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A Study of Filament Coatings for Positive Ion Emission

Philip Arnold Rognlie

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FILAMENT COATINGS FOR POSITIVE ION EMISSION A STUDY OF

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A THESIS

PRESENTED IN CANDIDACY FOR THE DEGREE OF MASTER OF SCIENCE

BY

PHILIP A. ROGNLIE B. S. in Ed. , U. N. D. 1931

UNIVERSITY OF NORTH DAKOTA

June 1934

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This Thesis, presented by Philip A. Rognlie as a partial fulfillment of the requirements for the degree of Master of Science in the University of North Dakota, is hereby approved by the Committee under which he has carried on his work.

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A STUDY OF

FILAMENT COATINGS FOR POSITIVE ION EMISSION

TABLE OF CONTENTS

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INTRODUCTION

In an ordinary electrical discharge the most evident effect is a stream of particles passing from cathode to anode. There is, however, something else present. There are some particles found to be travelling the opposite direction, i.e. from anode to cathode. If the aforementioned cathode rays are possessed of a negative charge it seems reasonable to assign a positive charge to these anode-to-cathode particles.

These particles were discovered by Goldstein¹ in 1886. Since **he used a perforated cathode the better to study these rays, and since these perforations might be termed canals, he gave these new rays the name, translated, "canal rays."**

Since that time these positively charged particles have been the object of considerable attention, and rightly so. They have played an important role in many instances, some of which will be enumerated.

The principle underlying the experimental determination of atomic weights is that of weighing ouantities of matter that combine in known proportions with Oxygen which has been assigned the arbitrary atomic weight of 16. Furthermore, it is also known that the chemical properties of a substance are not especially functions of the nuclear mass, but rather are dependent on the number of external electrons the atom possesses, that is, a function of the

1Goldstein, Berlin. Ber., 39, 691 (1886).

atomic number. The mass of these electrons is negligible when compared with the mass of the nucleus. The ratio of the mass of one proton to the mass of one electron has been found to be (1839 ± 1) to 1.

Theoretically then it is possible for there to exist several separate and distinct substances from the standpoint of mass that possess identical chemical properties. The only condition necessary for this is that these several substances all possess the same number of electrons external to the nucleus of the atom. If several varieties of this type of matter did exist, dividing the mass of one gram-molecule of the substance by N, Avogadro's number,would merely yield a value for the average mass of the molecule..

Such substances have been given the term "isotopes", and their existence has been demonstrated by experiments on positive rays. We can say that these positive rays are made up of streams_{of} **positive ions. The atomic weights of substances composed of isotopes then are found to be averages of the atomic weights of two or more isotopes. For instance, a combination of 77 atoms of Cl of atomic weight 35 with 23 atoms of Cl of atomic weight 37 will yield a substance having an average atomic weight of 35.46.**

One of the more important methods of determining masses of atoms that do not depend on a knowledge of atomic weight is that of measuring the ratio of the charge on an ion to its mass. Such an ion may be obtained in any one of several ways. One convenient method is to remove an electron from the atom in some fashion long enough for the ratio e/M to be measured. This electron removal can be accomplished in different ways. Electron bombardment will

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produce that effect. Emission can also be obtained from a hot filament.

Much of the original work on positive rays was done by J. J. Thomson¹. One of the classical experiments is his parabola method of measuring e/M of positive ions.

O Other workers in this same field are notably Aston and 3 **Dempster. Aston's big contribution was his mass spectrograph. Measurements of ionic masses with his apparatus permit of accuracy of 1 part in 10,000.**

Dempster's method also possesses the degree of accuracy obtained in Aston's measurements and is also a mass spectrograph.

