting the film thickness to give a reddish-purple reflection. (2) Another section of the specification deals with the mechanical properties—or hardness and durability of the films. The third part concerns incidentals such as handling, packaging, and marking which are unimportant from the viewpoint of this meeting. These specifications may change completely.

It is obvious, however, that nothing will be written into the specifications which a manufacturer, using good technique, cannot meet. You should read the specification for your own information and then throw it to one side because if you use a pure fluoride, you are bound to produce a film which will meet the specification for minimum reflection at some point within the color range. The use of good fluoride will eliminate 90% of the trouble in meeting the minimum requirement. The other requirement is color, and if you maintain the color in the reddish-purple range, which I will discuss in more detail later, you cannot help but produce a film which will meet all the requirements of the specification with regard to optical properties. In other words, all you have to do is to watch for the correct color when the film is being made and use good fluoride.

If you wish to procure equipment to measure these properties, you can of course do so, but it will not be necessary. You will not need any fancy equipment to prove whether you are correct. Keep the color within the range specified and use good fluoride. If you pay heed to the requirements which were discussed this morning; that is good vacuum, sufficient heat on the optics, and clean-

liness, which are the fundamentals of good technique, you will automatically produce films which will pass all the requirements of any specification which may be written.

I don't know of anything better than a visual color test, which can be made conveniently on large quantities. The objective method of producing a film which will automatically meet the specifications is to incorporate a photo-electric cell into the apparatus which will determine the proper thickness as the coating is being applied. A photo-cell has been tested for this purpose at the Gun Factory and found to perform satisfactorily. The Gun Factory coating machines were therefore designed so that a photo-cell could be incorporated at a future date. At present, however, such equipment has not been engineered to the point where we can install it on our machines. Ultimately we expect to do so, and I pointed out yesterday that it might be worthwhile for the manufacturers to give some thought to its incorporation into their equipment to eliminate the human element in judging the color.

The eye, however, is very sensitive and furnished an easy and sufficient way of determining the color. The color range is so broad, the new specification actually ranges from a deep brown to a purple blue that a coating operator has considerable latitude for the exercise of judgment in the color which will meet the specification. Visual determination of the color is sufficient and satisfactory.

The present Navy specification, as well as the Army's, permits films whose reflections have minimum points within the range 450 millimicrons to 650 millimicrons—450 millimicrons corresponds to a film which reflects yellow red yellow which is commonly known as a deep brown. 650 millimicrons is way over in the deep purple blue or blue. It has been our experience that it is almost a universal tendency to over-coat or make the films too thick, with the result that the thicknesses of the films crowd to the upper limit or one even outside this limit. What are actually desired are films lying within the range from about 500 to 560 millimicrons. The reason for this is that the eye has its maximum sensitivity for daylight at 550 millimicrons, but at night with the illumination equivalent to that of bright starlight, the eye will shift its point of maximum sensitive to 510 millimicrons.

It is obvious, therefore, that the film should be adjusted in thickness so as to have minimum reflection and therefore maximum transmission—in the range from 500 to 560 millimicrons in order to obtain maximum benefit from the film. In order to correct this tendency to over-coat, the upper limit is therefore being deliberately lowered to 600 millimicrons. It is an effort to force the production of thinner coatings. A film having a mini-

mum at 500 millimicrons shows a red purple red color by reflection whereas one having a minimum at 560 millimicrons is purple blue purple in color. A film having a minimum reflection at 600 millimicrons will be purple blue. Hence the new specification will allow a color range in the films from yellow red yellow (deep brown) to purple blue. The desirable color range, however, is narrower (500 to 560 millimicrons) and goes from red purple red to purple blue purple. The distinguishing feature about the desirable range is that it includes all films which show any red in the reflected light. Everybody doing coating should make it their moral duty to produce films in the desirable range (500-560 millimicrons) rather than films which crowd to the maximum allowable limits of 450 to 600 millimicrons. This means that films should be reddish purple. The majority of the coatings should show a distinct red by reflected light but it is realized that not all of them can be made within these limits because of other disturbing factors.

**Mr. Boydston:** The details of all tests performed on reflection reducing films as applied to optical elements and other kindred materials by the Fire Control Laboratory, Frankford Arsenal are to be found in the following technical research reports: —

TRR-43-19	TRR-44-3
TRR-43-23	TRR-44-5

These are long detailed reports and it is neither desirable nor worth-while to describe their contents in detail today. My aim is merely to describe briefly the tests which any samples sent to Frankford Arsenal must survive before approval will be given to accept that film in fulfillment of your contract.

At present films for military usage are classified into three groups:—

**Type A** Metallic fluoride high temperature baked **Type B** Metallic fluoride low temperature **Type C** Silicate Films

These are desirable in the sequence in which they are given. (Editor's note: U. S. Army Specification 51-70-4A, 28 January 1944 no longer separates reflection reducing films into these three classes.) Type A films only possess the durability desired of reflection reducing films used for military purposes. This type failed the least number of tests to which it was exposed. Type B films were considerably less durable and the best of them failed 6 tests while the poorer ones failed as many as 15 tests. The best Type C films failed 7 and the poorest

failed 15 tests. Undoubtedly this type of film would have failed additional tests had the procedure not been stopped because the films were so white by reflected light that the coating was barely discernible and damage caused by the tests could not be accurately detected. No film was said to fail unless damage was definitely seen to exist.

