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TECHNICAL REPORT ARLCD-TR-80043

PHOTOFLASH COMPOSITIONS DESENSITIZED BY COATANTS

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LARGE CALIBER
WEAPON SYSTEMS LABORATORY
DOVER, NEW JERSEY

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20. ABSTRACT. (Cont'd)

The method finally selected was contact coating from a wet medium employing an electrically conducting carbon black as coatant. During the program there was a scale-up from laboratory bench quantities to a 5 kg batch. For the larger batches a counter current mix muller was employed.

The method is safer than the non-proprietary standard methods previously used, since the material is wet or damp throughout the blending process. Furthermore, the coating, by its ability to leak off voltage, reduces the electrostatic hazards during the blending and further processing of the powder.

The product obtained is stable and equals the performance of the uncoated formulations.

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SUMMARY

A process for blending photoflash powder was investigated in which the powder was coated with material for the purpose of decreasing the hazard.

Various coatant candidates were applied by both coacervation and contact methods, and the resulting formulations characterized with respect to physico-chemical, sensitivity, stability, and performance properties.

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OBJECT

To establish a method of blending flash powders which is safer than previously used conventional blending, and which could be readily used by many manufacturers.

INTRODUCTION

Conventional techniques used in the past for blending of photo-flash powder have resulted in some explosions, damaging the facilities of several contractors including Kilgore Manufacturing, U.S. Flare and Bermite. These methods involved the tumbling of the dry ingredients.

A proprietary technique is employed by one contractor in which the blending is done after filling of the round with the individual ingredients. This blending operation, which eliminates the need for subsequent filling operations, is done remotely and minimizes possible injuries to personnel. However, in this method the powder does not completely fill the round, thereby detracting from the light output; also, the proprietary nature of the process restricts its use by other contractors.

Employing a blending process which is inherently safer is highly beneficial. In addition, if the resulting product has decreased sensitivity, the subsequent filling operation becomes less hazardous, thus resulting in the added benefit of a manufacturing process of enhanced safety.

A safe manufacturing method which could readily be used by a number of contractors and thereby increase the number of potential contractors beyond the limited number (in some cases a single one) now responding to current bids would be very advantageous.

The process decided on was one in which the composition or an ingredient of the composition is desensitized by a coatant. Two methods of applying the coating, coacervation, and contact, were considered and are done in the following manner:

(1) Coacervation: In this method the ingredients are suspended in a solution of the coatant by rapid stirring and a liquid added which decreases the solubility of the coatant in the resulting mixed solvents to the point where it comes completely out of solution. In so doing, the coatant surrounds aggregates of the ingredients, which fall to the bottom of the vessel when stirring is discontinued. The formulation is then separated from the supernatant and washed and dried.

(2) Contact: In this method the ingredient or composition to be coated is placed in contact with a solution or suspension of the coatant, either on a filter or by mix mulling. The solvent is allowed to evaporate leaving the product coated.

Using these methods for contact coating other formulations has resulted in products which are relatively low in sensitivity to initiation by friction and electrostatic charge. In the case of coacervated material, products are produced which are homogeneous, uniform, and free flowing. For example, delay compositions used in RAP rounds have been coacervated with 1% Viton A, and found to give a uniform product (ref 1).

Contact coating of igniters by organosiloxanes has been reported in U.S. Patent No. 3,110,638, Nov 12, 1963 by Murphy and Larrick (ref 2), to markedly decrease sensitivity of the compositions to friction and electrostatic discharge.

The work of Brenner and Timlin (ref 3), demonstrates the ability to contact coat metal powders effectively with chromate and micro-crystalline wax coatings.

EXPERIMENTAL

Blending Methods

Blending was accomplished either in the dry state by tumbling the ingredients in a rubber container together with rubber stoppers on a modified Abbe ball mill, or in a wet medium by gentle mulling as a paste. When laboratory bench quantities were involved, the wet blending was done in a mortar or evaporating dish; when scaled up to kilogram quantities a counter current batch mixer was used. In the wet blending, mixing was continued until the composition was only slightly damp, at which time it was removed to a pan for final air drying.