The substances used are mostly salts of the alkali metals, When the various substances used emit positive ions they are $+$ $+$ $+$ $+$ $+$ **Li , Na , and K , depending on the composition of the substance. Of course when Potassium Phosphate and Potassium Pyrophosphate were used K** *** **was the ion.**

Thomson, J. J. Phil. Mag., 13, 561 (1907); 20, 752 (1910); **31, 335 (1911): 24, 309 (1912). 1912 2Aston, Phil. Mag. J58, 709 (1919); "Isotopes", 2d ed. , Longmans (1924); Proc. Roy. Soc. 115, 487 (1927); 126, 511 (1930). ^Dempster, Phys. Rev., 11, *516 (1918); Proc. Nat. Acad. Sci. , 7_, 45 (1921); Phys. Rev., 18, 415 (l921); 81, 209 (1933).**

THE PROBLEM

The object here is then to make a comparative study of the emission of positive ions from coated filaments. The substances used in coating the filament are:

THE APPARATUS

-5-

3ome time ago Dr. Hundley had constructed apparatus for the then express purpose of measuring the ratio of charge to mass for electrons, it being an improvement on Dr. Hoag's method. As the apparatus stood it lent itself admirably to the present problem and so was employed with no alterations. Naturally the coils which were used in obtaining a magnetic field in the e/m studies were not used in this instance.

For this problem then the apparatus is essentially a two electrode vacuum tube, the elements being filament and plate. The tube proper is a piece of pyrex tubing 36 cm. long and 5 cm. in diameter. One end of this tube can be opened to permit entrance or renpval of a piece of hollow brass tubing whose outside diameter is comparable to the inside diameter of the glass tube. This brass tube is 13 cm. long and constitutes the receiving plate in the present set-up. This open end of the tube must be closed and be made air-tight for operating conditions. To accomplish this the edge of the tubing is ground flat and a piece of plate glass is placed over the opening and sealed by means of sealing wax. This forms a fairly satisfactory seal since the vapor pressure of the sealing wax at room temperatures is not disturbingly high. The other end of this glass tube is connected to the evacuating system.

This same tube is tapped on one side and there fitted with another glass tube with its remote end ground appropriately to receive an insert, ground to fit. This insert comprises the

filament assembly. The filament itself is a piece of platinum foil 19 mm. long, 1.036 mm. wide and 0.035 ram. thick. The filament leads in the assembly are two metal rods between the inner tips of which the filament is clamped. These rods are brought out of the glass by the use of glass-to-metal seals. To the tips of these extruding rods then is connected the source of filament potential. The filament itself is placed so that its sharp edge is direoted toward the receiving plate. Since this filament assembly is removable it is made air-tight by the application of sealing wax at the ground-glass joint.

This filament was used for all materials except for the first set of readings for Spodumene at which time a wider filament was used. In the picture of the apparatus the filament assembly has been placed atop the uoper coil for oicturization.

Immediately preceding this so-called vacuum tube in the apparatus is a vertical glass tube whose function is that of a mercury trap. The air upon being removed from the vacuum tube goes to the bottom of this trap and then up through a centrally-fixed tube and thence to the pump. This trap being vertically mounted permits the use of a cooling agent placed in a thermos bottle to be slipped over this trap and held in place. As the cooling agent was used crushed ice and salt which maintained a temperature in the neighborhood of -13°C for several hours. At this temperature the vapor pressure of mercury is negligibly small; of the order of .000048 cm. of Hg. In that way the pressure due to the mercury vapor in the system is kept at a minimum. Were it necessary to obtain a lower pressure for the mercury vapor dry ice or liouid

 $-6-$

air could be used to good advantage.

Next before the trap is placed the mercury diffusion pump which is standard in all respects.

To the glass tube which connects the trap and the pump is tapped the Me Leod gauge. No calibration for this gauge was necessary inasmuch as the absolute pressure within the system was not required. The apparatus was placed in operation only when the gas pressure in the system as read on the gauge was zero. The lower limit of the apparatus is of the order of 10⁻⁶ cm. of mercury. **Since the atmospheric pressure is used to raise the mercury level, the gauge is connected to the atmosphere and fore pump by a twoway glass stopcock. At this pressure the mean free path of the molecules is great enough to preclude the probability of collision of positive ion with gas molecule. Also arcing is not supportable at this pressure.**

Between the mercury pump and the fore pump is placed, first: A liter bulb containing Phosphorous Pentoxide which acted as a drying agent; taking up and holding the water vapor from the air which was let into the system each time the filament was removed. Second: This bulb is connected through a glass stopcock to the drying tube containing Calcium Chloride, which tube leads direotly through another glass stopcock to the fore pump, a standard Cenco Hy-Vac.