The film tests are broadly classified to determine:—

- 1. Optical Characteristics
- 2. Mechanical Characteristics

Tests for optical characteristics are basically those for transmission and reflection. Spectrophotometric transmission is measured in our laboratory by the General Electric Recording Spectrophotometer equipped with the old type integrating sphere. With this device only measured transmission curves are obtainable. By assuming what is thought to be nearly correct absorption values, reflectances at various wave lengths can be computed. However, the errors introduced by assuming absorption values are so large as to make the reflectances values, so obtained, of little help in judging film quality, therefore reflectances should be measured by Spectrophotometric methods. The Hardy instrument equipped with the newer type integrating sphere and the Cenco-Sheard Monochrometer in use at the Washington Navy Yard are two such devices for performing these measurements. Reflectances obtained by the use of these instruments can be plotted on a reflectance versus wavelength curve and the results considered reasonably accurate. The process is long and tedious but the accuracy of the results yielded seem to justify the method. Results of comparable accuracy but much less tedious in procedure may be made by the use of a recording Spectrophotometer. The Hardy instrument with the newer type integrating sphere is ideally suited for this problem. Unfortunately neither Frankford Arsenal nor the Washington Navy Yard have such a device available at the present time. It should be noted that one hears much debate concerning the accuracy of the Hardy recording Spectrophotometer and the limits of its reliability. Some believe that whether measuring transmission or reflection, its curves are reliable within 1 or ½ percent while others accept its accuracy at any wavelength as being 0.05%. My own experience leads me to believe that the Hardy instrument is not reliable beyond 0.5%.

We at Frankford Arsenal are using white light in measuring luminous transmission and reflectances. Our instruments are both simple and direct. The automobile head light, 50 CP lamp operated at 6.3 volts by storage batteries is used for a source of constant light of the correct color temperature. Its light is collimated to fall directly upon the Weston Photronic cell equipped with a Viscor filter both before and after reflection or transmission from the optics under test. White light transmission is computed from measured values by means of the formula  $T = \frac{F}{F_o}$  where  $F_o$  is the incident light flux and F is the transmitted light flux. We use thin samples of coated and uncoated optical glass for which the absorption is small. Any attempt to correct for absorption by subtracting its effect is nearly as bad as neglecting it altogether, since absorption seems to vary greatly and is dependent upon impurities in the melt.

White light reflections are determined by the same equipment and with the same care. Reflectance is given the formula  $R = \frac{F}{F_o}$  where  $F_o$  is the incident light flux and F is the reflected light flux. Research Report 44-3 is used as an alternate and check. When it is desired to measure the transmission of reflection for three color bands (red, green, blue violet) we interpose Wratten A, B and C5 filters. Every possible caution is exercised in the making of these measurements in order to eliminate errors which may arise from photocell fatigue or an inconstant light source.

Often our test include transmission measurement on completed instruments. Two essential methods are available:—

A. Brightness method

 $Transmission = \frac{Brightness\ with\ instrument}{Brightness\ without\ instrument}$ 

B. Light Flux Method

 $Transmission = \frac{Transmitted \ light \ flux}{Incident \ light \ flux}$ 

It is assumed that these fundamental methods of measurement are familiar to this group without elaboration.

Film Thickness and Related Color: There is a "one to one" correspondence between color and film thickness. Thickness determines color and color is a most reliable and sensitive indicator of film thickness. The worst trouble about trying to judge film thickness by color as seen by the human observer is that sometimes hue is masked, and more or less obscured due to vastly different saturations. Even experienced observers often find it difficult to state within a narrow wavelength band at which wavelength the minimum reflectance occurs. Fortunately, it is really not necessary to judge color more

definitely than the human observer sees it.

Accurate film thickness determination involves running an experimental spectrophotometric curve on each sample under strict test. Such a curve shows a minimum at some spectral color or wavelengths. The color by reflected light is the complement of this.

Scattering Light by Coatings: Attempts have been made by the laboratory to design apparatus to determine scattering in coatings. These attempts so far have been unsuccessful. They have indicated, however, that scattering is very small and when transmission, reflectance, and absorption values are given in our reports scattering has been neglected. Except for some silicate type films, scattering due to coatings seem to be no more than that due to carefully cleaned bare glass.

