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Physical Properties of Halthane Series Adhesives, Part 2 of 2

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DEVELOPMENT DIVISION

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Normal Process Development
Endeavor No. 101

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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 has been selected for program use and thus was chosen as the first of the three for physical property testing. Each system has certain advantages, and is designed for a specific application(*2*). Systems 73-14 and 73-18 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(*6*). 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 (as received) 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.

TENSILE ADHESION

One hundred gram samples were mixed for each series of tensile specimens and the tailings used 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 an isopropyl alcohol wash.

The butt tensile specimen bond area was 1.3 cm^2 and the lap shear was 3.2 cm^2. All samples were tested to failure at the constant crosshead speed of 1.27 mm/minute at either 25 or 75 C. Butt tensile and lap shear strengths are listed in Tables II through IV. The test series consists of 288 bonds and 13 individual mixing operations.

The Halthane 73-18 adhesive strength data are listed in Table II. A set represents one mix of adhesive to assemble the test specimens. All sets were from the same lot of raw materials and stoichiometry (64.4/35.6). The calculated mean strengths represent a group of 5 to 6 test specimens with all values retained.

There appears to be a strength difference between sets due to sample preparation/assembly. The loss at higher temperatures and gain upon aging is expected. The butt specimens in sets 9 and 10 were assembled in a different manner than in sets 7 and 8. The high value in set 10 is attributed to the larger adhesive gap.

The Halthane 73-14 adhesive strengths are listed in Table III. All sets are from the same lot and stoichiometry (64.3/35.7). The 73-14 adhesive generally is stronger than the 73-18. It is similar to the 73-18 in sensitivity to sample preparation/assembly and bondage.

The Halthane 87-1 adhesive strengths are listed in Table IV. All sets are from the same lot and stoichiometry (93.5/6.5). This adhesive system is much stronger than either of the other Halthanes in failure of butt or lap tensile specimens. The sensitivity to sample preparation/assembly is more predominate as evidenced by changes in bond strength and thickness.

The adhesion testing program was designed to account for raw materials, mixing, assembly, cure time, temperature, butt failure, and lap shear failure(5). All the variables as one population produced an average coefficient of variation of 15% with a range of 4 to 44% for the tensile adhesion failures. The individual Halthanes were 14% for 73-18, 20.5% for 73-14 and 11% for 87-1.

BULK VISCOSITY

Bulk viscosity of the urethanes was measured with the Brookfield RVT viscometer in a temperature-controlled bath. Each adhesive component was thermally soaked to originally correspond to the bath temperature(5). The curing viscosity changes were measured together with the exotherm temperature.

Halthane prepolymer 73 was measured at 25 C and the viscosity calculated 6.95 Pa•s. Curing agent HGH-18 was added, mixed and deaerated. The curing mass consisted of 257.6 g prepolymer to 142.4 g of curing agent. The viscosity change was measured at 10 rpm (spindles 3 through 7). Fig. 1 is a plot of the viscosity change with cure time and temperature increase. The HGH-18 viscosity was previously measured at 22.2 C and 0.74 Pa•s.

Halthane prepolymer 87 was measured at 25 C and the viscosity calculated 75.2 Pa•s. Tonox was added and mixed in the ratio of 374 g prepolymer to 26 g curing agent. The viscosity change was measured at 10 rpm using spindles 6 and 7. Fig. 2 is a plot of the viscosity change with cure time. There was no appreciable temperature rise due to curing, after 39 minutes, 3.6 C differential.

HARDNESS

The Shore Conveloader with the Shore A gauge was used to measure hardness at 22.2 C cure for nine hours. Five samples each 5.08 by 5.08 cm square and 0.64 cm thick were tested for hardness change due to curing. The measurements were averaged and the mean plotted with time on Fig. 3.

Table I. Halthane Mixing Ratios

Adhesives System	NCO/OH	Prepolymer Parts by Wt.	Curing Agent Parts by Wt.
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 73-18 (BKC-20901, Lot 6) Tensile Adhesion

Assembly Group (Set)	Test Temperature (C)	Cure (Days)	Mean Strength Values (MPa) Butt	Lap	Mean Gap (mm)
7	25	10	5.3	-	0.15
	25	35	4.3	-	0.15
	75	35	2.2	-	0.18
	75	10	3.1	-	0.13
8	25	10	3.1	-	0.15
	75	10	2.6	-	0.15
	75	55	2.9	-	0.15
	25	55	5.8	-	0.13
	25	10	-	0.8	0.08
	25	30	-	0.9	0.08
	25	55	-	1.4	0.05
9	25	10	-	1.1	0.05
	75	10	-	0.5	0.08
	25	30	-	1.4	-
	25	30	4.7	-	0.10
10	25	10	6.3	-	0.20

