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LOS ALAMOS SCIENTIFIC LABORATORY
OF THE UNIVERSITY OF CALIFORNIA ○ LOS ALAMOS NEW MEXICO

FORMULATION OF A LOW STATIC ACCUMULATING, RESILIENT VINYL COATING FOR EXPLOSIVE HANDLING EQUIPMENT

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LAMS-2798
UC-38, ENGINEERING AND
EQUIPMENT
TID-4500 (18th Ed.)

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REPORT WRITTEN: August 17, 1962

REPORT DISTRIBUTED: February 25, 1963

FORMULATION OF A LOW STATIC ACCUMULATING, RESILIENT VINYL COATING FOR EXPLOSIVE HANDLING EQUIPMENT

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Contract W-7405-ENG. 36 with the U. S. Atomic Energy Commission

ABSTRACT

A material system has been developed for use in coating machine tool equipment components to aid in preventing accidental detonation of high explosive materials during machining operations. A graphite-filled, plasticized polyvinyl chloride composition produces a resilient coating with capability to leak-off or dissipate charges of static electricity. The coating is tough, abrasion resistant, and capable of being repaired if torn or otherwise damaged in use.

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CHAPTER 1

OBJECTIVES

In machining high explosive (HE) shapes, accidental detonation of the material either by impact or the arcing of accumulated static electricity is a distinct possibility. Therefore, the development of a materials system to aid in the prevention of such accidents, specifically during the machining operation, was required.

1.1 The Problem

The tools used to machine HE materials at The Los Alamos Scientific Laboratory (LASL) are equipped with "wet pans" to catch and drain off the coolant fluid and the "chips" formed during the machining operation. These pans have historically been made of a non-sparking metal such as brass or aluminum. Although metallic pans prevent any accumulation of static charges, they do not provide protection against mechanical damage to the part if it happens to fall from the machine chuck, nor do they provide protection against the impact of HE with a hard object. Any material used in construction of a wet pan must (1) be resistant to corrosion or chemical reaction with the HE or coolant; (2) survive steam cleaning operations; and (3) be amenable to fabrication into one-of-a-kind, tailored units.

1.2 Potential Solutions

The initial approach to the problems of impact (resulting in either detonation or mechanical damage) and the accumulation of static electrical charges was to fabricate the pans by hot gas welding of rigid polyvinyl chloride (PVC) or polyethylene containing or coated with anti-static additives. At the time, anti-static treatments for PVC and polyethylene were not reliable. A second option was to fabricate the wet pans from welded aluminum sheet and subsequently apply a locally formulated, anti-static PVC plastisol or polyethylene coating.

1.3 Basic Specifications for a Coating

A satisfactory coating, as applied, should have good impact resistance and impact energy absorption properties, and also have relatively good electrical conductivity. It should bond well to the substrate material with no pockets or voids to allow the accumulation of HE particles. Chemical reaction with HE cannot be tolerated, and yet the material must be comparatively easy to repair in the event of accidental damage to the coating.

CHAPTER 2

EXPERIMENTAL COATINGS

A number of coating materials evaluated early in the program included neoprene sheeting, polyethylene sheeting, and several PVC plastisol formulations.

Neoprene sheeting (45-55 Shore A_2 durometer) was glued in place on a primed dural substrate. Gates Engineering Co., Western Div., Los Angeles 22, California, N-11 primer, and N-29/N-39 "liquid neoprene" adhesive were used for this operation. Anti-static properties were poor, and only a few pans were fabricated by this procedure for use where the impact problem was considered severe.

Polyethylene sheeting was cut, formed, and hot gas (N_2) welded as a self-supporting structure. A pan made for a milling machine by this process is shown in Figure 1. No more than six pans were made from polyethylene for specialized requirements while attention was devoted to development of a "conductive" plastisol coating.