To obtain a measurable flow of positive ions the filament must necessarily be heated to incondescence. This was accomplished by the use, at first, of a storage battery. In series with the filament and battery were placed two rheostats in parallel; one a

-7-

low-resistance, high-load rheostat, and the other, for fine adjustments, a high-resistance, low-load rheostat. A D. C. ammeter *ifln* the "A" battery circuit then read the current flowing through the **filament. Before the problem was half completed, however, the storage battery source was discarded in favor of a transformer. An ordinary steo-down toy transformer served excellently, using the 6 volt output. This then was connected across the filament through the same two rheostats as before. The current flowing through the filament was then read from an A. C. ammeter inserted in this circuit.**

Since the object of the plate in the vacuum tube is to draw over to it any positive ions formed at the filament it must be suitably charged; which it is when the negative pole of some direct current source is connected to it. Two 45 volt Burgess batteries were used in this capacity. Being new units their volt**ages were somewhat above the 45 mark; the two of them in series giving a potential of 93.3 volts. Since such small currents were had** in the plate circuit no decrease was encountered in the "B" **potential other than that arising from shelf disuse, which for the duration of this experiment was negligibly small. At this point might be added the notion of electric field. In a vacuum tube of** this type the efficacy of the plate in drawing over to it charged **particles is not so much a function of the voltage between the filament and plate as it is of the electric field between them.** This field is defined as the potential gradient or $\frac{1}{d}$ where V is **the effective voltage and d is the distance between the elements. Thus it is seen that when the distance is made small the resulting**

 $-8-$

field can be quite large even when using a low voltage.

In this plate circuit then there must be some measuring device so that the various filament coatings can be compared. For this, current measuring instruments were employed. When the currents were sufficiently small a Leeds and Northrup wall-type D'Arsonval **galvanometer was used. The current sensibility of this instrument —8 is .686 x 10 amperes per millimeter deflection. However, the** range of this instrument did not exceed 1.7 x 10⁻⁶ amperes. For **currents above this a Weston three-range milliammeter was used which read directly to 2/10 of a microampere.**

Lastly in the plate circuit was used a 10,000 ohm resistance in series with the other plate circuit components. The purpose of this resistance is to prevent the formation of arcs across plate and filament when the filament emits gases. With a currant so small, the voltage drop in the resistor is negligibly small. As an instance, when the plate current i3 one microampere the fall across the resistor is 10⁻² volt.

PROCSDURE

Most of the substances used in this problem were obtained in the mineral form. Before application then it was necsssary to pulverize them. Previous workers have tried mixing the pulverized one with varnish in order to have the material adhere to the fila**ment. In this instance, however, the material in auestion was merely mixed with distilled water. It was applied to the filament with a lettering brush whereupon quite a uniform coating was obtained. When the water had evaporated the filament assembly could be placed in the apparatus and sealed. Pumping could then be started, using the fore pump. When the pressure became low enough to support a discharge leaks in the seal, if any, could be located by using a probing electrode which was connected to one terminal of an induction coil, the other terminal being connected to a receiving plate in the system. It might be said, however, that if there were any leaks their existence could be ascertained by the sound of the fore pump. However, the probe method located each one exactly.**

Before the mercury pump was started the ice and salt solution **was prepared and the thermos bottle slipped over the trap. Generally the mercury puma would take hold within twenty five minutes after the fore pump was started. Since the mercury oump is exceedingly fast in operation little time was reauired to lower the pressure to the desired point. Usually by the time the wiring was completed the outfit was ready to be operated.**

-10-

Ordinarily when working with currents of the magnitude en**countered here it is considered best practice to use soldered joints wherever possible and only copoer wire for connectors. However,** in looking for comparative results, this precaution was not deemed **necessary. Oopoer wire was used throughout though, and each wire was used in the same place in the circuit for all reading's. The usual Fahnestock connections were used at most terminals.**

Total radiation was measured for two operating temperatures. The first reading consisted of one hour's run with the filament drawing 3.8 amoeres, readings being taken at one-minute intervals. The second hour of readings was taken when the filament current was 3.1 amperes.