Product Characterization

Work was carried out on the formulation to determine characteristics such as performance, sensitivity, and physico-chemical properties in the following manner:

Performance

The Poppy photoflash cartridge (Dwgs No. XP 111206 and XP 111218), a miniaturized component, was used as a test vehicle for determining light output of the formulations. Peak light intensity,

time-to-peak and integral light were determined from time intensity traces obtained using an ICI corrected photometric head, a memory oscilloscope with recording camera, and a 40 millisecond dual channel integrator and triggering cell. Light outputs were obtained shortly after loading of the rounds, and for surveillance purposes with the control and selected formulations, after storage at time intervals of up to one year at room temperature and 160°F.

Sensitivity

Impact

Picatinny Arsenal impact tester using a 2 kg weight as described in reference 4.

Friction

Picatinny Arsenal friction pendulum and Allegany Ballistics Laboratory tester (ref 4,5).

Electrostatic

Electrostatic equipment was employed which uses selected capacitances and a variable fixed gap. The breakdown voltage is not preselected with this equipment, but instead, the capacitors are charged and when the voltage for a particular medium and gap reaches the breakdown value, discharge occurs. Usually, the needle was placed in contact with the surface of the material under test, a zero air gap, the discharge then occurring through the air pores. With highly conductive samples the voltage is leaked off and an air gap must be provided to allow breakdown to occur. In the early testing minimal resistance was used in the discharge circuit, but in the later testing 5000 ohms was used in series when it was found the sensitivity increased at moderate resistances.

Compatibility

Compatibility of coatant with the formulation was determined using the vacuum stability test, which measures the quantity of gas involved after heating for 40 hours under vacuum at 120°C.

Physico-chemical

Flow

The ability to flow was determined by placing a standard weight of sample on a series of funnels of varying stem diameter, and counting the number of taps necessary to cause the sample to pass completely through, or determining the smallest diameter through which the sample passes with no tapping necessary.

Charge Buildup

The charge on a sample after passing through the funnels was determined in relative units, using a Portable Electrostatic Field Meter, Monroe Electronics Inc., Model 225 with Model 1007E Probe. A standard container setting on a standard surface was used to collect the sample, and the measurement was made at a standard distance after a standard time interval.

Apparent Density

The determination of weight per unit volume of the formulations was conducted by measuring the volume of a given weight of sample in a graduated cylinder after tapping until the volume no longer changed.

Thermal Conductivity

This property was determined by observing the temperature change experienced by a probe at an elevated temperature after contact with the formulation at room temperature, and comparison of values obtained with known materials. The instrument used was a Thermal Comparator manufactured by the Thermal Properties Research Co., Lafayette, Indiana. Further details on the method may be found in appendix A.

RESULTS AND DISCUSSION

Formulations were prepared starting with 20 gram batches and as the program progressed scaling up successively to 100 gram, and 300 gram batches with the more promising coatant candidates.

When the most promising candidate was selected based on sensitivity, physio-chemical, and illumination properties, scale-up to 5 kg was accomplished. From this batch, Poppy cartridges were loaded for storage surveillance. Details on the method of blending, done in a counter current mix muller may be found in appendix B.

Initial candidate materials selected for coating by coacervation were vinyl alcohol acetate resin (VAAR) and Viton A, a fluorinated polymer. Coating of these materials was also accomplished by contact. With the VAAR, the "as received" solution in methyl acetate was diluted with ethanol; with the Viton, a solution in acetone was used. Examination of the properties of the compositions so prepared indicated them to be at least the equivalent of products obtained by coacervation. In view of this, and the greater complexity of the coacervation process, it was decided to restrict further work with Viton A and VAAR to contact coating.