Uniformity of Film: Evaporating from essentially a point source on to a flat optics holder containing production runs of optics to be coated must result in non-uniformity of film thickness with its related effects upon color and reflectances. Compensation within the vacuum of some type must be used to avoid this. When submitted optics come to us, we examine for these effects, furthermore we look for pinholes and scratches in coating. In addition to this, "sputter" or "spatter" marks are religiously sought. These last-named marks are serious since they not only damage the coating but the underlying glass as well. These nonuniformity of film effects indicate faulty coating technique and when found, indicate need for adjustment of such techniques. In looking for all such defects examination should be made with the "unaided eye." We should furthermore be certain that recorded faults are actual film faults and not faults of the glass blank emphasized by coating.

#### **Tests for Mechanical Characteristics**

Cleaning of Coatings: Four cleaning methods are used by our testing laboratory. All optics are checked to see if these cleaning methods damage the coating. The details of these cleaning methods, why they were used, and what results were obtained are to be found in TRR #44-5.

Briefly, cleaning method #1 is very mild on the coating but effectively cleans. Cleaning method #2 is the cleaning procedure used in optical assembly at Frankford Arsenal. Cleaning method #3 was designed to see if Black Navy Sealing Compound could be removed without damage to coatings. Cleaning method #4 was designed to see if India ink used around the edges could be cleaned off without doing damage.

Abrasive Tests: The equipment we use on abrasion tests has been made so as to cause a pad of clean dry absorbent cotton

exerting a force of one pound on the coating to rub in one direction across it 50 times. The test is not severe and most coatings stand it without damage. Before examining for effect the coatings are cleaned, using cleaning method #1 referred to above. A variation of this test consists in dipping the cotton pad in calcium carbonate and sprinkling this compound freely over the surface to be rubbed prior to rubbing. We start the equipment and rub as many rubs as are necessary to produce a given color change, and later to remove completely the coating from the glass at some point of the rubbing cycle. The number of rubs to produce these effects are recorded as a measure of abrasion resistance of coatings. These tests are indicative of and not to be taken as accurate measures of abrasive resistance.

Water Resistance of Film: It is imperative that coatings for military use be insoluble. The tests we now run are:

- a. Distilled Water Drop Test: A drop of distilled water about in  $^1/8''$  in diameter is placed on the coated surface. This drop is allowed to dry normally, leaving a spot. Removal of the spot thus formed by cleaning technique #1 is indicative of insolubility. This turns out to be a severe test and many coatings will be removed at the spot by such treatment.
- b. Relative Humidity Test: Coated optics are exposed to an atmosphere at  $120^{\circ}$  F. and 95% relative humidity for 24 hours. They are then removed, washed and examined for effect.
- c. Intermittent Condensation Tests: Two intermittent condensation tests are carried on in our testing laboratory. We shall call one the high temperature intermittent condensation test and the other the low temperature intermittent condensation test. In the high temperature test a circular horizontal turntable carrying samples under test revolves on its axis 15 rph. A fixed partition divides the two halves of the turntable. On one side, live steam emerges from three jets and condenses on the samples passing underneath. During onehalf cycle each sample is wet and hot and during the other half cycle each sample is dry and cold (relatively). All samples of coated optics are submitted to 10 and 40 such intermittent condensation cycles. In the low temperature intermittent condensation test samples are placed in a thermostatically controlled cold box. The temperature is slowly lowered. At several temperatures on the way down to -60° F., the ultimate low temperature goal, the films are removed from the cold box to allow condensation and freezing on their surfaces. This is done ten times within the room temperature to -60° F. temperature range.

Salt Spray Tests: Three salt atmosphere tests are applied to coated optics submitted to us. All tests use the same large cylindrical glass chamber containing  $1\frac{1}{2}$  pounds of salt per cubic foot of water salt solution. The cylinder contains sufficient of this solution to give a depth of about three inches. An aquarium motor bubbles air through this solution. The solution and enclosed space above the solution is heated electrically and its temperature is controlled thermostatically at predetermined values. In each test coated optics samples remain in this atmosphere for 24 hours.

- a. Salt atmosphere test #1 is run at room temperature (24° C.)
- b. Salt atmosphere test #2 is run at 35° C.
- c. Salt atmosphere test #3 is run at  $50^{\circ}$  C.

These tests are progressively more and more severe due to temperature change. Nearly all coated optics regardless of type pass test a without damage to the coating except to produce numerous pinholes or the enlargement of invisible pinholes to visible sizes. The samples remain sensibly dry throughout the test. Only high temperature baked coated optics pass test b. The coating is severely damaged and removed in all other types of coated optics. Pinholes are not produced or emphasized. The samples are wet with droplets throughout this test. In general no coated optics pass test c. The coating is removed or severely damaged even on high temperature baked samples by this test. It is only occasionally that samples pass this severe test.

*Temperature Stability of Coatings:* Attempts are made in our tests in the Fire Control Laboratory to determine the temperature stability of coatings. A brief outline of the tests follow:

- 1. High Temperature (Room temperature up to  $200^{\circ}$  F.)
  - a. Effects due to thermal expansions.
  - b. Effects due to change of index of refraction and/or a change of film thickness—color changes.
- 2. Low Temperature (Room temperature down to –60° F.)
  - a. Same as a. above
  - b. Same as b. above

Results of these tests may be found in TRR #44-5.