Table III. Halthane 73-14 (BKC-20901, Lot 6) Tensile Adhesion

Assembly Group (Set)	Test Temperature (C)	Cure (Days)	Mean Strength Values (MPa) Butt	Mean Strength Values (MPa) Lap	Mean Gap (mm)
4	25	10	6.2	-	0.16
	75	10	3.5	-	0.15
	75	35	2.2	-	0.13
	25	35	4.1	-	0.14
5	25	10	5.0	-	0.13
	75	10	2.7	-	0.13
	75	56	5.2	-	0.10
	25	56	8.7	-	0.14
	25	10	-	0.64	0.07
	25	30	-	1.02	0.07
	25	56	-	3.66	0.09
6	25	9	-	0.68	0.07
	25	30	-	1.56	0.06
	75	9	-	0.37	0.06
	25	30	6.47	-	0.14
7	25	10	5.47	-	0.14

Table IV . Halthane 87-1 (BKC-20902, Lot 7) Tensile Adhesion

Assembly Group (Set)	Test Temperature (C)	Cure (Days)	Mean Strength Values (MPa) Butt	Lap	Mean Gap (mm)
1	25	10	10.22	-	0.17
	75	10	6.65	-	0.17
	75	35	7.07	-	0.13
	25	35	8.83	-	0.08
2	25	9	8.91	-	0.17
	75	9	3.00	-	0.21
	75	55	2.94	-	0.14
	25	55	8.85	-	0.16
	25	10	-	0.98	0.06
	25	30	-	2.04	0.10
	25	55	-	2.75	0.05
3	25	10	-	3.17	0.06
	25	30	-	3.01	0.07
	75	10	-	1.42	0.06
4	25	30	14.09	-	0.12
5	25	10	15.55	-	-

