MASTER

PHYSICAL PROPERTIES OF HALTHANE SERIES
ADHESIVES, PART 1

H. W. Lichte

DEVELOPMENT DIVISION

APRIL - JUNE 1976

Normal Process Development
Endeavor No. 101

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PHYSICAL PROPERTIES OF HALTHANE SERIES ADHESIVES, PART 1

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INTRODUCTION

The Halthane series of urethane adhesives synthesized by Lawrence Livermore Laboratory(1) are being studied for application to Pantex assembly processes. The series consists of three each two-part systems designated 73-14, 73-18, and 87-1. [The 73-18 system to be used] was chosen as the first of the three for physical property testing. Each system has certain advantages, and is designed for a specific application(). Systems 73-14 and 7318 have a lower viscosity than 87-1. System 73-18 has a lower tensile and compressive modulus than 73-14 and 87-1. The increasing order of pot life would rank 73-18, 87-1, and 73-14. All three systems reach two-third hardness in 16 hours.

These new adhesives will be investigated and utilized by several production plants in the ERDA complex. The Pantex study effort will include measurement of bulk viscosity, cure time, butt tensile strength, lap shear strength, hardness and compatibility. One significant part of the study is familiarization with process and handling requirements that may be unique with the Halthane series.

DISCUSSION

The mixing formulation for each of the three systems is different. The raw materials and mixing ratios for this study were supplied by LLL. The development process for manufacture of the prepolymer at BKC currently suggests different stoichiometry for each batch. The formulations used for this study are listed in Table I.

The containers for each component were protected from contamination and moisture in the ambient laboratory environment. The curing agents were solidified in the cans and required a two hour 60 C soak to liquify. A vacuum mixing system was used in all sample preparations. One hundred gram samples were mixed for each series of butt tensile specimens which allowed for a cure hardness verification sample.

The adhesive bonded aluminum to aluminum samples were held in alignment fixtures for three days before disassembly. The aluminum adherends were previously cleaned with toluene followed by a isopropyl alcohol wash.

The average bond thickness was 0.13 mm. The bonded area was assumed 1.3 or 6.4 cm² for the PX or LLL adherends, respectively. All samples were tested to failure at the constant crosshead speed of 1.27 mm/minute at either 25 or 75 C. Butt tensile strengths are listed in Table II.

The small (1.3 cm²) samples were tested for later correlation with large samples and to facilitate testing with a smaller load cell and Instron Machine. Use of the smaller machine may allow testing of the desired 30 day cure time by improved test scheduling.

The Shore A hardness of the adhesive mix tailings was measured for cure verification one week after sample bonding. The mean values were:

<u>System</u>	<u>Shore A Hardness</u>
73-18	60
73-14	75
87-1	72

These values may be low because of bubbles formed on the surface of the curing sample due to the aliquots drawn for the adhesive specimen preparation.

Bulk viscosity was measured with the Brookfield RVT viscometer at 10 RPM. The prepolymer 73 was 8.35 Pa's at 23.8 C with the No. 3 spindle. The two curing agents HGH-14 and HGH-18 were 0.688 and 0.740 Pa's, respectively, at 22.2 C with the No. 1 spindle after being soaked at 60 C the previous day. The prepolymer 87 was 84.7 Pa's at 23.9 C using the No. 6 spindle. It had been soaked two hours at 60 C six days prior and the container opened twice in the interval.

The tensile adhesion test results with the larger (6.4 cm²) Halthane samples infer a slight drop in strength due to age of the bond. The bond strengths overall are less than previously reported(2), which may be attributed to the simple cleaning procedure in lieu of the ASTM D2651(3) acid etch which normally produces the highest adhesion values(4).

Future work in the next quarter will include testing of the 87-1 and the conclusion of the 73-14 work.

Table I. Halthane Mixing Ratios

<u>Adhesive System</u>	<u>NCO/OH</u>	<u>Prepolymer Parts by Wt.</u>	<u>Curing Agent Parts by Wt.</u>
73-14	1:0.9	No. 73 (Lot 6, BKC 20901) 64.3%	HGH-14 35.7%
73-18	1:0.95	No. 73 (Lot 6, BKC 20901) 64.4%	HGH-18 35.6%
87-1	1:1	No. 87 (Lot 7, BKC 20902) 93.5%	TONOX 60/40 6.5%

Table II. Halthane Butt Tensile Strength

<u>Adhesive</u>	<u>Test Temp. (C)</u>	<u>Cure Time (days)</u>	<u>Butt Tensile Strength, Mean (MPa)</u>	<u>Remarks</u>
73-18				
Set 1	25	45	5.5	9 large samples
Set 2	25	35	6.1	10 large samples
Set 3	75	35	3.9	9 large samples
Set 4	75	42	3.6	5 large samples
Set 5	75	42	4.6	6 small samples
Set 6	25	35	5.5	5 small samples
73-14				
Set 1	25	10	6.6	5 small samples
Set 2	25	10	6.8	6 small samples
Set 3	75	10	3.6	3 small samples

REFERENCES

1. L. P. Althouse and H. G. Hammon, Chemistry Department Tech Note 75-24, LLL, July 14, 1975.
2. L. P. Althouse and H. G. Hammon, "Development of Halthane Adhesives for Phase 3 Weapons: Status Report," LLL, UCID 16990, December 29, 1975.
3. ASTM D2651-67 (1973), "Preparation of Metal Surfaces for Adhesive Bonding," 1975 Annual Book of ASTM Standards, Part 22.
4. N. J. DeLollis, Adhesives for Metals, Industrial Press Inc., New York, 1970.