The PVC plastisol samples were all dip coated over a primed dural or brass substrate. The primer used throughout the program

Figure 1

Welded Polyethylene Wet Pan

was "Unichrome 219 PX", a product of The United Chromium Div., Metal and Thermit Corp., New York 17, New York. All test specimens were prepared by a loosely standardized process: (a) oven preheat specimen plate 40 minutes at 175-180°C; (b) dip plate into test material with 40 seconds dwell allowed; (c) remove and drain until material flow ceased; (d) oven fuse 25 minutes at 175-180°C or until full strength of coating is obtained; (e) trim off any "drip beads" prior to testing. If test specimens were to be primed, the primer was applied either by brush or spray and allowed to air dry prior to the preheat. (Since the test program has been completed, it has been found that a maximum preheat time of 20 minutes at 175-180°C will produce maximum adhesion between substrate and coating. Newer procedures take this fact into account.) If only one side of the test specimen was to remain coated, excess material was trimmed off with a scalpel.

2.1 Composition of Experimental PVC Plastisol Coatings

All of the experimental coatings were derived from the following basic PVC plastisol.

TABLE I

Basic PVC Plastisol

Ingredient	Parts by weight
1. Geon 121	100
2. Flexol DOP	80
3. Flexol 4GO	20
4. Paraplex G-60	10
5. Barinac	4
6. Bentone #34	1.5
7. DCMRF	0.5

2.1.1 Material Sources and Identification

1. Geon 121 is a high molecular weight vinyl chloride polymer of minus 200 mesh size especially developed for use as a plastisol base. The manufacturer is B. F. Goodrich Chemical Co., Cleveland 15, Ohio.

2. Flexol DOP is a primary plasticizer manufactured by Carbide and Carbon Chemical Co., Division of Union Carbide and Carbon Corp., New York 17, New York. Chemically it is di(2-ethylhexyl) phthalate.

3. Flexol 4GO is another Carbide and Carbon product included here to increase low temperature flexibility, reduce compound viscosity, and control increase in viscosity with age. The chemical identification is polyethylene glycol di(2-ethyl hexoate).

4. Paraplex G-60 is a polyester type plasticizer which serves to increase hardness and abrasion resistance of the fused compound. It also acts as a light and heat stabilizer to some extent. The Resinous Products Division of Rohm and Hass Co., Philadelphia 5, Pennsylvania. is the source for this material.

5. Barinac (barium ricinoleate) is the primary system stabilizer. It is supplied by the National Lead Co., New York 6, New York.

6. Bentone 34 is used as a flow control agent and is an orgonophillic bentonite manufactured by the National Lead Co.

7. DCMRF is a silicone mold release fluid obtained from Dow Corning Corp., Midland, Michigan, and is used as an internal lubricant. DC-200, 350 centistokes viscosity is equally acceptable.

2.1.2 Formula Variations

To this basic plastisol were added varying amounts of graphite (Dixon Flake Graphite #635, Joseph Dixon Crucible Co., Jersey City, New Jersey), and plasticizers such as Flexol DOP or DIDP (diisodecyl phthalate obtained from Monsanto Chemical Co., Organic Chemicals Div., St. Louis, Missouri, and used as a partial viscosity control agent and primary plasticizer). Graphite loadings of 10, 20, and 30 w/o based on the original plastisol were made, producing mixtures which retained dip-coating capabilities. In order to obtain higher graphite concentrations, it was necessary to extend the "basic" plastisol with additional plasticizer. The increase in plasticizer was varied from 10-50 w/o of the original plastisol with the Flexol DOP, and from 10-60 w/o with the DIDP. The 50% extension of the plastisol with Flexol DOP allowed a maximum workable graphite concentration of 37.5 w/o; and the 60% extension with DIDP allowed a workable maximum concentration of 38.3 w/o graphite. The 60% DIDP extended, 38% graphite-filled composition is rather viscous and has a tendency to retain bubbles. (A single pass through a 3 roll paint mill will reduce this tendency considerably.) All samples used in the collection of data were mixed by hand using only a spatula and beaker to mix in the additives. The "basic" plastisol was prepared in a Hobart dough mixer by first blending the liquid ingredients and then sifting the dry powders slowly into the mixing bowl to prevent their agglomeration.

2.2 Test Data

Electrical resistance tests were made on a series of different materials including neoprene, PVC plastisol and four PVC plastisols containing graphite or "Thermax" carbon black. (Thermatomic Carbon Corporation, Sterlington, Louisiana.)

