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EVALUATION OF TWT (TRAVELING WAVE TUBE) CATHODE
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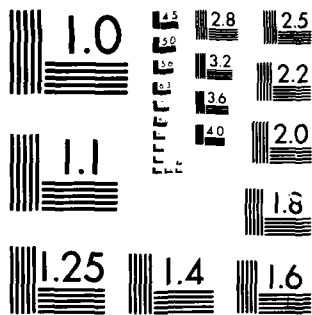
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Evaluation of TWT Cathode Impregnant Manufacturing Procedures

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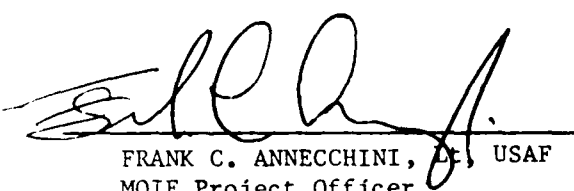
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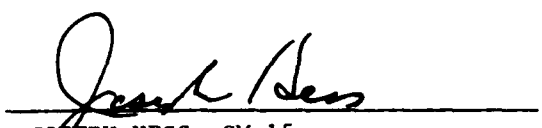
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This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.



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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Impregnant manufacturing procedures were evaluated as part of a program to develop guidelines for producing reliable dispenser cathodes. Such procedures must be controlled in order to eliminate variations in impregnant composition that can give rise to unpredictable cathode performance. Experiments revealed that variations in impregnant composition result primarily from contamination by moisture, incomplete mixing, and incomplete		

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reaction. Procedures developed to overcome these problems are discussed. The following recommendations are made: (1) Alumina must be dried prior to weighing to avoid inaccurate formulation due to moisture. (2) Multiple dry and wet mixing steps are needed to thoroughly homogenize the starting materials. (3) Calcination temperatures of $1300 \pm 60^\circ\text{C}$ are required to form thoroughly reacted barium calcium aluminates. (4) Thermogravimetry is useful for testing impregnants and starting materials for moisture contamination.

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I. INTRODUCTION

Better process control is required in the manufacture of traveling wave tube (TWT) dispenser cathodes so that more reliable and reproducible cathodes for space applications can be obtained. Current procedures for making dispenser cathodes have been developed empirically over the years and do not always contain sufficient controls over materials and methods to yield devices with the high level of reliability required for space applications.

This report describes a series of experiments to identify procedures critical to the production of reproducible cathodes. Based on these experiments, specific recommendations were developed to increase cathode reproducibility and production efficiency. Although it is also important, the effect of trace contamination on the performance of these impregnants in cathodes is not addressed here.

II. IMPREGNANT SYNTHESIS

The starting point of the synthesis of barium calcium aluminates in this study is the Philips Emet procedure outlined in Fig. 1:¹

1. Pure starting materials, barium carbonate (BaCO_3), calcium carbonate (CaCO_3), and aluminum oxide (Al_2O_3 ; alumina), are carefully weighed in the amounts required in the final formulation.
2. The starting materials are homogenized by ball milling of the dry ingredients, and a slurry is formed manually with distilled water.
3. The slurry is dried at low temperature and then is calcined at high temperature to form the barium calcium aluminate.

Each of these procedures is examined in more detail in the following sections.

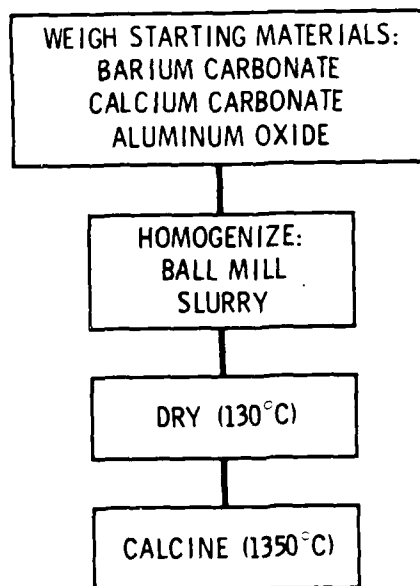


Fig. 1. . Block diagram of the Philips Emet procedure for synthesizing barium calcium aluminate dispenser cathode impregnants.

III. WEIGHING

The initial weights of barium carbonate, calcium carbonate, and aluminum oxide determine the concentrations of barium, calcium, and aluminum in the final impregnant. Much of the error in weighing can be eliminated using a well-maintained balance and a good weighing technique; however, the Philips Emet procedure does not account for the error introduced by the adsorption of moisture from the atmosphere. Moisture in the starting materials can cause uncontrolled variations in formulation and result in irreproducible cathode performance. In this section, weight gain as a function of relative humidity at room temperature is reported and a technique for screening impregnant starting materials for contamination by moisture is described.