In addition to the Viton and VAAR, coatants applied by the contact method were; DOW 200 Silicone Fluid in 1,1,1 trichloroethane; Dow-Corning XZ-6-1082 in 1:1 acetone-toluene solution; Anstac M solution, TWEEN 80 in ethanol; Carbon Black (Cabot, Regal SR); CAB-O-SIL; calcium resinate and sulfur. These were used with the Type III Class A photoflash powder consisting of 30% potassium perchlorate, 30% barium nitrate and 40% aluminum. In addition to investigation of formulations in which the coating was applied during blending, a formulation was employed in which the aluminum was pre-coated with isostearic acid. Also, a formulation containing 8 μ aluminum instead of the nominal 20 μ specification material was investigated.

When compositions were blended wet, the mixtures were evaporated to dryness. Compositions employing Viton A and VAAR required granulation while damp; with these formulations a further grinding step was used, otherwise densities were found to be excessively low.

Some key physio-chemical and sensitivity data comparing the control photoflash composition with compositions having coatants are found in table 1.

Values given for flow are in relative units, the greater the number the better flowing the material. The sensitivity values, obtained in one particular location, are valid only for comparison among themselves and should not be compared with data obtained employing other equipment. Of considerable interest is the increased electrostatic sensitivity of the photoflash formulation when the finer aluminum is used.

Light output characteristics of selected formulations loaded into Poppy photoflash cartridges may be found in table 2.

Based on the results of the observed characteristics, it was decided to limit further work to the coatants vinyl alcohol acetate and carbon black, both at the 1% level.

The investigation continued comparing formulations coated with vinyl alcohol acetate resin and two different Cabot Corp. carbon black types, Regal SRF-S and Sterling R with the standard uncoated photoflash composition.

Negligible quantities of gas were produced by all of the above formulations in the vacuum stability tests, indicating the coatants to be compatible with the base composition.

Friction sensitivity was obtained at the Allegany Ballistics Laboratory. Description of the method and results may be found in reference 5. The compositions employing VAAR and Sterling R were found to be slightly less sensitive than the composition employing Regal SRF-S, which in turn was approximately as sensitive as the control composition having no coatant.

Additional data was obtained on light output from Poppy cartridges loaded with the formulations. The output from the VAAR coated formulations was significantly lower than those obtained from the two carbon-black coated and control compositions which were equivalent to each other (table 3).

Comparison of the two carbon blacks left little to choose from, including additional impact sensitivity testing employing the Picatinny Arsenal tester, as described in reference 4. The sample containing the Regal SRF-S had a value of 33 inches, while that for the sample with the Sterling N had a value of 31 inches, as did the control composition.

Storage surveillance to determine stability was carried out on the formulation with the final selected coatant candidate, carbon black. A 5 kg batch of composition was prepared in a counter current mix muller, and Poppy cartridges filled. Cartridges used as controls were also filled with the standard Type III Class A photoflash powder.

The cartridges were placed in storage both at 71°C (160°F) and at room temperature, and removed at intervals of up to one year. The rounds were then fired at ambient temperature to determine light output. The values of integral light, the property used to assess stability of the formulation, were then compared with those obtained prior to storage (table 4).

No spontaneous ignitions or any other adverse incidents were encountered during storage. Light output at each storage interval for the rounds with coated composition was about equal to that of the control rounds for both ambient and elevated temperature storage

conditions. Values were slightly higher after the final two storage intervals than those obtained initially. These results indicated the formulations to be stable.

Earlier in the program it was implied that the carbon black coated formulation was less sensitive to electrostatic discharge than the uncoated material (table 1). However, the comparison was made for testing at different conditions, the uncoated material with the needle touching the surface of the formulation and the coated material with an air gap. As indicated earlier, with the needle in contact or in close proximity to the carbon coated material causes the voltage to leak off. During the further development of the electrostatic equipment and procedures under another program it was found that results would be markedly affected by parameters such as sample thickness, air gap and resistance of the discharge circuit. Accordingly, when the carbon black coated and uncoated material were compared at comparable conditions the sensitivities were equivalent. As the air gap increases a greater amount of energy is required to bring about ignition; also, as is well known, as the gap increases a greater voltage is required for breakdown. With 5 kilohms in the discharge circuit about the lowest energy values are obtained for ignition. These values of a few tenths of a joule are still well above the energy of 0.015 joules, generally considered to be the level that can be generated by a person.