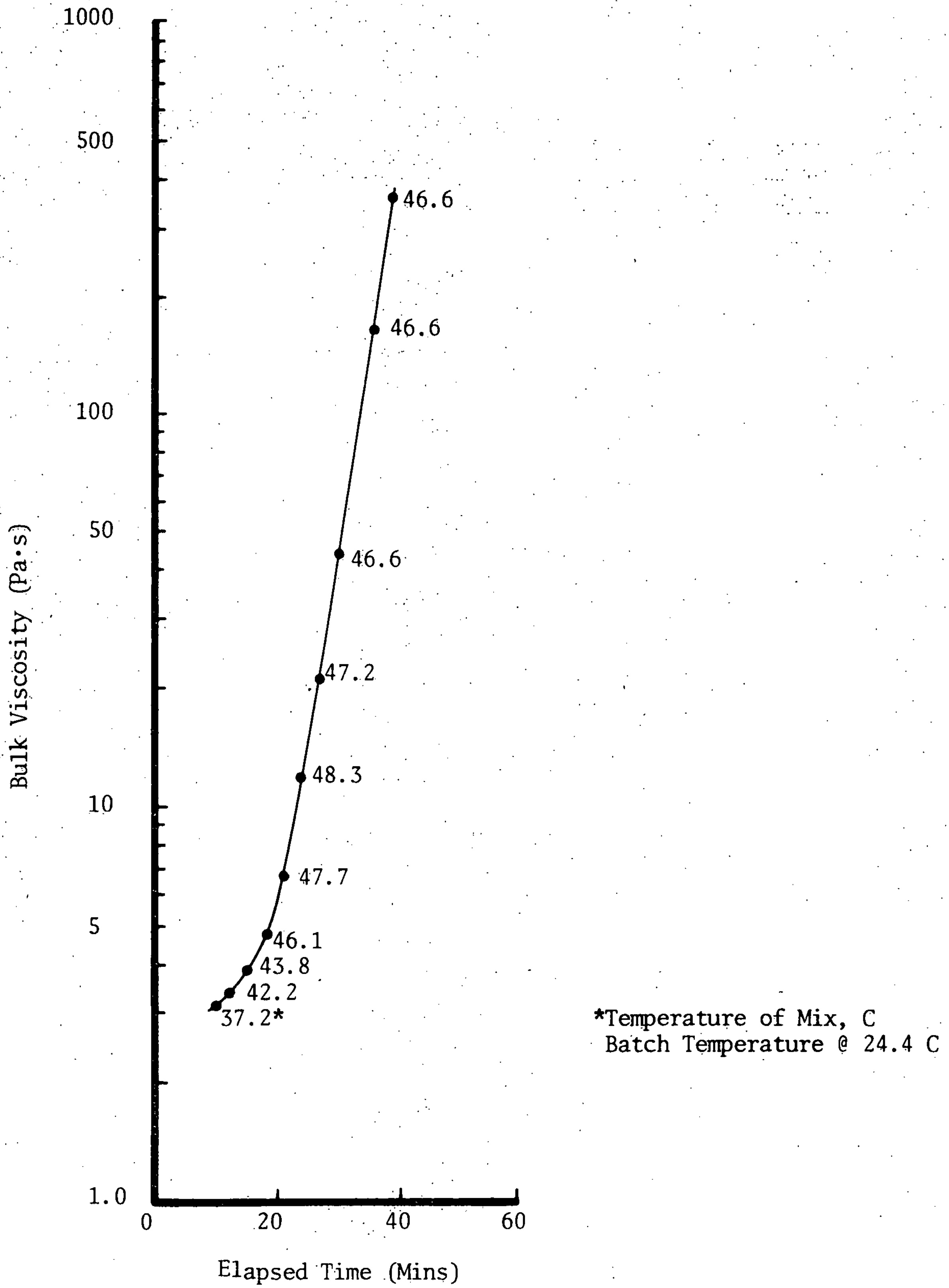

Fig. 1. Halthane 73-18 Viscosity Change with Cure

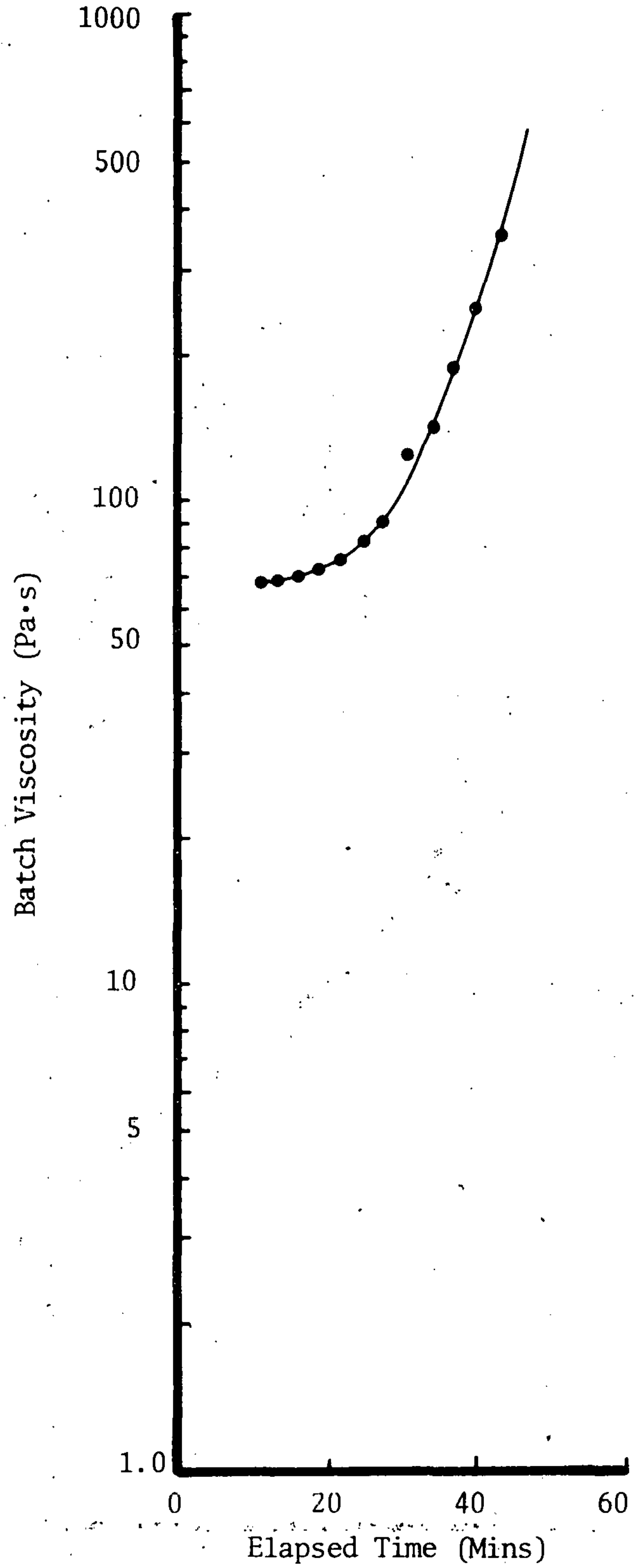

Fig. 2. Halthane 87-1 Viscosity Change with Cure

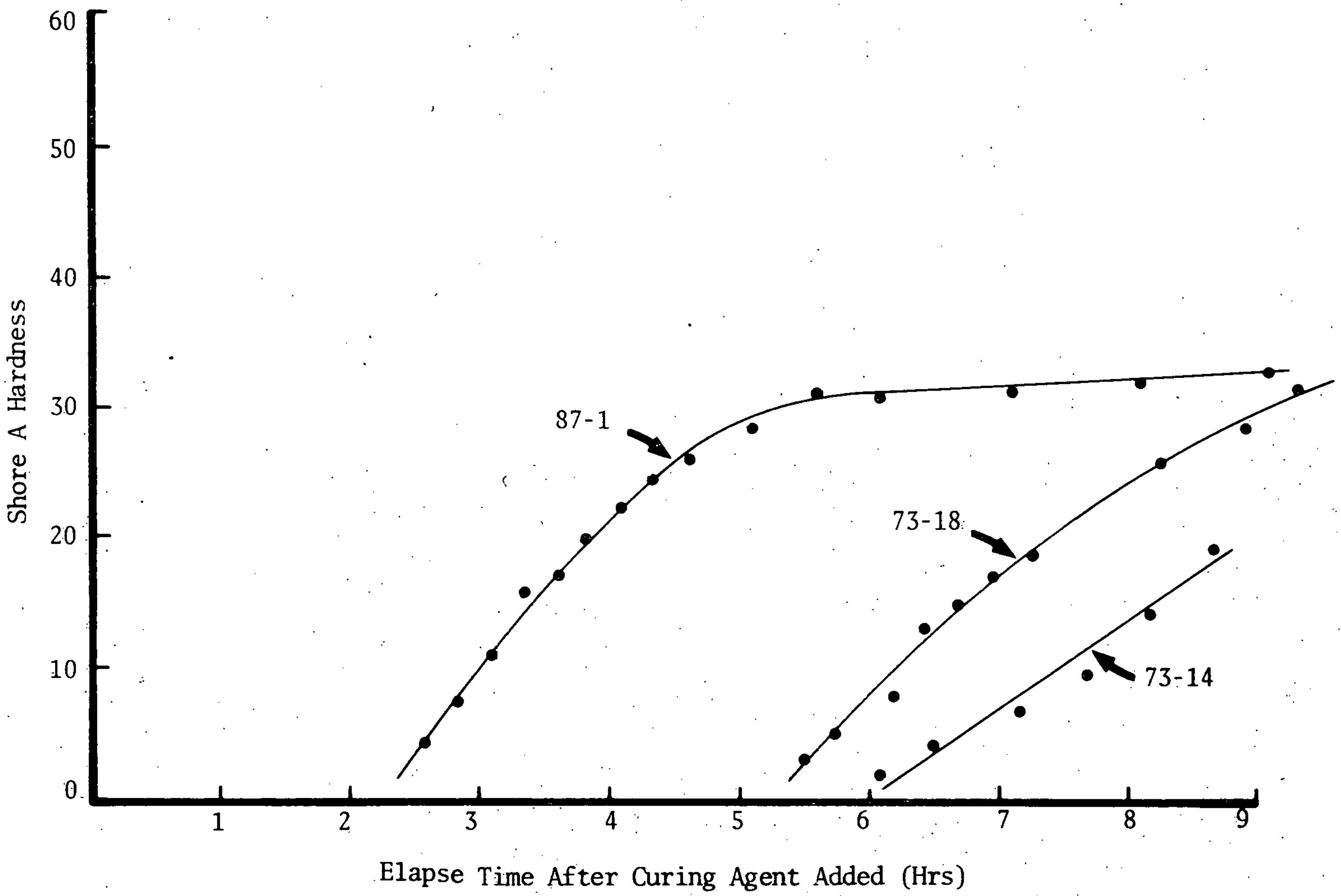

Fig. 3. Halthane Shore A Hardness Change with Cure

The Halthanes 87-1, 73-18 and 73-14, respectively, were 36, 50, 55 Shore A after 24 hours and 36, 51, 69 Shore A after 72 hours. The maximum coefficient of variation between like samples was 8% for 73-18, 5% for 87-1 and 12% for 73-14.

CONCLUSIONS

The Halthane series of adhesives have sufficient adhesive strengths and cure times for most assembly processes at Pantex. The efficiency of preparation and handling of the three systems are nearly equal. Physical properties are more consistent between samples for the 87-1. The 73-18 system is ranked in the middle and 73-14 as the least consistent. The 87-1 system is high in viscosity and adhesive bond strength. The low viscosity of 73-18 and 73-14 allows more consistency in bond thickness control. The Halthane 73-18 system in this series of development tests is considered best for processing/assembly capability.

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