TABLE II

Resistance of Materials

Material	Resistance (Megohms)
Neoprene (glued to dural)	5000*
"Basic" PVC Plastisol, no additives	8.4
+0.5% Carbon Black	11-12
+10% Graphite	7.6
+20% Graphite	5.4
+30% Graphite	2.1
+60% DIDP +38.3% Graphite	0.1

*Measured on a DC Bridge in air.

Because the prospective use of the coated pans was to be with water base coolants, the resistance measurements were made with tap water as one electrical contact. A 60 cycle alternating current was used to avoid polarization. The samples tested were 3 in. x 3 in. x 0.25 in. brass or dural plates completely coated to within 0.5 in. of one 3 in. x 0.25 in. face. Coating thickness applied to the test plates varied somewhat because of variations in the physical properties (primarily viscosity, and thermal conductivity) of the different compositions. All test plates were prepared by the standard process which has been described, without modification to control the deposit thickness. The higher filler content mixtures produced the greater thicknesses. Individual sample thickness varied a maximum of ± 0.030 in. from an average of 0.080 in. for the series.

The samples were immersed 1.5 in. into water contained in a 4.0 in. dia brass can. Electrical connections were made to the can and to the sample plate. The applied voltage was measured on a 1/4% Dynamometer. The current was measured by observing the voltage drop across a 5000 ohm resistor with an oscilloscope and a differential amplifier. Accuracy of the current measurement was about 3%. Overall accuracy was about 5%. All measurements were made by the LASL Electrical Standards Laboratory. Figure 2 is a block diagram of the test apparatus.

The resistance of the graphite filled samples appears to be linear with respect to filler concentration. The data are plotted as Figure 3. Resistance characteristics of 30% graphite filled coating is voltage dependent as shown in Figure 4.

2.2.1 Peel Strength of Coatings Applied to Primed Metal Substrates

A series of tests was made on primed brass and dural substrates in both wet and dry conditions to determine adhesion of the coating to the substrate. The tests were made by LASL Group GMX-3 and indicate the peel strength of these coatings. Values reported are averages for 3 samples of each type tested at 22°C.