This consideration, together with the narrow gap range over which a breakdown can occur, might indicate that there is but little hazard threat from electrostatic discharge. This might be true when considering macro samples of quiescent material. However, with material under motion, and where a small cloud can form, high voltage may be generated over a wide gradient of air gaps. This could well present a hazardous set of circumstances, particularly if a micro sample of material with fine aluminum is isolated.

The hazard is reduced by the greater ability of the carbon coated material to leak off any charge being generated. This, together with the wet blending of the formulation makes for processing which is safer, and a product which is safer to handle than that experienced in the standard method previously used in the manufacture of photoflash cartridges.

CONCLUSIONS

It has been demonstrated that a method of blending photoflash powder may be used which is safer than the nonproprietary standard methods previously used. The method involves wet blending in a counter current mix muller, simple equipment currently in use by or

readily available to a large number of contractors. The coating of the formulation during blending with an electrical conducting carbon black reduces electrostatic hazards during the blending and further processing of the powder.

The product obtained is stable and performs as well as formulations which are uncoated.

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2. M.F. Murphy and B.F. Larrick, "Controlled Sensitivity Igniter Composition and Method of Producing Same," Patent No. 3,110,638, Nov. 12, 1963, assigned to U.S. Government.
3. W. Brenner and J.J. Timlin, "Environmental Protection of Pyrotechnic Products and Devices," Report No. 1 Feb 1973, Contract DAAA 21-73-C-0117.
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5. L.S. Treadwell, Specific Problem Report No. 618 "Friction Sensitivity Testing of Pyrotechnic Photoflash Compositions," Allegany Ballistics Laboratory, HERC No. 75-4, Report No. A0276-520-03-001, December 22, 1974.

Table 1. Physical and sensitivity properties of formulations

| Coatants ^a | Density, g/cm ³ | | Flow | Residual chge | Impact inches | Sensitivity Electrostatic | |
|------------------------------|----------------------------|------|------|---------------|---------------|---------------------------|---------------------------|
| | bulk | tap | | | | Joules | No. ignited per 10 trials |
| None | 1.31 | 1.75 | 16 | 160 | 10.5 | 1.0 | 5 |
| Dow 200 | 0.86 | 1.47 | 7 | 20 | 7 | 3.6 | 3 |
| Viton A | 0.97 | 1.30 | 40 | 200 | 5 | 2.6 | 5 |
| VAAR | 1.22 | 1.62 | 50 | 160 | 9.5 | 2.7 | 0 |
| Anstac M | 1.19 | 1.72 | 11 | 8 | 11 | 2.6 | 0 |
| Fluorolube | 1.92 | 1.50 | 7 | 10 | 8.5 | 2.5 | 8 |
| Isostearic acid ^b | 1.00 | 1.62 | 7 | 500 | 8.7 | 1.7 | 0 |
| Carbon black ^c | 1.30 | 1.68 | 21 | 8 | 11 | 2.5 ^f | 0 |
| Calcium resinate | 1.29 | 1.66 | 16 | -- | -- | 2.4 | 2 |
| TWEEN 80 | 0.86 | 1.36 | 7 | 8 | -- | -- | -- |
| CAB-O-SIL | 1.19 | 1.57 | 21 | 20 | -- | 1.0 | 2 |
| Sulfur | 1.37 | 1.75 | 19 | 20 | -- | 0.9 | 8 |
| DOW-Corning XZ-6-1082 | 1.28 | 1.62 | 17 | 10 | -- | 2.1 | 2 |
| None ^e | 1.03 | 1.63 | 6 | 40 | -- | 0.5 | 10 |

^aCoatants employed at 1% level.