Fading or Weathering Unit Tests: A National Accelerated Fading or Weathering Unit, Type XV was used in these tests. The water trough coil rheostat was turned so as to give maximum heat to the water inside the unit which was at 165° F. at all times during these runs. This kept the relative humidity inside at approximately 75%.

The coated optics samples were suspended from the revolving samples holder so as to be at the normal distance from the carbon arc filtered by the standard Corex glass filter. All samples were exposed for 40 hours of continuous operation. For results see TRR #44-5.

Anti-Fog Tests: The effect of using U. S. Navy Anti-fogging compound on coated optics was tested. These tests were run simultaneously with the Low Temperature Intermittent Condensation Tests already discussed. In fact, they were run on the same samples by using the anti-fog solution on one side and leaving the other side without it. In general the results may be briefly stated as follows:

- a. Both sides fogged badly at temperatures -52° F., -40° C., -35° C., -10° C., -6° C.
- b. It was not until the specimen were up to -4° C. that the anti-fog compound began to show an advantage over the untreated side.
- c. From  $-4^{\circ}$  C. on up to room temperature the treated side progressively proved more and more advantageous. It was not until the temperature was raised up to  $+9^{\circ}$  C., however, that the treated side showed no fogging at all.

Oil Absorption Tests: Since a large portion of Fire Control Optical Instruments are lubricated with grease or oil, the possibility of volatilized films of these substances under service conditions affecting the efficiency of coating was considered. A coating which loses its efficiency when exposed to oils and greases actually used in the assembled instrument is not suitable. In an oven at 200° F. we placed a beaker containing Keystone 84H Grease. Coated optics were placed about three inches above this grease. As time progressed the samples were removed, tested for white light transmission, which was compared to their transmission before exposure. None of the samples seemed to be grease absorptive under these conditions. One reason for this phenomenon may be that the high temperatures probably drove off any grease film which might have formed as fast as it went on the samples. The test is thus not free from serious criticism and it is felt that the results are inconclusive. Details may be found in TRR #44-5.

The test procedure in testing coated optics for effect of Bendix Pioneer Ball Bearing Oil consisted in rubbing each sample with this oil and allowing it to remain on the coating for five minutes, swabbing, rubbing and "polishing" the samples dry and apparently clean with soft cloth; after which its white light transmission is measured and compared with its value before treatment.

The efficiencies of certain so-called silicate coatings were radically lowered by this treatment. All coatings lost some transmission thereby. In general coatings regained their light transmission after degreasing in isoproponal vapors. Detailed results may be found in TRR #44-5.

In conclusion from the tests outlined it may look as though we expect the impossible of coatings used for military purposes. Nothing could be farther from the truth. As an encouraging last remark it should be said that a concern making coated optics need not fear inspection if they use good techniques, pure fluoride, and a high temperature bake.

#### DISCUSSION

**Dr. Lyon:** The preceding discussion about standard tests sounds very complicated, but after you cut through the haze surrounding all of this talk, you still come back to the fact that all you have to do is use good technique. Watch the color, use good fluoride, keep the equipment in excellent working order, clean the surfaces thoroughly and you won't require any of the equipment described, because your films will automatically pass any specification which will be written. I can assure you that the test for durability which is now incorporated in the Navy specification will be made much more severe. We hope to eliminate those films which will soften on exposure to humid atmospheres, but if you use a good technique you need have no fear of any severe test we may write into the specifications.

**Mr. Raines:** There is a complete report available on all tests conducted at Frankford Arsenal. Any of you may have a copy of that report by addressing a letter to the Design Division at Frankford Arsenal.

**Col. Welch:** The other day I asked Mr. Raines and Mr. Denton whether it was possible to make an objective test instead of a color test. I don't like the color test very well. We never subject our inspectors to a color blind test.

**Dr. Lyon:** I don't believe I can say much about the subject of color blind coaters. When we started large scale production, I interviewed the help sent to me from the optical shop. I handed them some optics and asked if they could recognize the various colors. If they could, I concluded they would be suitable as coating operators.

Mr. Quin: For the past year in taking applicants into that particu-

lar department, we have had a medical department test their eyes for color sensitivity. We have found some peculiar cases, even to the point where a person will recognize the primary colors, but the pastel shades will not register.

**Mr. Berlant:** We have made it a practice to submit every worker in the coating department to a color test, using flat plates. After a slight amount of practice the girls could identify colors to a remarkably close degree. In view of the range of color which is permissible, I don't foresee any difficulty whatsoever of operators or inspectors selecting colors that are well within specifications.

With reference to the use of proposed instruments for color determination, you should consider the fact that we sometimes find large and weak images and sometimes an image that the operator can only find by moving the light or by changing the position of his head. We have never yet had any trouble determining the color within the specified range.