BLOCK DIAGRAM

RESISTANCE TEST APPARATUS

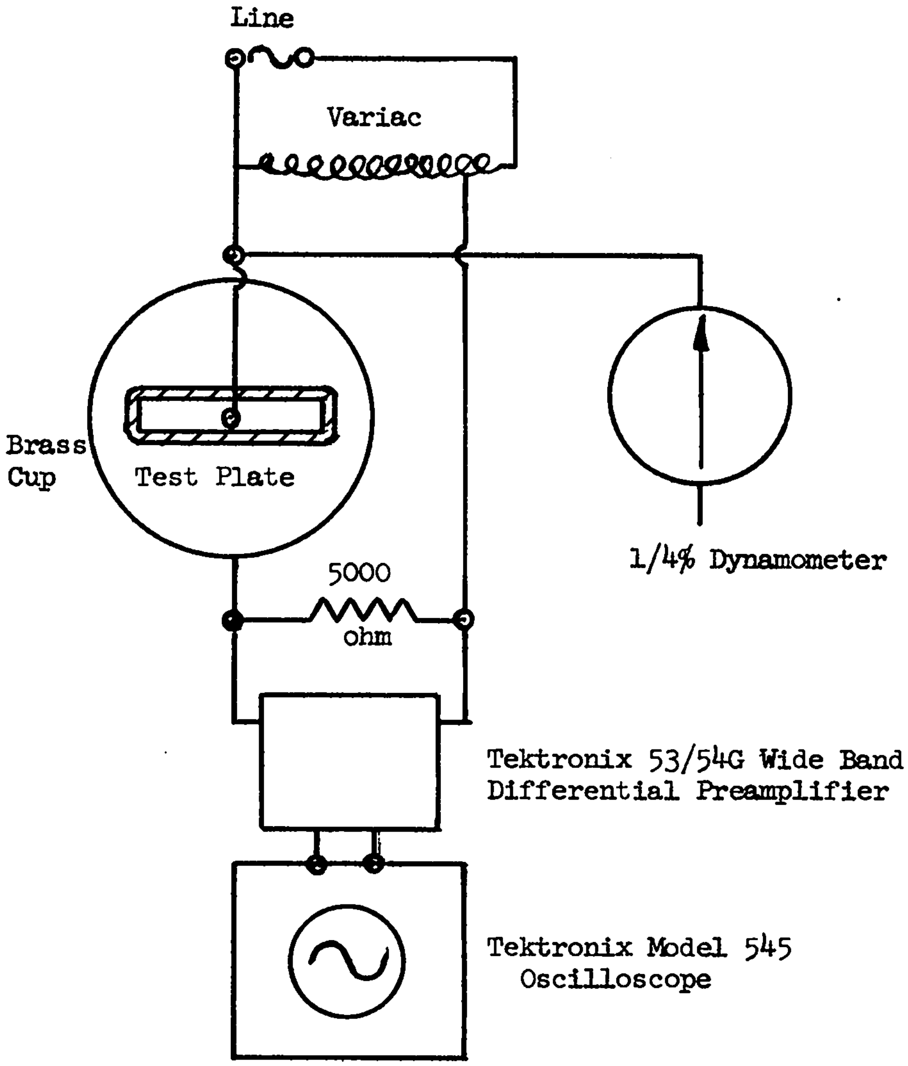

FIGURE 2

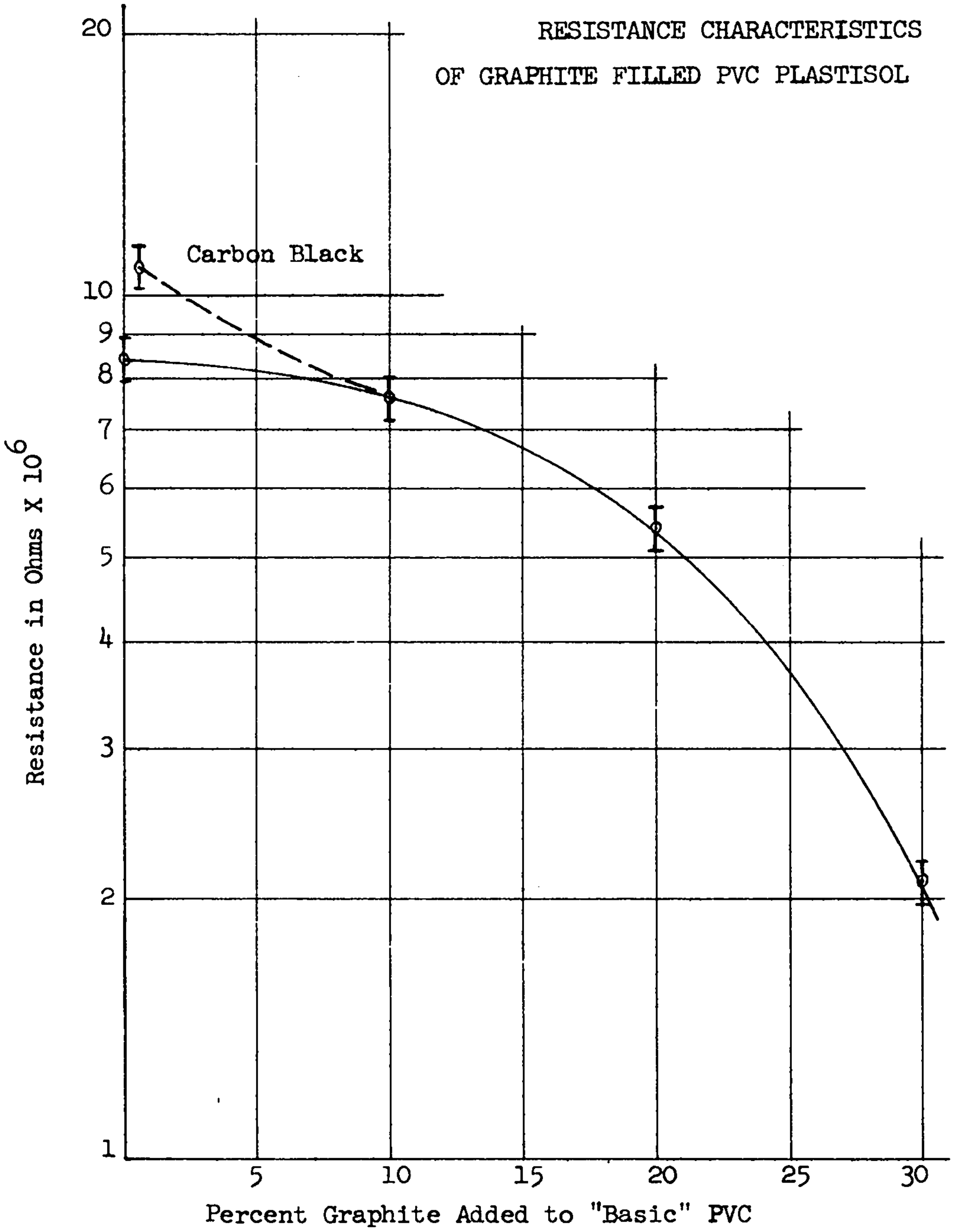
RESISTANCE CHARACTERISTICS
OF GRAPHITE FILLED PVC PLASTISOL
Carbon Black
Resistance in Ohms X 10^6
20
10
9
8
7
6
5
4
3
2
1
5
10
15
20
25
30
Percent Graphite Added to "Basic" PVC