Occasionally it was found necessary to change from milliammeter to galvanometer or vice versa during a reading. This change could be effected in a few seconds and with the filament running so no change was made in the conditions nor were any readings missed on that account.

The temperature of the filament when drawing 3.1 amperes was measured by a Leeds and Northrup optical pyrometer and was found to be 904°C.

-11-

$\mathbf{V} = 92.3 \text{ volts}$

SPODUMENE

Flashed at 3.5 amperes for 15 seconds, Brought back to run at 3.1 amperes.

II Flashed at 4.2 amperes for 15 seconds; then brought down to 3.1 amperes.

"17-

-13-

16

Filament current = 2.8 amperes V = 92.3 volts

-10

POTASSIUM PHOSPHATE

POTASSIUM PHOSPHATE

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POTASSIUM PYROPHOSPHATE

POTASSIUM PYROPHOSPHATE

-27-

POTASSIUM PYROPHOSPHATE

At this point the filament was flashed for 15 seconds at 3.5 amperes. Readings follow:

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RESULTS

Perusal of the data will show that at one time the plate current is given in microamperes and at another it is expressed in millimeter deflections. This is purely for purposes of con**venience in that a current of 14 microamperes corresponds with a deflection of more than two thousand mm. Therefore in cases where the majority of readings lie beyond the range of the wall galvanometer the readings have been all converted into microamperes. Conversely those cases where the majority of the readings fell within the 250 millimeter range of the wall galvanometer have all readings in millimeters deflection. However, no confusion should result since in any comparison like units will be employed.**

The first of the substances tried was Spodumene, a mineral containing Lithium. The first trials on Spodumene are included for their interest value. At that time a trifle wider filament was used, thus allowing higher currents. When the filament drew 3.92 amperes the greatest plate current was 3.2 microamperes which corresponds to a deflection of 466 millimeters. From there it fell off fairly rapidly and then tapered off slowly to follow the exponential curve.

For a filament current of 4.98 amperes a plate current of 14 microamperes was obtained. This, as before, corresponds to a deflection of 2038 millimeters. This time the current fell off not so rapidly as before. This might lead one to believe that the filament, at this high temperature had become activated and thus

-29-

quite uniform emission was secured. This idea is somewhat further borne out by the next set of readings which were taken for a filament drain of 4.8 amperes. Here the microamperes in the plate **circuit ranged from a high of 3.0? to a low of 8.03, which tends to substantiate the activation idea. The last two readings of the last trial on Spodumene are missing due to the burning out of the filament between the 58th and 59th readings. This filament was 1.156 mm. wide whereas the one used on the other substances was 1.026 mm. wide.**

Although the second set of readings with the common filament occurred last from the point of view of time, it will be considered. next. Before taking readings on this substance it was flashed for 15 seconds at a temperature corresponding to a current of 3.5 amperes and then brought down to 3.1 amperes and read. The plate current built up from a value of 1.9 microamperes to a value of 3.63; and fell to 1.94 inside 30 minutes. Not quite the same effect was noted here before, perhaps because of two things. First, the temperature was not as high as in the previous experi**ment; and second, the readings were not taken at the flash temperature. However, there may be some doubt as to the validity of the second reason in that on activated filament is not operated continuously at the activating temperature.**

Following this little test it was again flashed for 15 seconds but this time at a filament current of 4.3 amperes. Upon lowering the current to 3.1 amperes and taking readings the plate current was found to respond only slightly and fall off from 3.94 to 1.97 microamperes in 9 minutes.

-30-

For Spodumene zero emission occurred at a filament drain of 2.17 amperes.