The starting materials recommended by Philips were ACS reagent grade barium carbonate and calcium carbonate supplied by Mallinckrodt, and Linde type-B 0.05 μ m γ -alumina powder. An experiment was performed to examine moisture uptake of these materials. A total of 40 to 50 g of each starting material was dried overnight at 170°C and then weighed to ± 10 mg or $\pm 0.2\%$ of the component weight in a dry argon atmosphere. Either polyethylene or glass storage containers and weighing dishes were used, and the material was handled with a nickel-stainless steel spatula. The materials were exposed to controlled relative humidities at room temperature and then reweighed. The effect of humidity on weight gain is plotted in Fig. 2 and listed in Table 1 after 5 and 22 to 24 hr.

The weight gain for barium carbonate is approximately linear with relative humidity, reaching a maximum of 0.24 weight percent at 23°C during exposure to 100% relative humidity. Calcium carbonate adsorbed 0.03 weight percent of water. Both compounds show little additional weight gain between 5 and 24 hr of exposure to 100% relative humidity.

Alumina exhibited a weight gain up to about 6 weight percent at 100% relative humidity after 24 hr; the weight gain is fairly linear with relative humidity. In addition, dry alumina required at least 24 hr for its weight

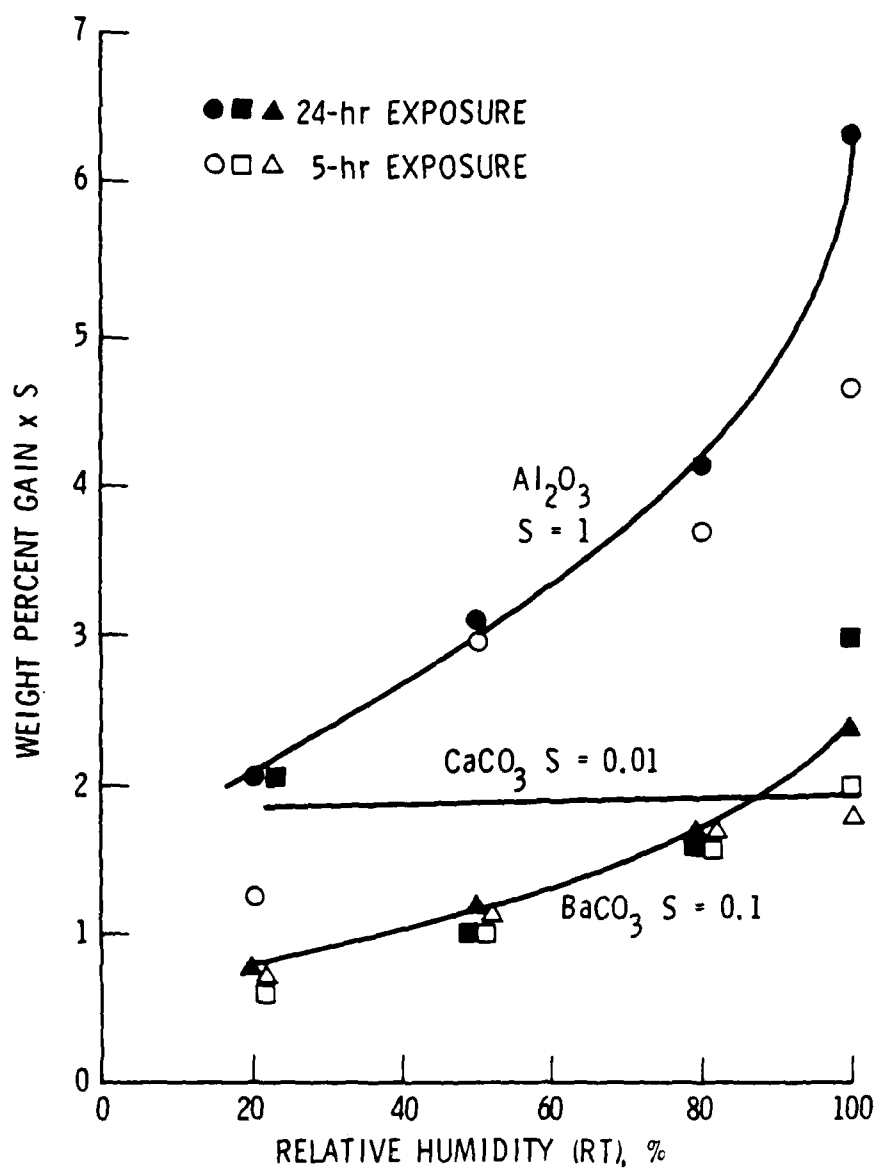


Fig. 2. Weight gain of starting materials due to humidity at 23°C after 5 and 24 hr.