FIGURE 3

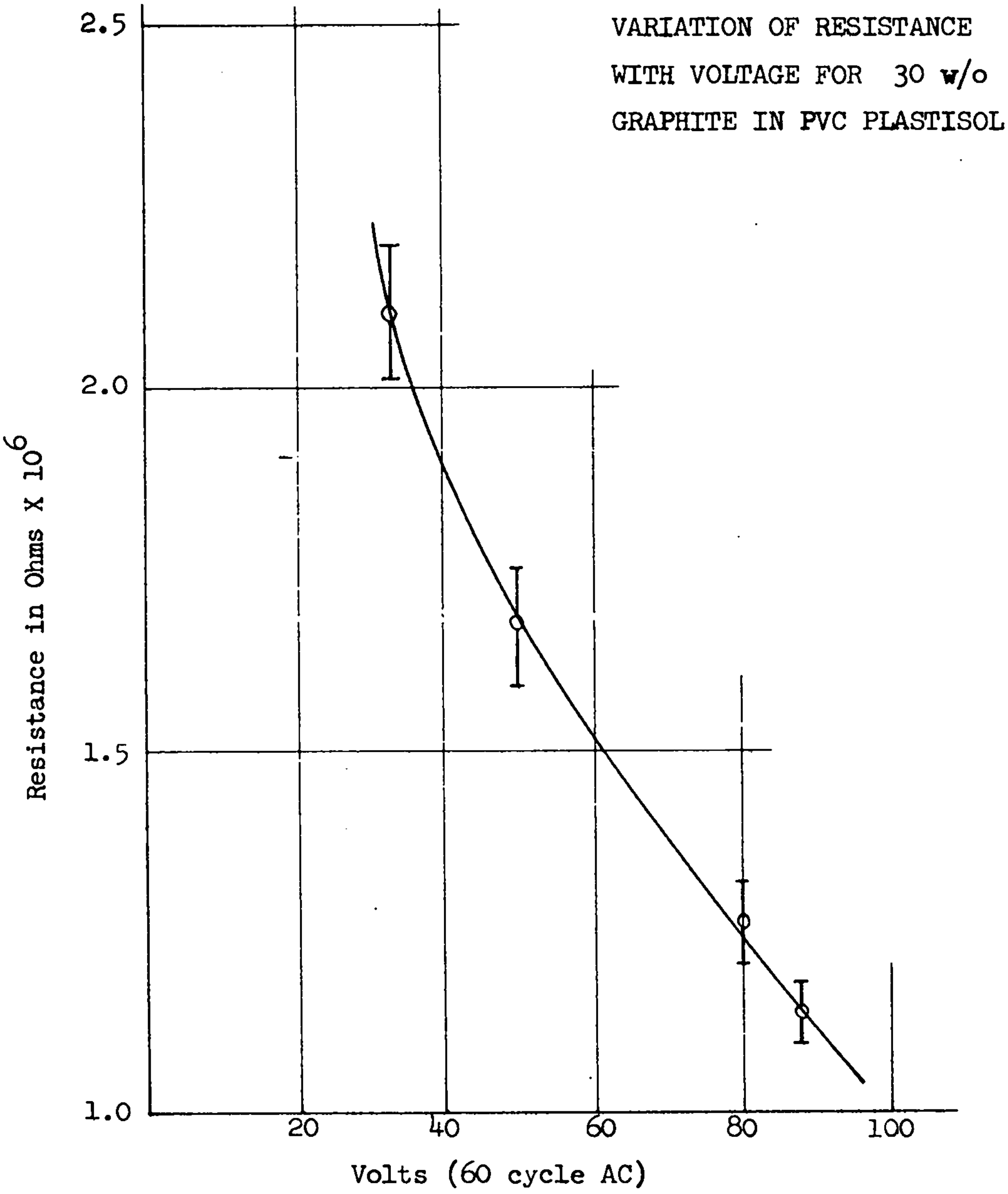
VARIATION OF RESISTANCE
WITH VOLTAGE FOR 30 w/o
GRAPHITE IN PVC PLASTISOL
2.5
2.0
1.5
1.0
Resistance in Ohms X 10^6
20
40
60
80
100
Volts (60 cycle AC)