Dr. Gardner: May I explain another way of testing a film for thickness? By means of a lens, focus the image of an incandescent filament upon the surface to be coated, thus producing a small bright spot, or preferably, a bright narrow line. Observe this through a direct vision prism (not complete direct vision spectroscope), or through a grating replica. Before the production of the coating one should see a bright continuous spectrum. As the coating forms the regions of the spectrum for which the reflection is decreased will darken. When the dark region occurs within the desired range of wavelengths the deposition is complete. This is a suggested method that has not been tried. The image of the filament produced on the surface should be as small and bright as possible. In general, for a lens, there will be two images, one from the upper and one from lower surfaces, but they can be readily distinguished. If the image is in the form of a bright line, the grating replica should be held with its lines parallel to the longer dimension of the image. In normal times replicas can be secured at moderate price from the Central Scientific Co. or the Gaertner Scientific Corp., both of Chicago.

**Dr. Wright:** The interference colors by which the correct thickness of an anti-reflection film is judged, is not greatly different from the "sensitive tint" of the Newton color scale or of the "sensitive tint" of a colorless birefracting crystal, such as quartz, of the correct thickness and orientation and observed between crossed polarizing prisms or films. The sensitive tint is a bluish or purple red (mauve, magenta) of low intensity and changes rapidly with slight change in

thickness of the of the crystal plate or film. The color represents white light minus the central of the spectrum which has been eliminated through interference. If the thickness of the crystal plate or of the film is decreased somewhat, the blue and green spectrum colors are eliminated and the interference color shifts toward the red; if the thickness is increased, the red portion of the spectrum is eliminated and the interference color shifts to blue. In anti-reflection films on glass the saturation of the color depends on the ratio of the refractive index of the film to that of the glass. If the ratio is correct, the amplitude of the ray reflected at the outer surface of the film is equal to that reflected at its inner surface and the interference is complete. In other cases, the interference is less complete and the resulting color less saturated. It is for this reason that that the interference colors of anti-reflection films on light crown glasses of low refractive index are less saturated than those on dense flints of high refractive index.

**Dr. Lyon:** I have one suggestion which hasn't been brought out. When coating crown glasses which have low indices of refraction, colors will not be very pronounced. The colors are much brighter on high index glasses. Therefore, when you are coating crowns, always use a piece of high index glass in your bell jar as the pilot sample or monitor. You will be able to judge your colors much better if you will observe this high index glass as a test sample when adjusting for proper film thickness.

**Dr. Williams:** I have just one question concerning inspection on production. Now, is it desired that the optimum color of the film on that element be observed at the center on the optical axis of the lens, or does the Navy and Army want us to split the difference? Because if you get it right on the optical center, of course, it's going to be awfully thin on the edges of many lenses.

**Dr. Lyon:** Our instructions have always been to make films too thin rather than too thick. This permits maximum benefit for night use which is desired. Since there is universal tendency to over-coat and that over-coating will continue despite our best efforts, I would say that you should aim for the correct color at the center of the lens and let the color on the remainder of the lens come out what it will.

**Mr. Raines:** In some optics at least, the entire lens is not completely used and it would be better to keep the right color in the center.

### Addenda



**I.** The following is a typical coating cycle used for the deposition of a high temperature baked magnesium fluoride film.

This cycle, described by Mr. Wallace of American Cystoscope Makers, Inc., has been in operation by that company for several months.

"The optics are mounted in a jig, the jig placed in the jar, the sealed and the mechanical pump started and the heating elements turned on. In ten to twelve minutes, the pressure has been reduced to a point where the diffusion pump can be turned on. This is operated on 'high' for a period of three minutes and then switched to 'low.' Approximately thirty minutes elapse before a vacuum is obtained suitable for coating. As soon as the proper vacuum is reached, heating elements are turned off, and filming started. Filming time takes from two to three minutes, after which the heaters are once more turned on and the film is baked under vacuum for ten to twenty minutes, depending upon the size of the optics. Thereafter the glass is given a few minutes in which to cool before being removed from the bell jar. The overall cycle consumes approximately seventy minutes."

**II.** Minneapolis-Honeywell Regulator Co. advised that they are using a 12,000 volt glow discharge unit which is turned on shortly after closing the circuit of the diffusion pump and left on for approximately five minutes. They are of the firm opinion that this aids in liberating the gases within the bell jar that are occluded in the pores of the metal, glass and fluoride, thereby shortening the period of temporary rising and falling pressure after their heaters are turned on.

**III. Process A.** Frankford Arsenal, by reason of the great variety of optics that they are called upon to film both for production and experimental purposes and so that they may best be able to instruct new facilities in the basic technique of filming, has evolved a list of essential equipment and materials and a sequence of operations for the application of high temperature baked magnesium fluoride films, which it believes represents the optimum in simplicity and successful films. This is known as Process A and is hereafter described in detail.