Table 1. Weight Gain of Starting Materials Due to Humidity at 23°C

Compound	Relative Humidity (%)	Exposure Time (hr)	Weight Gain (wt%)
BaCO ₃	20	5	0.07 ± 0.00
		22	0.08 ± 0.00
	~50	5	0.11 ± 0.01
		23	0.12 ± 0.01
	80	5	0.16 ± 0.00
		24	0.16 ± 0.01
	100	5	0.18 ± 0.06
		24	0.24 ± 0.02
CaCO ₃	20	5	0.006 ± 0.005
		22	0.02 ± 0.01
	~50	5	0.01 ± 0.00
		23	0.01 ± 0.00
	80	5	0.015 ± 0.000
		24	0.015 ± 0.001
	100	5	0.02 ± 0.02
		24	0.03 ± 0.02
Al ₂ O ₃	20	5	1.25 ± 0.07
		22	2.05 ± 0.07
	~50	5	2.95 ± 0.07
		23	3.15 ± 0.07
	80	5	3.70 ± 0.14
		24	4.15 ± 0.07
	100	5	4.65 ± 0.07
		24	6.35 ± 0.07

to stabilize in humid air. Therefore, humidity causes weight gain in alumina that can lead to an aluminum deficiency in the impregnant. In contrast, barium carbonate and calcium carbonate are much less hygroscopic. This difference in moisture adsorption is expected because the alumina has small grain size and very high surface area relative to the carbonates.

Generally, a compound must be dried prior to weighing when the accuracy required is greater than the weight gain due to moisture uptake during storage and handling. Thus, for most applications alumina will require drying.

Because moisture contamination of impregnant starting materials can cause errors in formulation, a procedure is required to determine the extent of such contamination. Thermogravimetry (TG) is a rapid and convenient procedure for measuring the amount of loosely bound water in the starting materials. In TG, the temperature of the sample is linearly increased as the weight loss of the sample is recorded. In Fig. 3 an improperly stored 5:3:2 starting material was heated at a rate of $14^{\circ}\text{C}/\text{min}$ in air flowing at $700\text{ cm}^3/\text{min}$. The data in the figure show that at temperatures below 780°C a mass fraction of 0.046 was lost. This fraction is consistent with water loss from the alumina starting material discussed in this section. The other breaks in the curve, corresponding to calcium carbonate and barium carbonate decomposition, are discussed in Section V. By running each starting material alone, TG can also be used to evaluate the extent of water contamination in individual starting materials. Thus, TG provides a convenient and sensitive way to control moisture contamination of impregnant starting materials.