^bPre-coated aluminum used. Compositions blended dry on ball mill.

^cCabot Corp, Regal SR

^dRelative units. For flow, the higher the value, the better the flow.

^eAvg. particle diameter of aluminum used was 8 μ , instead of the nominal 20 μ specification material.

^fValue obtained with needle to sample air gap of 250 mils; the high conductivity of the composition prevented a voltage buildup when the needle was in contact with the sample.

Table 2. Light output characteristics of formulations

| Coatant ^a | Performance in Poppy Cartridge | |
|------------------------------|--------------------------------|--|
| | Charge wt., g | Integral, light, avg. 10 ⁵ candlesec |
| None | 18 | 1.71 |
| Dow 200 | 16 | 1.42 |
| Viton A | 16 | 1.36 |
| VAAR | 18 | 1.58 |
| Anstac M | 18 | 1.42 |
| Fluorolube | 16 | 1.47 |
| Isostearic acid ^b | 17 | 1.42 |
| Carbon black ^c | 21 | 1.84 |

^aCoatants employed at 1% level.

^bPre-coated aluminum used. Compositions blended dry on ball mill.

^cCabot Corp., Regal SR.

Table 3. Light output from formulations^{a, b}

| Coatant | Charges wt., g | Integral light 10 ⁵ candlesec | Std. dev. |
|--------------------------------|-------------------|---|-----------|
| None | 20 | 1.88 | 0.29 |
| Vinyl alcohol acetate resin | 18 | 1.40 | 0.13 |
| Carbon black, Sterling R | 19 | 1.84 | 0.12 |
| Carbon black, Regal SR-F-S | 21 | 1.84 | 0.27 |

^aCoatant at 1% level.

^bLoaded in Poppy cartridges, twelve rounds per set.

Table 4. Storage surveillance of formulations^a

| Storage conditions | Item | Storage intervals, months | | | | | |
|--------------------|----------------------|---|--------------|--------------|--------------|--------------|--------------|
| | | 0 | 1 | 3 | 6 | 12 | |
| Room Temp. | Control ^b | Integral Light 10 ⁵ Candles Standard Dev. | 1.87 0.14 | 1.91 0.14 | 1.79 0.19 | 2.07 0.21 | 2.15 0.13 |
| | Coated ^c | Integral Light 10 ⁵ Candles Standard Dev. | 1.87 0.15 | 1.93 0.12 | 1.31 0.14 | 2.00 0.16 | 2.09 0.18 |
| 160°F (71°C) | Control ^b | Integral Light 10 ⁵ Candles Standard Dev. | 1.87 0.14 | 1.88 0.16 | 1.83 0.20 | 2.11 0.21 | 2.13 0.22 |
| | Coated ^c | Integral Light 10 ⁵ Candles Standard Dev. | 1.87 0.15 | 1.86 0.18 | 1.79 0.13 | 2.09 0.19 | 2.10 0.18 |

^aLoaded in Poppy photoflash cartridges. Twenty rounds per set prior to storage. Ten rounds per set at each storage interval.

^bType III, Class A photoflash powder.

^cType III, Class A photoflash powder with 1% Carbon Black, Cabot, Type Regal SRF-S coatant.

APPENDIX A
THE THERMAL COMPARATOR METHOD FOR THE MEASUREMENT OF THE THERMAL
CONDUCTIVITY OF PYROTECHNIC MIXTURES

BY
A.J. BEARDELL

The thermal conductivity of the desensitized photoflash composition has been suggested as a method for quickly monitorizing the coatant content of the mixture. The instrument used for this purpose is called a thermal comparator, since the thermal conductivity on the test sample is obtained from a calibration curve derived from reference standards.