### Process A

#### Equipment:

Any standard type evaporator may be used. National Research or Distillation Products units are quite satisfactory, or units similar to these may be made by the individual manufacturer. The components of satisfactory vacuum units include:

- 1. *Pyrex Glass Bell Jar* (18" in diameter by 27" high, <sup>3</sup>/<sub>8</sub>" thick).
- 2. Rubber Gasket (between bell jar and base plate).
- 3. *Base Plate* (made of metal fine ground on the top side by mechanical milling).
- 4. Mechanical Pump (a Hypervac 20 or Kinney #VSD 556).
- 5. *Diffusion Pump* with Baffle (D.P.I., #LC-500 or #LC-275 are commonly employed).
- 6. Vacuum Gages: One for rough vacuum and one for high vacuum. Either a thermocouple or a Pirani gage is usually used for rough vacuum and an ion gage is standard for high vacuum.
- 7. Filament Transformer: This transformer is to reduce line voltage to a top voltage which will be low enough to insure the proper filament current. The top filament voltage on the Distillation Products machine is 24 volts and On the National Research machine it is 8 volts.
- 8. Spatter Shield: A 4" disk of metal (preferably steel) fastened to an arm holding it one or two inches above the filament. The supporting arm is suspended on a pivot bearing. The opposite end of the arm is close to the inside surface of the bell jar and the magnet or piece of iron is attached to this end. By manipulation of a magnet outside the bell jar the shield may be moved either over the filament or away from it.
- 9. Glass Holding Rack: The glass is either on a flat or approximately spherical rack 18" from the filament. The diameter of the sphere about which the rack is shaped should be slightly less than the distance from the filament to the glass, that is, 16" or 17". If a flat rack is used, a compensator of the R.C.A. type is used to assure an even coat.
- 10. Heater: A conventional type heater employing nichrome wire attached to and insulated from a metal reflector is used. The reflector is spherically shaped with a radius somewhat longer than that of the glass rack. The size and amount of nichrome wire used can be varied, but several "musts" should be obeyed to obtain the proper results.

- A. The resistance of the heater should be low enough to draw at least 750 watts. This may be accomplished by a parallel circuit if necessary.
- B. At least 10 feet of wire or 10 feet of coil (if toaster size wire is used) should be employed to give proper distribution. The circuit should be rigged up so that the wire draws 750 watts at dull red heat. About 20 feet of #19 nichrome wire on 110 volt AC works very well.
- 11. *Tesla Coil:* A high voltage, high frequency discharge coil is used to clean the glass. It must be certain that a high enough voltage and frequency is obtained. Central Scientific Company #80721 leak detector is satisfactory.
- 12. Evaporation Filament: A flat coil wound from tungsten .040" in diameter. For N.R.C. machines a piece of wire 4-6" long is used and the distance between the filament holders is about 2½". For the D.P.I. machine a piece of wire 6-8" long is used and the distance between the filament holders is about 3½".
- 13. *Porcelain Crucible:* A Coors porcelain crucible #000 size is used. For convenience the crucible support is placed on a vertical thread shaft so that the crucible may be raised or lowered.
- 14. Filament Distance Gage: A piece of glass or clean metal between  $\frac{1}{32}$  to  $\frac{1}{16}$  of an inch.
- 15. *Magnesium Fluoride*: Made at the Arsenal almost exactly in the manner recommended by Dr. Lyon. The American Cystoscope Makers Incorporated makes and sells fluoride which is quite satisfactory.
- 16. *Celanese Napkin:* Acetate rayon about 16" square, hemmed on all four sides.

## **Operations:**

A. Cleaning: The lenses are first washed in a 2 to 3% solution of warm water with clean, soft cheese cloth. This is followed by a rinse in tap water and the surfaces which are to be coated are then rubbed with a Wet-Me-Wet pad and allowed to become almost dry. The Wet-Me-Wet is then wiped off with cheese cloth, two wipings with different cloths are usually employed. The elements are finally wiped over with a lintless Celanese napkin to remove any remaining chalk or dust. After the optics have been racked and the jar has been placed on the coating machine, but before the bell jar is lowered, all elements are given a last going over with a brush discharge from the Tesla coil.

B. Operation of Vacuum Equipment: The mechanical pump is first turned on and after a pressure of about 100 microns is shown on the thermocouple or Pirani gage and diffusion pump is started. The glass heater is turned on about five minutes after the mechanical pump has been started and raised to top heat over a period of five to ten minutes. It is not practical to coat at vacuum of less than 1 x 10<sup>-4</sup>. When this pressure has been reached, the filament is heated at about one-fifth of top voltage for about three minutes, with the spatter shield over the filament. The filament is then gradually raised to a top voltage over a period of two minutes and left there for thirty seconds. If there is no evidence or appearance of spattering, the spatter shield is then moved to the side with the magnet and coating is started. Regardless of what is being coated a flint lens rejected for scratches or chips, etc., is cleaned exactly the same as the rest of the work and placed in a position for viewing by reflected light and used to monitor the thickness of the coat. In viewing by reflected light the angle of incidence and reflection should be held to 30° or less. It is usually attempted to stop the coating on the light side, that is, at a reddish-purple color. The evaporation usually takes from three to eight minutes. It is shorter on the D.P.I. machines because of the larger filament transformer which permits about 900 watts to be used in the filament. On the N.R.C. machines the top obtainable voltage is 8 volts, which will permit only about 300 watts to be used with a shorter filament. Coating is never started until the elements to be coated have been heated a long enough time to make certain that they have reached a temperature of over 400° F. Experience based on many temperature and hardness measurements from various heating cycles has made it unnecessary to place a thermocouple in the jar for each run. The lenses are usually heated for a period of 40 minutes before coating. Most Porro prisms get from 10 to 20 minutes more. Larger work, such as very large prisms, may get as much as  $1\frac{1}{2}$  hours of heating.