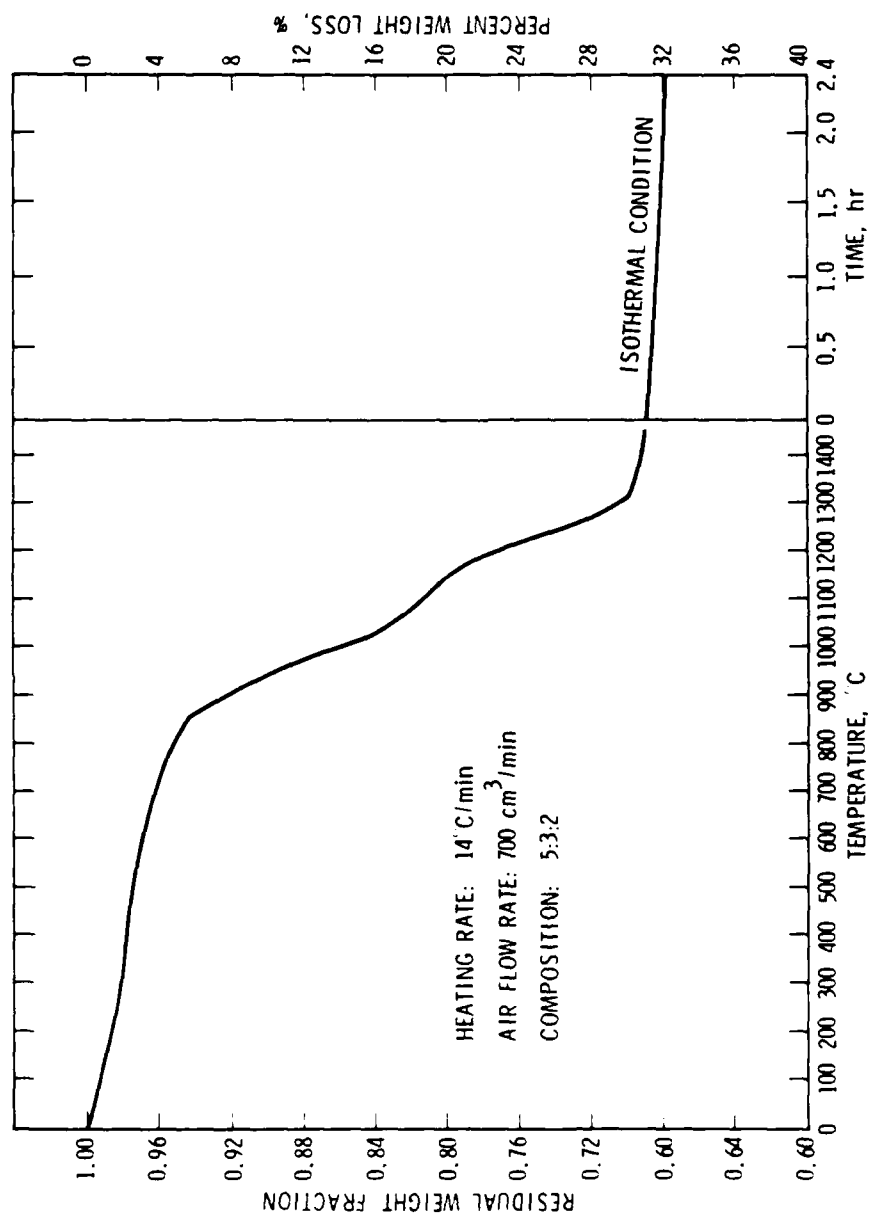


Fig. 3. Thermogravimetry of moisture-contaminated impregnated starting materials.

IV. MIXING

We developed an improved mixing procedure that results in complete reaction of the starting materials to form homogeneous barium calcium aluminate impregnants. The Philips Emet mixing procedure consists of only two steps: dry mixing followed by wet (slurry) mixing. Dry mixing homogenizes and grinds the powder; wet mixing increases the contact between particles and forms reactive hydroxides. However, more extensive mixing than that specified in the Philips procedure is required. The repeated use of dry and wet mixing steps increases the reactivity of the mixture and ensures complete and uniform reaction in subsequent processing steps. In this section, improved procedures are detailed and the advantage of more extensive mixing is illustrated.

The following improved mixing procedure was examined. A total of 50 g of starting material was loaded into a 000 size Burundum ball mill jar containing 7 to 10 Burundum cylinders, 13/16 in. long $\times$ 13/16 in. in diameter. The dry starting materials were milled for at least 24 hr and were occasionally scraped from the sides of the jar with a nickel-stainless steel spatula. Caking of the powder was less severe if the jar and the cylinders were dried in an oven at 130°C immediately before use.

The wet, or slurry, mixing step further homogenized the raw materials. After ball milling, the powder was scraped with a nickel-stainless steel spatula into a glass beaker; about 30 ml of water was added to form a paste with the consistency of heavy cream. This mixture was stirred with the spatula for another 5 min; it was then dried in the beaker for 1 hr in an air oven at 130°C. The dried slurry was ground in an agate mortar, and water was once again added to produce a slurry. This wet mixing procedure was repeated up to three times on a sample before loading the slurry into an alumina boat for drying at 130°C and calcining.

An experiment was performed to determine the effect of slurry mixing on the composition of the $5\text{BaO}:3\text{CaO}:2\text{Al}_2\text{O}_3$ impregnant. The raw materials were ball milled and then were manually slurried and dried at 130°C. The slurry and dry cycle was done up to three times. After calcination at 1350°C for

3 hr the impregnants were dissolved in 2M HCl and were analyzed by flame atomic absorption/flame emission spectroscopy (AA/FE), a technique that measures the concentration of acid-soluble ions. Apparent discrepancies in the measured impregnant compositions were due to unreacted, insoluble phases, such as alumina starting material in the impregnant.

The observed aluminate composition as a function of the number of slurry mixing steps is listed in Table 2. The AA/FE results were converted to mole ratios and normalized to BaO = 5 to simplify the comparison to the expected formulation, $5\text{BaO}:3\text{CaO}:2\text{Al}_2\text{O}_3$. The uncertainties in the table are standard deviations based on the average of three samples.

The calcium and aluminum concentrations are consistently low in the impregnant slurried once. Furthermore, aluminum shows a large variation in the aluminates made from materials slurried once although the results are within the expected formulation. This variability is due to an unreacted aluminum oxide phase in the impregnant, which was confirmed by ESCA examination of the precipitate remaining in the 2M HCl solution as shown in Fig. 4. Samples slurried three times before calcination were very close to the expected composition. Therefore, the slurry mixing step will increase the reactivity of the calcium carbonate and the alumina and thereby ensure that a more uniform impregnant composition can be obtained.

We simplified the alternate mixing procedure to reduce the handling of the materials and to improve homogeneity. The procedure consists of dry milling the powder for 2 to 24 hr; adding the 30 ml of water directly into the ball mill and wet milling for about 5 min; drying the impregnant in the ball mill jar at 130°C; and repeating the dry and wet milling cycle up to a total of three times. More uniform material results from this procedure in the ball mill jar with less chance of contamination from handling.

Table 2. Effect of Slurry Formation on the Observed Formulation of the (5BaO:3CaO:2Al₂O₃) Impregnant

Number of Slurry and Dry Cycles	Observed Formulation (mole ratios)		
	BaO ^a	:	CaO : Al ₂ O ₃
1	5	:	2.85 ± 0.14 : 1.81 ± 0.29
3	5	:	2.95 ± 0.14 : 1.94 ± 0.07

^aNormalized to Ba = 5.