Next in order came the sources of Sodium ions; Cryolite, Nephelite, and Albite. These were first operated for 60 minutes each with the filament drawing 2.8 amperes. In the order named, the substances attained maximum plate currents corresnonding to 17, 190, and 76.millimeters deflection. For output then, Nephelite is superior to Cryolite and Albite. Both Nephelite and Albite are comparatively steady in their emission.

When these three were run at a 3.1 ampere filament current the following things were noted: The maximum plate currents were found to be proportioned to 14, 465.9, and 524.2 Millimeters deflection respectively for Cryolite, Nephelite, and Albite. There is but one exception. The reading for the 26th minute of Cryolite is missing since at that time the reading was off the scale. However, it came back almost immediately. The 465.9 and 524.2 mm. ere palpably conversion figures since the galvanometer scale does not go beyond 350 mm. Again Nephelite and Albite are quite steady, Nephelite being the more so.

The sources of Potassium ions were divided into 2 groups. The first includes Ort.hoclase and Leucitd, minerals both. At the 2.8 ampere filament rating the Orthoclase produced a maximum deflection of 407.7 mm. and the Leucite at maximum deflection of 190 mm. Of the two Leucite seems the more steady. When the filament **drew 3.1 amperes the maximum deflection due to Orthoclase was 207 mm. and that due to Leucite was 451.4 mm. Again Leucite seems the steadier.**

 $-31-$

Then the second group of Potassium compounds are rather interesting. The substances are Potassium Phosphate and Potassium Pyrophosphate. The first of these is quite deliquescent and when the **filament was put into the system there was quite a good deal of water introduced at the same time. By the time the pressure in the system permitted operation the filament coating was still damp. At 2.8 amperes zero deflection was noted on the galvanometer. It was then raised to 3.1 amperes but a zero deflection still obtained. However, within two minutes it started increasing. That was the start of a long run. In 25 minutes the current became 1 microampere and it continued climbing. One hundred fifty read**ings were taken that day and one hundred fifty more the next day. **That explains the drop from reading 150 to 150'. The current attained a maximum value at the 248th minute at 5.29 microamperes. The current remained remarkably steady for long periods of time. When the filament was removed the material was flaky and bunched on the filament. One niece about 3 mm. in length stood out from the side of the filament. Inside of a few minutes, however, the substance had deliquesced once more.**

Something like the same effect was noted in the case of Potassium Pyrophosphate. It, however, does not take up moisture quite as did the other. It was allowed to run at 3.8 amperes while the other was not. It also started at zero and attained a maximum at 49 minutes when the deflection was but 4.6 millimeters.

Immediately following this reading, however, the filament current was put at 3.1 amperes and the plate current started out at .39 microampere. After 128 minutes a maximum current of 4.52

-33-

microamperes was had in the plate circuit. So it rose to note **quite as high a value as did the Potassium Phosphate. After that time it decreased more or less steadily. After 160 minutes a flashing was tried; 15 seconds at 3.5 amperes reactivated the filament but the emission fell off quite rapidly.**

CONCLUSION

Here then are results obtained from studying different sources of positive ions under similar conditions. And the first inference to be made might be that if a prolific source of positive ions is desired, a substance ought be picked that oan be melted down on the filament.

If a source of Sodium ions is wanted, Nephelite is a good material to use both from the standpoint of actual emission and from the standpoint of uniformity of output.

Likewise a good source of Potassium ions is Potassium Phosphate. It has a relatively high output and furthermore it stands up well under continuous use. Of course, at the higher filament rating, 3.1 amperes, Potassium Pyrophosphate was almost as good from the standpoint of emission but does not stand up quite as long.

As was noted, Spodumene is a fine source of Lithium ions. This substance has been used considerably by workers in the study of positive ions and appears to be acoepted almost as a standard source for Lithium ions.

It may also be noted that of the substances tested there was none that failed to produce emission. Also it appears that of the materials used here the Potassium ions are obtained more easily than are the others. That is, they are more prolific sources of their ions at the operating temperatures than are the Sodium sources. This is of course a generalization and cannot hold in every case.

-34-