FIGURE 4

TABLE III

Peel Strength of Coatings

Substrate	Condition	Peel Strength (lbs/in. of width)
Brass	Dry	16.1
Brass	Wet*	17.6
Dural	Dry	5.8
Dural	Wet*	2.5

*Specimens conditioned in water for 24 hours prior to test.

Peel test specimens were bars coated on one side only with the "basic" PVC plastisol +0.5% carbon black. ASTM-D903-49 procedure was followed.

CHAPTER 3

STANDARD PROCESS FOR COATING WET PANS

Although two different types of coatings are presently applied to wet pans at LASL, the basic process is the same. The part is primed, air dried, and then suspended in a manner so as to hold inside surfaces upward, but tilted slightly. Suspension is normally done by attaching an improvised combination of cables and bars to the pan mounting lugs and arranging the resultant cradle linkages to achieve correct pan position. Adjustments must be made individually for each type or style of pan to produce smooth coverage on critical surfaces. Standard preheat is 20 minutes at 175-180°C in a mechanical convection oven. The part is immersed in the plastisol for 30 to 50 seconds, removed, drained, and returned to the oven for 20 to 35 minutes at 175-180°C to fuse the coating. Complete fusion is considered to have occurred when the coating surface acquires a polished sheen. Fusion time required is dependent on both mass and geometry (as it affects drain time).

Coating thickness can be increased by repeating the dip and fusion stages of the process. After the final fusion cycle, the coated article is cooled and trimmed as required. (Cooling may be by immersion in water or by air convection.) If the coating on the pan exterior does not interfere with mounting or machining operation, it is left in place; otherwise, it is knife cut, and removed. Surfaces which are to be left free of coating are not primed in order to permit easy removal of the surplus coating.

Figures 5 and 6 are photographs of typical coated wet pans.

3.1 Coating Formulations

Two types of filled PVC plastisol formulations are currently utilized at LASL in production coating of wet pans. The formulation in Table IV is employed where conductivity is required. The formulation for resilient, non-conducting PVC plastisol is listed in Table V. (All of the ingredients have previously been identified as to primary function and source.)

TABLE IV

Formulation for Conducting PVC Plastisol

Ingredient	Parts by Weight
Geon 121	100
Flexol DOP	80
Flexol 4GO	20
DIDP	130
Paraplex G-60	10
Barinac	4
Bentone #34	1.5
DCMRF	0.5
Dixon Flake Graphite #635	215