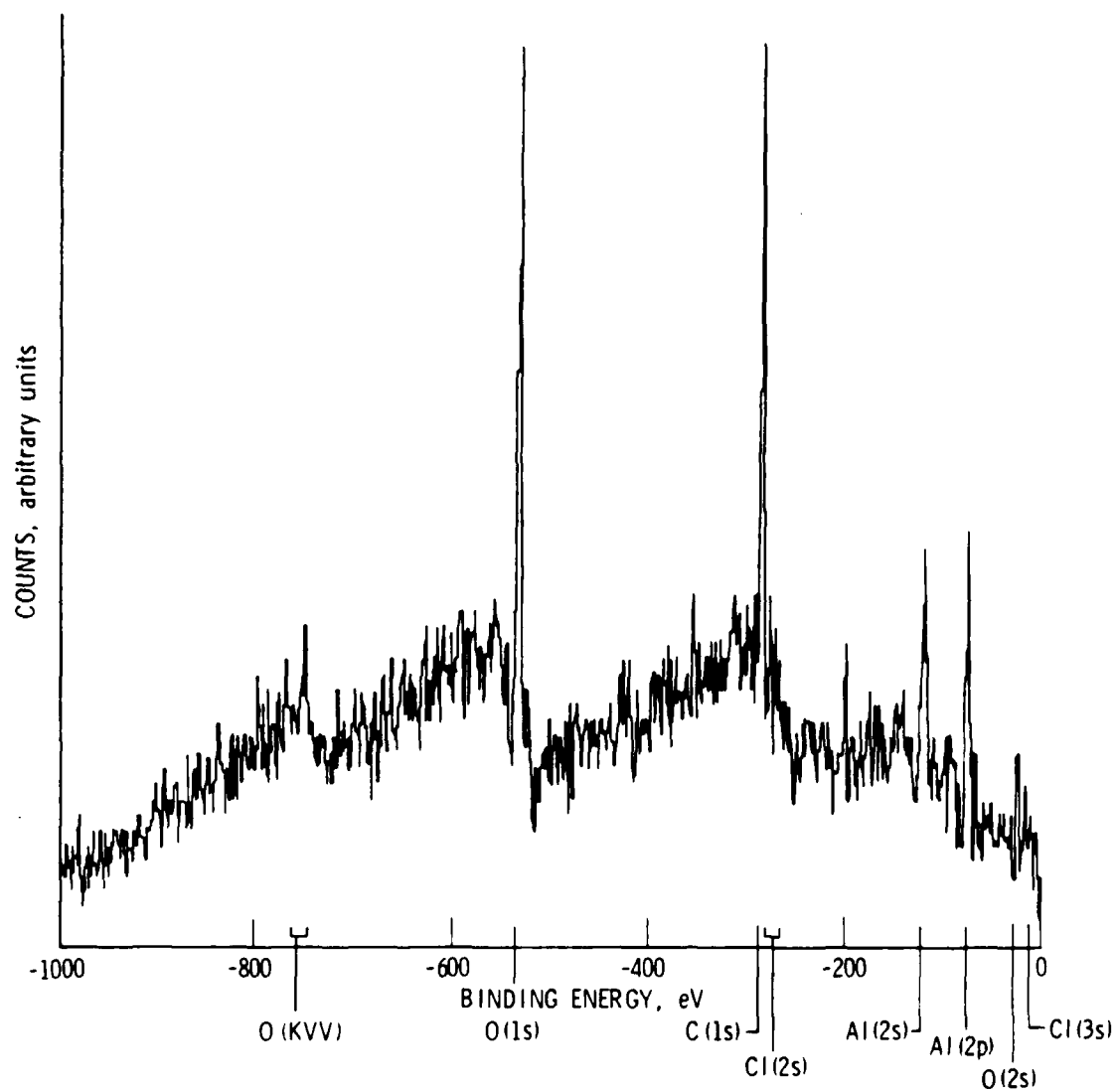


Fig. 4. ESCA spectrum of the alumina precipitate remaining after impregnant dissolution in 2M HCl.

V. CALCINATION

During calcination in the Philips procedure, the raw materials are heated to 1350°C. At this temperature, the carbonates decompose and react to form homogeneous barium calcium aluminates. Homogeneous impregnants are required for reproducible impregnation of porous dispenser cathodes. The current practice is to calcine the impregnant at 1250 to 1350°C for a time interval depending on the temperature and batch size.

In this section, the processes that occur due to heating or calcining the starting materials are investigated. First, TG is used to determine the minimum temperature required to drive off carbon dioxide from the carbonate starting material mixture. Second, ways of handling the impregnant after calcination to avoid contamination by moisture are examined. Third, the effect of calcining temperature on the homogeneity of the impregnant is investigated.

Knowledge of the decomposition temperatures for the barium carbonate, calcium carbonate, and aluminum oxide starting material mixture described in Section III is needed so that the time-temperature schedule for calcination can be established. These decomposition temperatures were determined by TG.

