Adhesives file

Technical Memos from Mound Laboratory

MONSANTO RESEARCH CORPORATION, MIAMISBURG, OHIO

CONDUCTIVE ADHESIVES

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Mound Laboratory is a recognized leader in many fields relating to atomic energy, such as calorimetry, reactor fuels research, and isotope separation using thermal diffusion columns. However, there are many other programs at Mound Laboratory which are important, but which have not received the publicity that these more glamorous applications have received.

Typical of these programs is the work of the organic chemistry group of the Research Department, who are responsible for developing plastics and adhesives for specialized applications in the atomic energy program. Unlike industrial organizations, this small group does not attempt to develop new plastics and adhesives, but tries to utilize the available technology for unique applications.

Among the more recent developments coming from this group were conductive adhesives flexible at low temperatures. Although a number of highly conductive adhesives are commercially available and are widely used in industry, most of these materials are inflexible, especially at –65°F. These materials are silver-filled, amine-cured epoxy formulations with resistivities as low as 0.001 ohm-cm.

When these commercial adhesives are used to join materials having different coefficients of linear expansion, they fail when the parts are subjected to thermal cycling. For these situations a flexible adhesive is necessary.

Polyurethane, silicone and polysulfide-based conductive adhesive formulation were investigated in order to develop an adhesive with the following properties:

Maximum electrical resistivity	0.1 ohm-cm
Minimum ultimate elongation at –65°F (film)	10%
Minimum tensile strength (plug) at room temperature	500 psi
Minimum peel strength (ASTM D1676-61T)	20 lbs. per inch.

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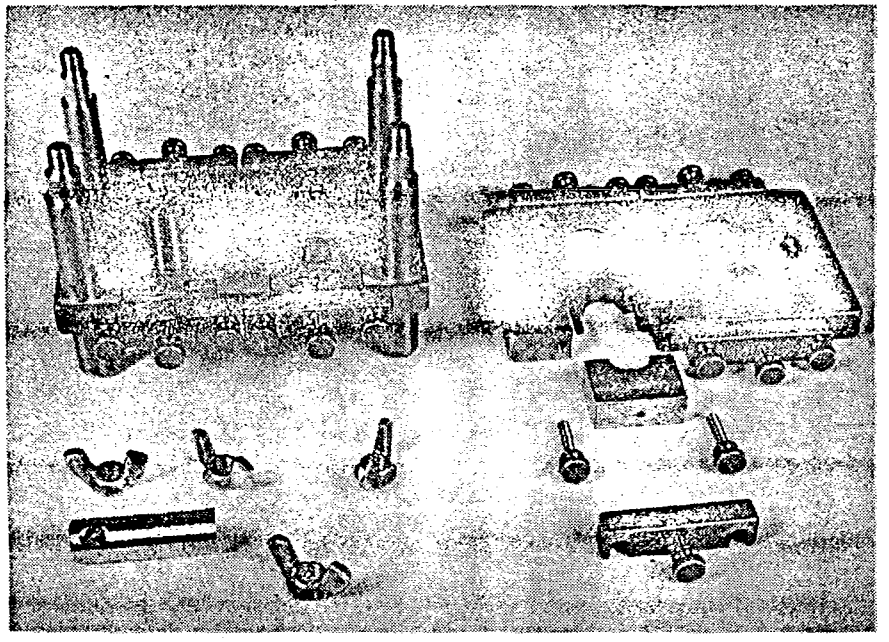
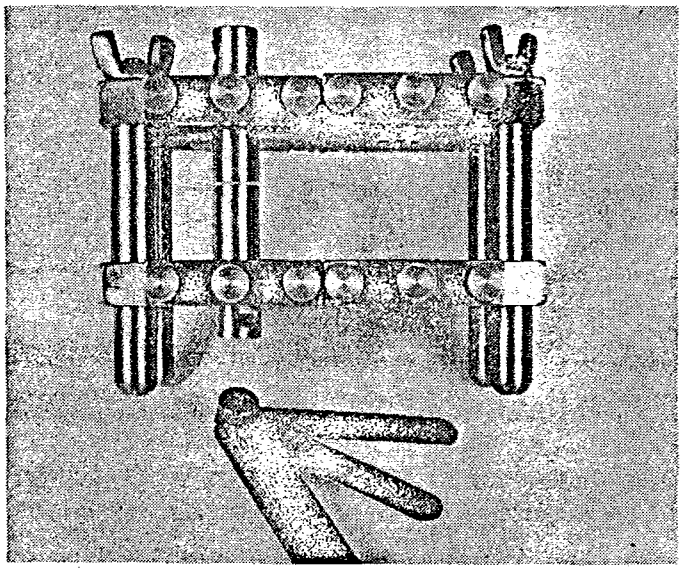


Special jig for bonding conductive adhesives, shown disassembled. One of the brass sample plugs is in the jig.