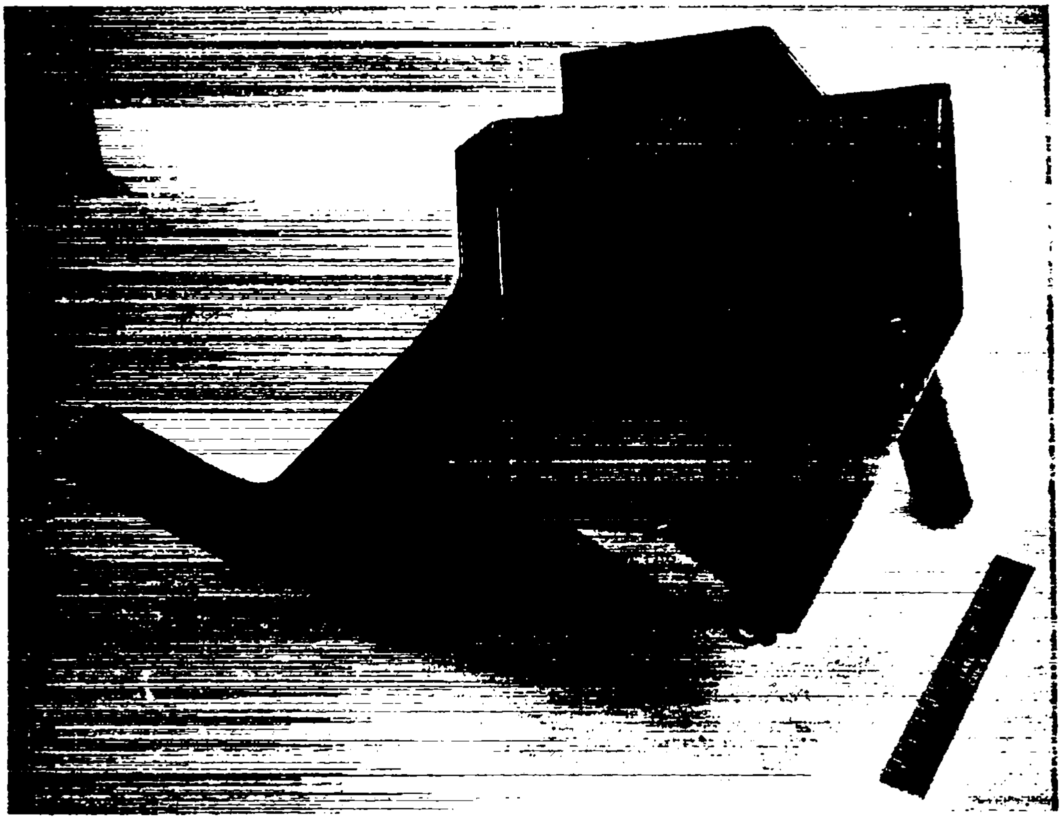

Figure 5

PVC Coated Lathe Pan

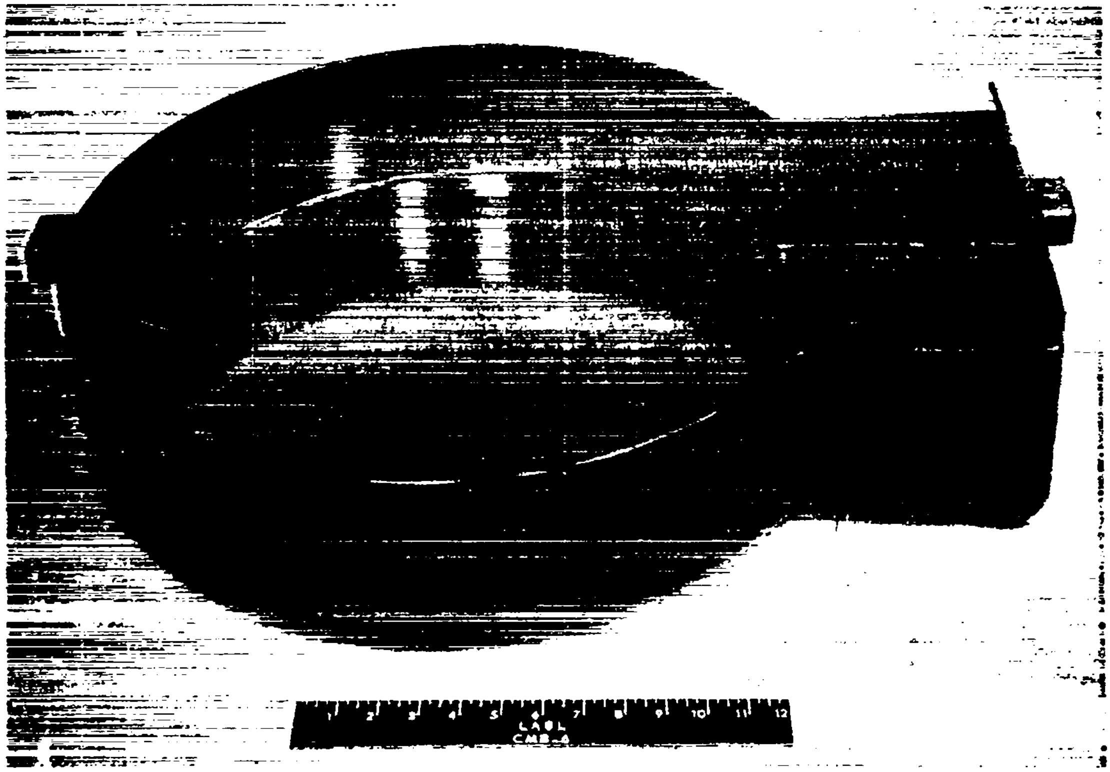

Figure 6

PVC Coated Mill Pan

TABLE V

Formulation for Resilient, Non-Conducting PVC Plastisol

Ingredient	Parts by Weight
Geon 121	100
Flexol DOP	60
DIDP	20
Flexol 4GO	20
Paraplex G-60	10
Barinac	4
Bentone #34	1.5
DCMRF	0.5
Carbon Black	0.5

Since the Ordinance Safety Manual specification requiring conducting coatings with resistivity less than 1 megohm/in. has been deleted from the LASL coating standards, the formulation listed in Table V has been the more popular coating material.

3.2 Repair of Coatings

Coatings which are damaged in use can be repaired in the field, although defective parts are normally stripped and recoated. Stripping a coating from a primed substrate is difficult and usually achieved only by mechanical peeling using a putty knife. More often, a defective coating is carbonized by thermal abuse in an oxidizing atmosphere, and the debris scraped away.

Preparation for field repairs can be made by knife cutting around the flawed area, peeling the coating away within the cut, sanding the metal, and applying primer. Much care has to be exercised if there is any probability of encountering HE inclusions trapped under a flap of coating, and steam cleaning is normally required. After the primer has been air dried, liquid plastisol is placed in the cut-out area, and fused in place with a heated iron or exposed to a jet of hot N_2 gas. A patch can best be blended and bonded to the surrounding sound coating by use of a heated iron or tool shaped to the coated surface contour.

Some pans have been in regular use for 4 years with no sign of wear or coating failure.

3.3 Other Applications of the Coating System

Work done with the coating formulations described has been by no means limited to wet pans, although they constitute a major fraction of applications. Other machine tool components which can come in contact with HE have been coated either selectively or over all of their exposed surfaces. Lifting fixtures have been padded; table tops, cooling grills, riser carts, mold carts, and other