Two features appear in a TG curve that can be used to characterize the calcination:² (1) the temperature at a break in the curve represents the initialization temperature of a reaction, and (2) the slope of the curve represents the reaction rate. The experimental conditions, including sample size, sample holder geometry, gas flow rate and composition, and thermocouple placement and calibration, can alter the details of TG curves. In general the cumulative errors, particularly from rapid temperature ramping rates, overestimate the initialization temperatures. Thus, TG was used to set upper limits on temperatures associated with decomposition reactions. The data shown in Fig. 5 were obtained for a 30 μg sample of 5:3:2 mole ratio of $\text{BaCO}_3\text{:CaCO}_3\text{:Al}_2\text{O}_3$. Argon was flowed at 100 cm^3/min to remove the carbon dioxide product, and the temperature ramping rate was 10°C/min.

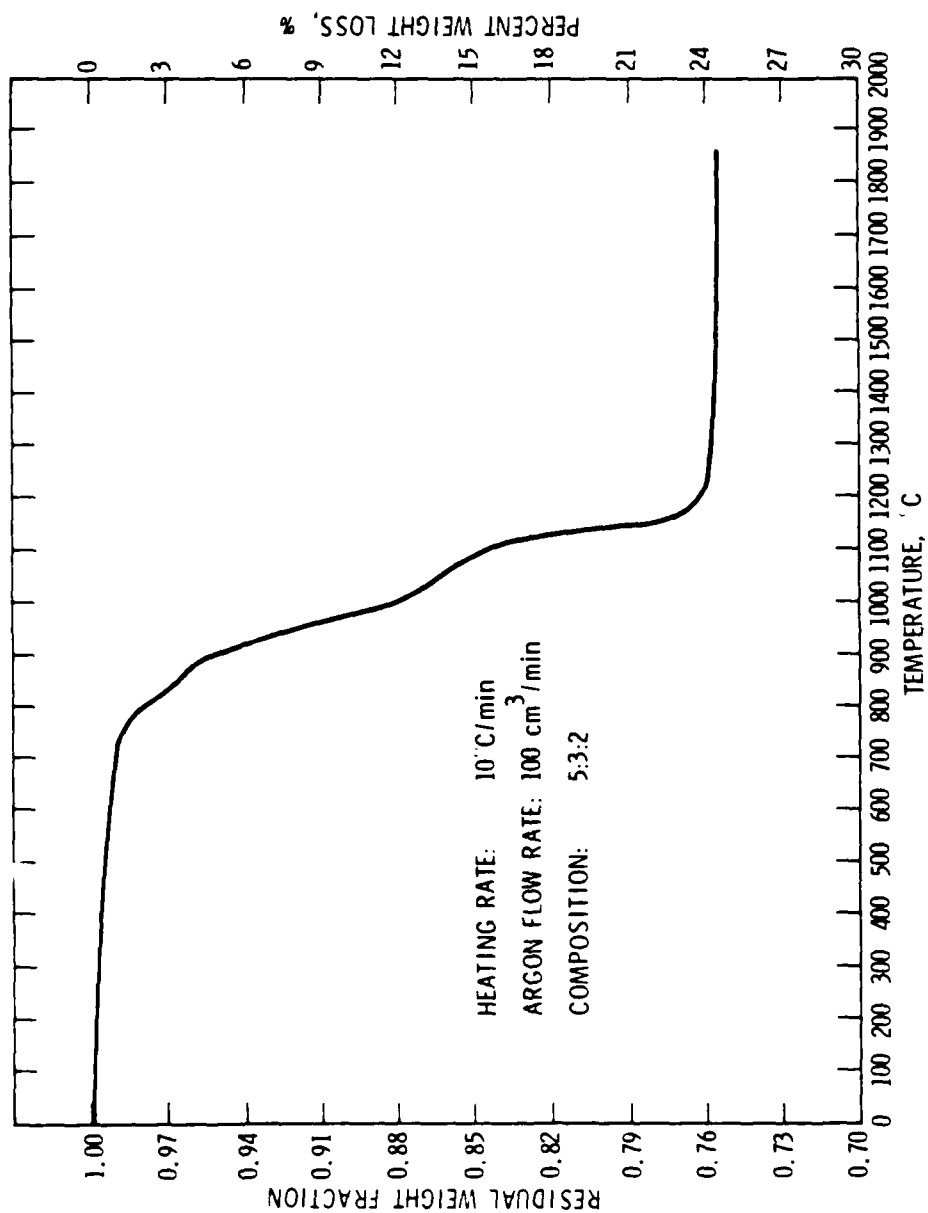


Fig. 5. Thermogravimetric analysis of barium calcium aluminate starting materials.

Figure 5 evidences several breaks in the curve. Three important regions of mass loss are:

1. Room temperature to 785°C, where the mass fraction lost is 0.017 due to the evolution of water vapor;
2. 785 to 1000°C, where the mass fraction lost is about 0.10 due primarily to CaCO_3 decomposition; and
3. 1000 to 1200°C, where the mass fraction lost is 0.13 due mainly to BaCO_3 decomposition.