The magnesium fluoride is ground with a porcelain mortar and pestle to pass through 80 mesh screen and is placed in a Coors #000 porcelain crucible level to the top. More fluoride is added every run or every other run to replace that which has been evaporated. About every dozen runs the whole crucible is recharged. The bottom half of the crucible is usually charged with reground, used fluoride, as this part is merely insulation. The filament is wound in a flat coil

which is clamped horizontally between the filament posts and the distance between the filament and fluoride is 1/16 to <sup>1</sup>/<sub>32</sub> of an inch. An easy way to control this distance is to place a gage consisting of a piece of glass or metal of the proper thickness on the crucible and then raise it until the gage is withdrawn. During the preheating of the fluoride, the ion gage should be watched to determine if there is any great drop in vacuum. If the vacuum drops greatly and does not return during the pre-heating, the spatter shield should not be removed, as evaporation under these conditions is likely to cause soft coats and coating will be extremely slow in any case. If the loss of vacuum persists, the filament should be turned down. If the proper degree of vacuum is regained it is evident that the fluoride is outgassing. Continued heating of the fluoride should finally result in attainment of the proper vacuum with the filament hot.

When the optics have been coated to the proper color, the filament and diffusion pump are turned off and the glass cooled for five minutes or seven minutes. At this point the mechanical pump is shut off and it usually takes from two to three minutes to bring the pressure in the jar up to atmosphere. In the case of large work the cooling period may be extended to as much as 20 minutes.

## **Maintenance of Apparatus:**

The inside of the bell jar and the top of the base plate are cleaned every day. Care should be taken that no powdered fluoride gets between the gasket and the bell jar or the gasket and the base plate. The bell jar gasket is changed almost every week and is frequently rubbed with a rag moistened with castor oil so that a thin film of castor oil remains on the gasket.

The rubber gasket and rubber tubing connections on the machine are replaced at least every three months and sometimes as often as every six weeks. The seals on the electrical leads through the base plate are less frequently serviced. The oil in the mechanical pump is also changed regularly. The Kinney pump oil is changed once a month and the Hypervac oil once a week.

# Conference Attendees



Representative and Company Aeller, G. L. National Research Corp. Alderson, S. Anchor Optical Corp. Alexander, C. H. Distillation Products, Inc. Allison, G. W. Mergenthaler Linotype Co. Bailey, W. B. Zenith Optical Company Bateson, Sydney Research Enterprise, Limited Bechtold, Edwin W. Frankford Arsenal Becker, William Mergenthaler Linotype Co. Been, Robert B. New York Navy Yard Behrndt, Carl Distillation Products. Inc. Berlant, E. H. U. S. Instrument Co. Blauvelt, W. W. Mergenthaler Linotype Co. Blue, Stephen R. Mergenthaler Linotype Co. Boydston, R. W. Frankford Arsenal Brady, L. J. Dioptric Instrument Corp. Brezeale, Lt. Francis Bureau of Ordnance, USN Buhrman, G. L. International Industries, Inc. Capitano, Nelson J. New York Ordnance District Carr, Capt. D. J. Optical Instrument Committee Colbert, William H. Liberty Mirror Works

Cooley, J. P.

New York University

Representative and Company Cox, Dean Distillation Products, Inc. Dawson, Capt. L. N. Frankford Arsenal Delamater, P. G. Merganthaler Linotype Co. Del Yoster, Henry Semon Bache & Co. Denton, Richard A. Frankford Arsenal Derau, John V. Nash-Kelvinator Corp. Domzal, Erwin A. Detroit Ordnance District Eikenberry, W. M. Robertson-Houchin Optical Co. Emens, Capt. F. M. Rochester Ordnance District Fairweather, W. W. Boncshur & Holmes Optical Co. Faulhaber, Thomas Cleveland Ordnance District Ferguson, W. F. C. Mergenthaler Linotype Co. Fitter, E, A. U. S. Optical Supply Corp. Foisy, G. A. Mergenthaler Linotype Co. Fox, John M. National Research Corp. Fricke, E. A. Frankford Arsenal Friedman, Harry S. Kollmorgen Optical Corp. Gage, H. L. Mergenthaler Linotype Co. Gardner, Irvine C. National Bureau of Standards Geier, George Keuffel & Esser Co. Gilmour, G. A. Bell & Howell Co.