assorted hardware have been coated. Minor adjustments in the plastisol formulations have occasionally been made to achieve specialized properties. Most frequently, the Bentone 34 content has been increased to cause pick-up of a thicker coating on a single dip. One special modification, the addition of monomeric diallyl phthalate and a peroxide catalyst, has been utilized to make a large gain in toughness and impact resistance. This specially modified system has a short life in storage, as well as other disadvantages such as decreased resiliency.

3.4 Storage and Age Effects

Storage life at ambient room temperatures of the formulations listed in Tables IV and V is the order of several months. Stirring with a "Lightnin" type propeller mixer is advisable if the plastisol has not been disturbed for 10 to 15 days. If the tank of material has received severe thermal abuse, the contents should be strained through a 6-8 ply bag of cheese cloth to remove gelled agglomerates, stirred, and allowed to stand 12 to 20 hours to rid itself of air bubbles which affect coating quality. Aged material will develop a tendency to produce slightly heavier coatings than freshly prepared plastisol. This factor has proven to be no real problem, and a slight reduction in part preheat time can be made to compensate for age if required.

It is normal LASL practice to hold a 400-500 gallon tank of the composition given in Table V on hand. Fresh material is added as required after "used" material has been strained.

CHAPTER 4

CONCLUSION

A process has been developed to deposit and bond conductive, impact resistant and shock absorbing PVC base coatings to metallic substrates. These coatings are especially valuable when exposure to HE in massive or chip form is anticipated. There are modifications which might be made to improve conductivity, ease of application, or both; however, development work on the program was stopped as soon as useful formulations had been devised to meet minimum specifications. Potential usefulness of this material extends to coating machine components other than wet pans, which may be involved in explosives fabrication operations.