The mass fraction of the original sample weight lost from 785 to 1200°C is about 0.23, which is in excellent agreement with the expected mass fraction of 0.24 for carbon dioxide driven off from a 5:3:2 impregnant. Furthermore, the temperature range observed in this experiment is consistent with the decomposition temperatures for CaCO_3 in interval 2 and for BaCO_3 in interval 3 of 600 to 1050°C reported by Ogden.³

To model calcination more accurately, TG was used under isothermal conditions. About 30 mg of 5:3:2 impregnant starting material was heated to about 1050°C and held at that temperature with an argon flow rate of 100 cm³/min. At this temperature, 4 hr was required to completely calcine the impregnant. (See Fig. 6.) Thus, as long as the calcining temperature is greater than the decomposition temperature of the carbonates, calcination will occur, limited only by the removal rate of carbon dioxide in a large batch of material. Temperatures from 1250 to 1350°C may be required for calcination times of hours to days.

Once the impregnant has been calcined, contamination by moisture must still be avoided. Exposure of the impregnant to humid air can result in the formation of compounds that will affect the melting behavior of the impregnant. To study these reactions, a calcined impregnant was exposed to 100% relative humidity (defined at 20°C) as the impregnant was cooled from 1300°C to room temperature. The data in Fig. 7 show that weight gain due to the reaction of the impregnant with water begins at about 290°C and becomes rapid at temperatures lower than 50°C. Thus, the hot, calcined impregnant must be removed from the furnace and be stored immediately in a desiccator to avoid contamination.

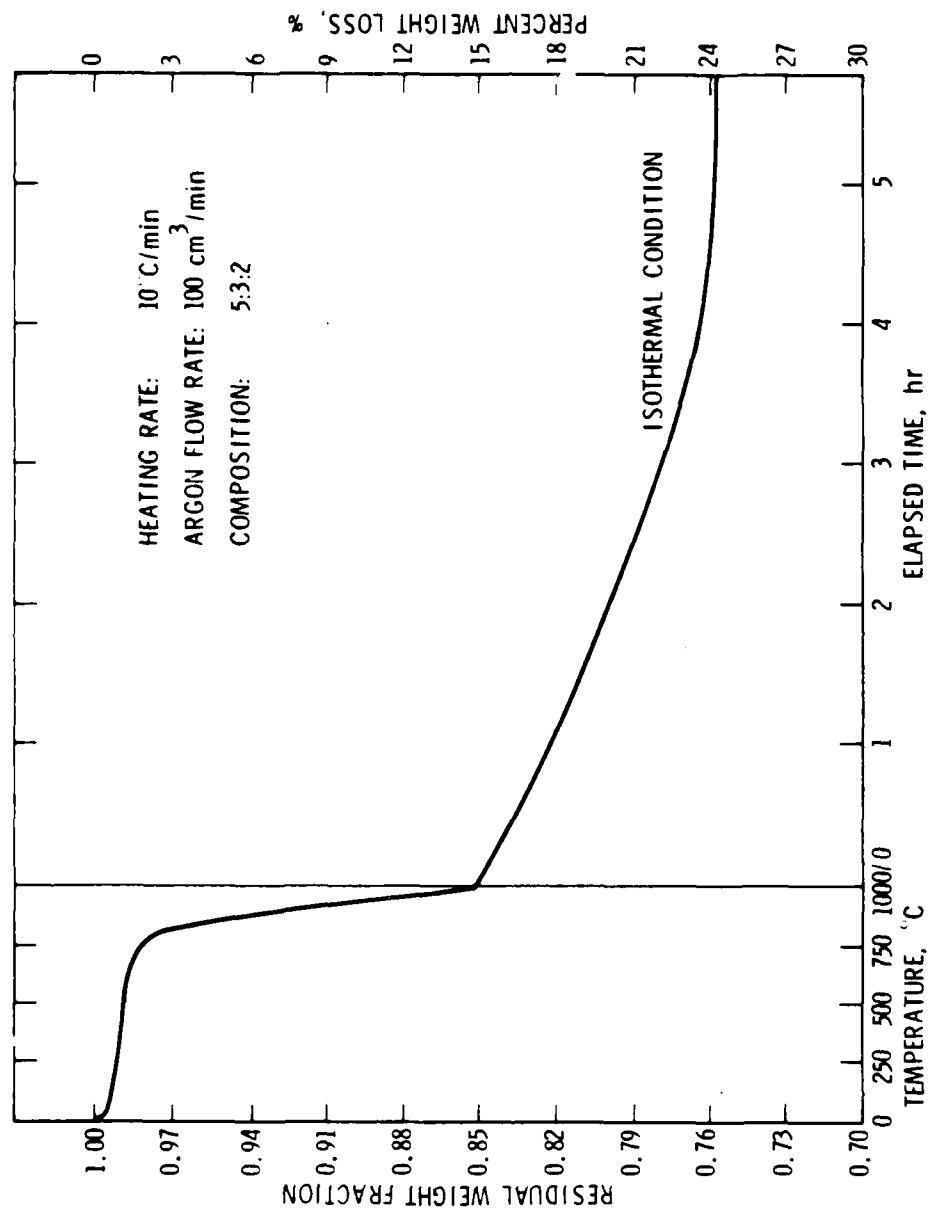


Fig. 6. Isothermal TG of barium calcium aluminate starting material.