The instrument registers the rate of cooling experience by a heated probe upon contact with the surface of the test material. The probe assembly, (fig. A-1) consists of a thermocouple sensing tip, heater, and thermal reservoir, held at temperature T_1 above the room temperature T_2 . Upon contact of the probe tip of thermal conductivity λ_1 , with the surface of the material at room temperature and having thermal conductivity λ_2 , the tip immediately drops to an intermediate temperature T_c , which registers either digitally or on a strip chart recorder as an emf. A plateau is reached that represents a near steady state temperature level and it is this level that relates to the thermal conductivity of the material.

The relationship of the thermal conductivity and the emf of the probe is acquired using reference standards, materials of known thermal conductivity. A typical calibration curve is shown in figure A-2.

A typical procedure is as follows: The photoflash composition is lightly pressed into a pellet at about 2000 psi loading pressure, a recommended pellet size is 0.5 inch in diameter and 0.25 inch thick. The instrument is allowed to warm-up for about 30 minutes and set so that the probe temperature is about 15°C above room temperature. The pellet is then placed over the probe well and the probe is allowed to contact the sample. The emf will quickly reach an equilibrium value which is recorded. Since the probe tip touches the sample in only a very small area ($\sim 0.1 \text{ in}^2$), it is recommended that at least five readings be taken at different points on the sample. In addition, measurements should be made on at least three samples of the same material. The whole operation for a given mixture should require no longer than 15-20 minutes. The measured emf is then used to obtain the thermal conductivity from the calibration curve.

Runs were performed on a photoflash composition in which the carbon content was varied between 0 and 1.5% - the results are summarized below:

| <u>Photoflash designation</u> | <u>Carbon content of photoflash mixture (%)</u> | <u>λ W cm⁻¹, C⁻¹</u> |
|-------------------------------|---|---|
| 555 M | 0 | 0.0051 |
| 555 B | 0 | 0.0053 |
| 161 M | 0.5 | 0.0048 |
| 59 M | 1.0 | 0.0035 |
| 62 M | 1.5 | 0.0026 |

It is noted that there is about a 10% drop in λ when 0.5% carbon is added to the mixture. The value decreases 35% at a 1% carbon level and by 50% at the 1.5% carbon level. Thus, the carbon concentration appears to have a pronounced effect on λ . However, the absolute value of λ is in all cases extremely low, which is normal for loose or near loose powders.