The special jig is shown assembled with the brass plugs bonded end to end with conductive adhesive. The cylindrical brass plugs have a cross-sectional area of approximately 1/4 square inch and are bonded end to end with the conductive adhesive. Prior to bonding, the brass plugs were degreased with methyl ethyl ketone, dipped for three minutes in a mixture of 55% (by volume) phosphoric, 25% nitric, and 20% glacial acetic acid, subsequently rinsed with distilled water and dried. The bond thickness was mechanically set at 0.010 inch. After cure, the resistance of the bond was determined with a Kelvin bridge using a current of not more than 60 milliamperes. The strength of the bond at room temperature was determined by pulling the plugs apart using an Instron Testing machine at a cross-head speed of 0.02 inch per minute.

Figure 1

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Polyurethane Based Adhesives

Polyurethane-based formulations were first investigated. It was found that when silver flake was used as the filler in a room temperature curing formulation, the specific resistivity of the adhesive was higher than 100 ohm-cm. Nickel powder-filled formulations also had high resistance, but those formulations containing conductive carbon blacks had electrical resistivities as low as 2 ohm-cm.

By combining carbon black and silver powder in polyurethane formulations, much lower resistivities were obtained. These flexible formulations were within the desired resistivity range.

The most successful of the polyurethane based conductive adhesives contained the following ingredients:

Component	Parts by Weight
Adiprene L-167[a] prepolymer	15.2
Butyl glycidyl ether	3.8
Continex CF[b] carbon black	7.0
J2 Silver Powder[c]	71.8
4,4'-Methylene bis-(2-chloroaniline)[a]	2.2

[a]E. I. DuPont de Nemours, Inc.

[b]Witco Chemical Corp.

[c]Engelhard Industries

Immediately after this formulation was cured at 110°F for 16 hours, its electrical resistivity was 0.5 ohm-cm. When the formulation was aged at 160°F for two weeks, the resistivity dropped to 0.13 ohm-cm. The plug tensile strength was 600 lbs. per sq. in., the peel strength was 15 lbs. per in. and the ultimate elongation was 9%. Thus, the properties of this formulation closely approached the desired specifications. The apparatus for bonding the tensile test specimen is shown in Figure 1.

Silicone-Based Adhesives

Silicone based conductive adhesives are not commercially available; however, an experimental silicone formulation filled with carbon black was obtained from one manufacturer. This formulation had an electrical resistivity of over 1000 ohm-cm after curing 24 hours at room temperature and liberated acetic acid while the formulation was curing.

A number of silicone-based adhesive formulations were developed, but all were unsatisfactory. The formulations were extremely flexible at -65°F, (40% ultimate elongation) but the plug tensile strength and the peel strength were unsatisfactorily low.

Polysulfide Adhesives

Several polysulfide-based adhesive formulations were prepared which appeared promising. These materials were cured by means of metal oxides. The best formulation contained the following ingredients:

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Component	Parts by Weight
Polysulfide LP-33[a]	16.8
Manganese dioxide	1.0
Activator Paste[b]	2.4
Continex CF Carbon Black	75.4
J2 Silver Powder	4.4

[a]Thiokol Corporation

[b]A mixture of 16.6 weight % each of benzene, meta-dinitrobenzene and dibutyl phthalate, and 50.2 weight % Monsanto's Aroclor 1254.

The tensile strength of this formulation was 280 lbs. per sq. in. and the peel strength was 6 lbs. per in. Although these values were lower than are desired, the adhesive exhibited a satisfactorily low specific resistivity.

Only one formulation, the polyurethane adhesive, approaches the desired tensile strength and peel strength with the required electrical resistivity and heat stability. Further modifications of the three adhesive bases are being made, and some of the more recently developed formulations appear promising.

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