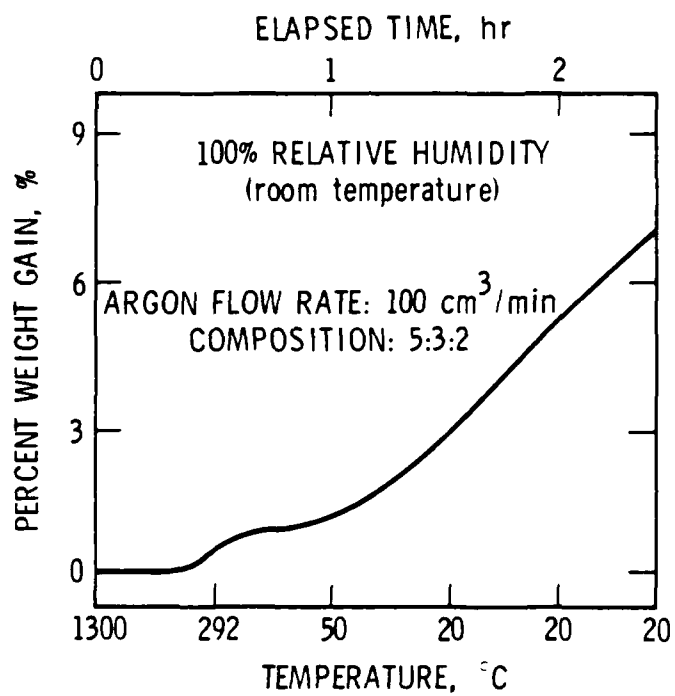


Fig. 7. Weight gain in a calcined impregnant due to moisture exposure.

The effect of the variation in calcining temperature on the aluminate-forming reaction was investigated. Three 2-in.-long alumina boats were filled with raw material from a single batch. They were placed at different positions in a tube furnace so that the material in each boat was calcined at a different temperature. The aluminates were analyzed by flame atomic absorption/flame emission spectroscopy for barium, calcium, and aluminum after calcination for 3 hr. The results are given in Table 3. The calcium and aluminum concentrations are somewhat low in the two low-temperature samples, indicating the presence of unreacted Al_2O_3 and CaO . (The analytical technique is insensitive to these oxides because they are only slightly soluble in the 2M HCl used to dissolve the aluminates for analysis.) A few percent of the unreacted material is probably not significant during impregnation since it should be quickly dissolved in the melted impregnant during impregnation. Therefore calcining temperatures within 60°C of 1300°C do not affect the properties of the impregnant, and temperatures as low as 1250°C are sufficient to calcine the impregnant.

Specific recommendations of calcination time and temperature cannot be made because furnace conditions vary. However, some general guidelines have been developed based on the results reported here. Two types of tests are required to develop a calcination schedule for a specific furnace. First, consistent with current practice, impregnants must be calcined until the expected weight loss is observed. Second, the calcined impregnant should be analyzed to confirm that the starting materials have been fully reacted. Because of its lack of sensitivity to the small grain size of the alumina, X-ray powder diffraction is not a reliable way to confirm the complete reaction of the alumina starting material. Chemical separation of unreacted alumina may be required.

Table 3. Effect of Temperature on Formulation of
(5BaO:3CaO:2Al₂O₃) Impregnants Calcined
for 3 hr

Calcining Temperature (°C)	Observed Formulation (mole ratios)		
	BaO ^a	:	CaO : Al ₂ O ₃
1216	5	:	2.89 : 1.89
1293	5	:	2.85 : 1.92
1343	5	:	3.11 : 2.02

^aNormalized to Ba = 5.

VI. SUMMARY AND RECOMMENDATIONS

Conventional processing (based on the Philips Emet procedure) must be improved so that reproducible, homogeneous cathode impregnants for space dispenser cathodes can be produced. Several recommendations to improve impregnant processing based on this work are summarized here.

1. Weighing deficiencies of up to 6% can result when wet alumina is used. Alumina must be dried prior to weighing.
2. Complete mixing of finely powdered starting materials is essential to ensure complete reaction during calcination. Several dry and slurry mixing steps are required to achieve homogeneity.
3. The calcining conditions in a particular furnace must be set by testing for the complete reaction of the materials in addition to achieving the theoretical weight loss.
4. Thermogravimetry can be used to test impregnants and starting materials for contamination.

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