Representative and Company Goetzenberger, Ralph L. Optical Instruments Committee Grafton, Capt. S. M. New York Ordnance District Hacquet, Roland V. Universal Camera Corp. Hammer, Walter Bonschur & Holmes Optical Co. Hanson, C. A. Mergenthaler Linotype Co. Harmon, C. M. Optical Research, Inc. Hartig, Lillian G. American Cystoscope Makers, Inc. Heacock, R. H. RCA Victor Division Hearn, J. W. Mergenthaler Linotype Co. Heroman, Capt. L. C. Frankford Arsenal Herrman, Carl W. Distillation Products, Inc. Hesse, L. Frank Robinson-Houchin Optical Co. Hett, John H. U. S. Optical Supply Co. Hull, G. F. Universal Camera Corp. Hunt, R.E. Nash-Kelvinator Corp. Jacobs, Donald A. National Bureau of Standards Jewett, Frank B. Jr. National Research Corp. Jones, D. C. Research Enterprise Limited Juengst, Winston C. American Cystoscope Makers, Inc. Kahn, Jacob Ultima Optical Instrument Corp. Kavinsky, Gregory U.S. Instrument Corp. Keenan, William J. Eastman Kodak Co. Krusberger, Joseph F. Mergenthaler Linotype Co. Lanza, Silvio A. Mergenthaler Linotype Co.

Representative and Company Leader, William U. S. Instrument Co. Levitt, J. W. Franklin Institute Liddel, Lt. Urner Bureau of Ships, USN Liebman, Roy Kollmorgen Optical Corp. London, Lt. Harry Bureau of Aeronautics, USN Lyon, D. A. U. S. Naval Gun Factory Mandell, Bernard U. S. Instrument Co. Mariani, M. Semon Bache & Co. Mattern, R. P. Minneapolis-Honeywell Regulator Co. Moore, E. L. U. S. Optical Supply Corp. Morse, C. Y. U. S. Optical Supply Corp. Morse, R. S. National Research Corp. Mueller, A. C. Bell & Howell Co. McCallum, F. C. Spencer Lens Co. McCarthy, E. L. Perkin-Elmer Corp. McCormick, W. F. Mergenthaler Linotype Co. McEvoy, Capt. L. C. Jr. Chicago Ordnance District McRae, D. B. Eastman Kodak Co. Newcomer, H. Sidney Dioptric Instrument Corp. Newton, B. Semon Bache & Co. Peterson, Stanley Bausch & Lomb Optical Co. Pettus, J. H. RCA Victor Division Phillips, P. H. Cincinnati Ordnance District Pittman, John J.

Eastman Kodak Co.

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Pride, Gilbert E.
Spencer Lens Co.
Prager, J.
Optical Instrument Corp.
Purcell, J. D.
U. S. Naval Observatory
Quadt, J.

*Mergenthaler Linotype Co.* Quin, Elmer G.

Eastman Kodak Co.

Raines, Arnold Frankford Arsenal

Pappaport, S. *Ultima Optical Co.* 

Roach, W. T.

Eastman Kodak Co.

Rosenberry, Harold Anchor Optical Corp.

Schilling, E. A. *Anchor Optical Corp.* 

Schroyer, Carl B.

*Farrand Optical Co.* Simmons, A. B.

Eastman Kodak Co. Shusset, Lewis I.

Pittsburgh Ordnance District

Skipper, H. E. U. S. Naval Observatory

Smith, Harry A. Herschede Hall Clock Co.

Snyder, R. B. Bell & Howell Co.

Sobel, Samuel Semon Bache & Co.

Staring, Lt. M. H. U. S. Naval Observatory

Steinbach, Charles Frankford Arsenal

Stewart, Capt. G. R. Frankford Arsenal

Storms, Ens. L. B.

U. S. Naval Gun Factory

Representative and Company Street, C. C.

Perkin-Elmer Corp.

Street, Virginia T.

Perkin-Elmer Corp.

Sturr, Major A. W.

Tank Automotive Center

Tomkinson, D. D.

Perkin-Elmer Corp.

Tyler, John E.

National Research Corp.

Vance, Lt. C. F.

New York Navy Yard

Wain, Louis L.

Boston Ordnance District

Wallace, F. C. Zenith Optical Co.

Wallace, F, J.

American Cystoscope Makers, Inc.

Wappler, F. C.

American Cystoscope Makers, Inc.

Welch, Co1. G. B. Frankford Arsenal Williams, R. C.

University of Michigan

Wilson, J. T.

Perfex Corp.

Witherspoon, R. O.

Cleveland Ordnance District

Wormser, Eric M.

Universal Camera Corp.

Wooters, Glenn C.

Gundlach Manufacturing Corp.

Wright, F. E.

Joint Optics Committee ANMB

Yahr, M. J.

RCA Victor Division

Zak, T. J.

Bausch & Lomb Optical Co.

Zelnis, Major E. P.

Office of the Chief of Ordnance

Zook, M. Alva Jr.

U. S. Naval Gun Factory