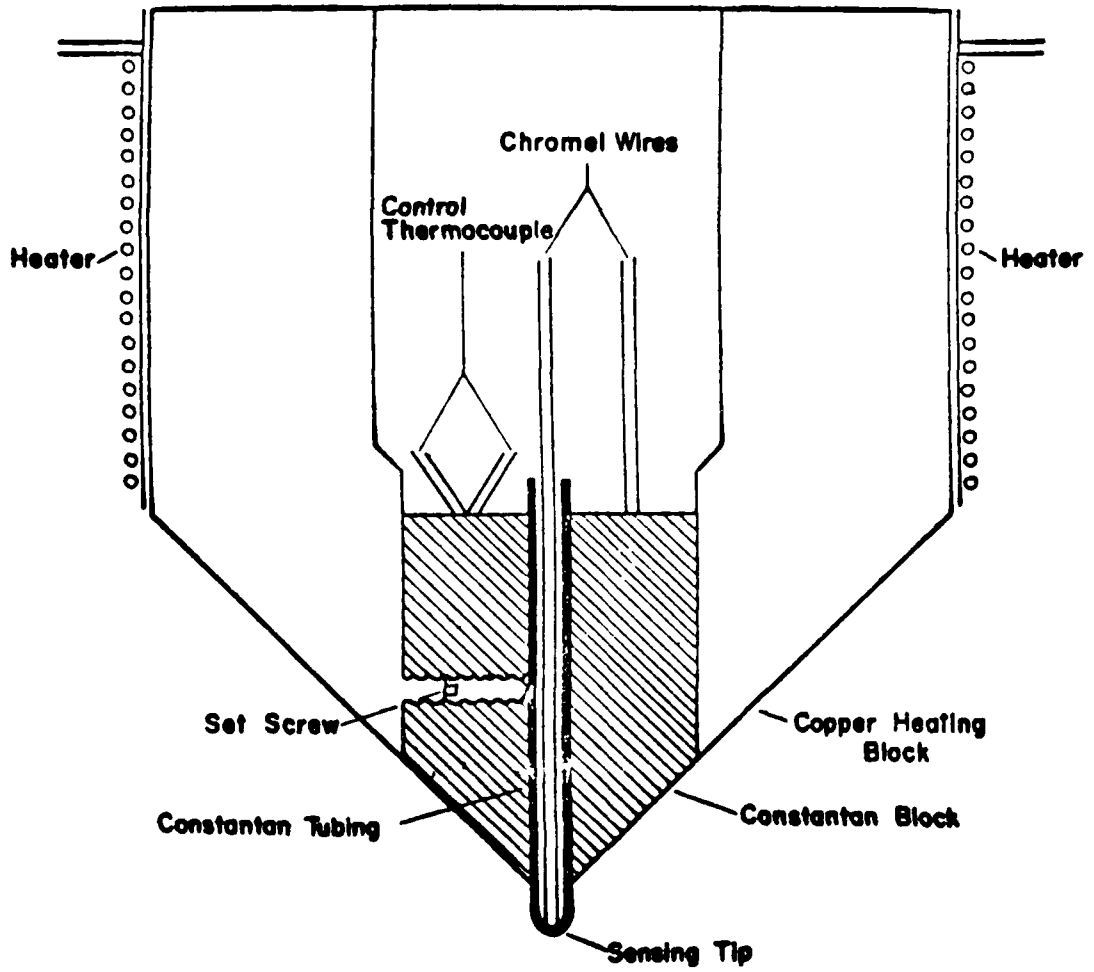


Figure A-1. Schematic of probe assembly

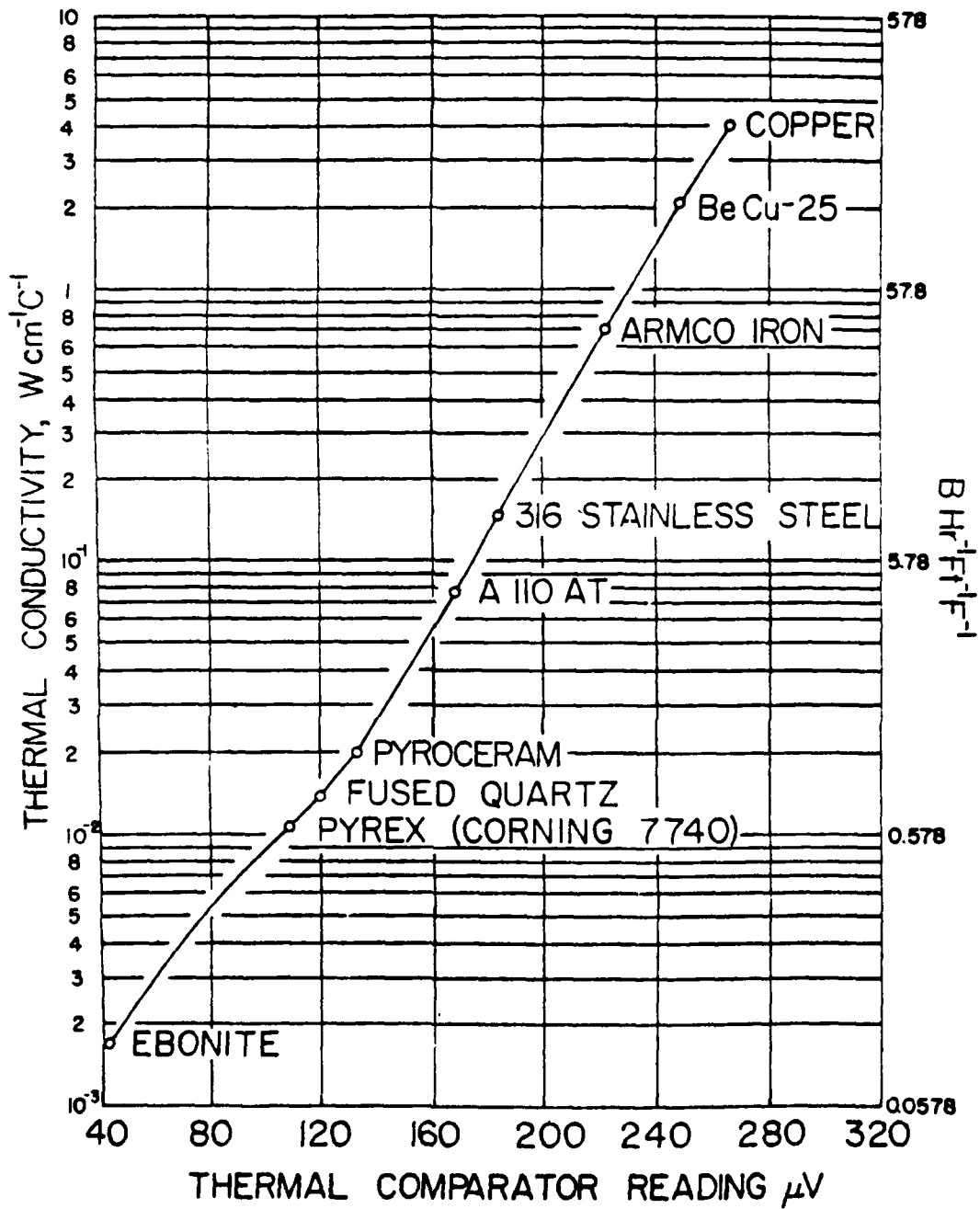


Figure A-2. Typical thermal comparator reading, emf in μV

APPENDIX B
BLENDING PROCEDURE

The following procedure is used for a 1 kilogram batch of carbon black coated photoflash composition prepared in a counter current mix muller:

Weigh out the potassium perchlorate and barium nitrate separately, place in the bowl of blender and spread out evenly with a non-sparking conductive spatula. Measure out about 250 mL of absolute alcohol and dampen the powder with a small quantity of the alcohol. Weigh out the carbon black and add to the bowl. Finally weigh out the aluminum powder and add. Lower the wheel and blade assembly into the bowl, add about half of the alcohol and allow mixer to run for about three to five minutes. With the spatula, gently scrape the ingredients from the sides of the bowl, the blade, and the wheel, meanwhile adding the remainder of the alcohol in the area being scraped. Run the mixer 1 to 2 minutes, check the sides, blade, and wheel for any powder buildup, and if necessary gently scrape down again and continue running the mixer for about 5 minutes. After stopping the mixer, allow about a minute to elapse, then enter the mixing room. With spatula, transfer the composition to a flat pan and allow to dry.

LIST OF MATERIALS

Potassium perchlorate-MIL-P-217A, 20 μ
Barium nitrate-MIL-162D, Class 2
Aluminum, ALCAN, Lot 388, MIL-A-23950A, 18 μ
Aluminum, ALCOA, coated with 2% stearic acid 16 μ
Aluminum, ALCOA, 8 μ
Sulfur-MIL-S-487B
Calcium Resinate MIL-C-20470A
Vinyl Alcohol Acetate Resin-MA28-18, Union Carbide Corp.
Viton A, Fluorocarbon resin, E.I. DuPont deNemours
Silicone, Fluid, 200, 100,000 CS Viscosity, Dow Corning Co.,
Midland, MI
Anstac M-Chemical Development Corp., Danvers, MA
Fluorolube, GRADE 230, Hooker Chemical Co.
XZ-6-1082, Dow Corning Co., Midland, MI
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