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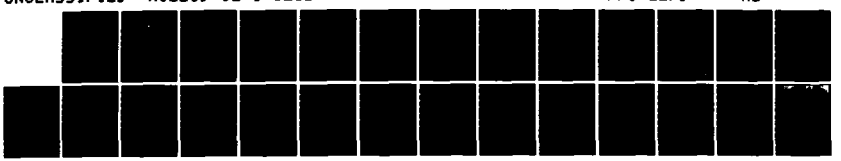

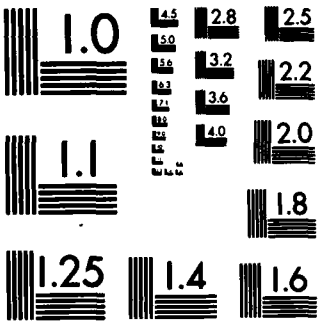

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REPORT NO. NADC-81108-60

AD-A135 390

DEVELOPMENT OF A STABLE EPOXY RESIN SYSTEM FOR COMPOSITE REPAIR

Jonas Weiss
CIBA-GEIGY CORPORATION
PLASTICS AND ADDITIVES DIVISION
. Ardsley, New York

MARCH 1983

FINAL REPORT
CONTRACT NO. N62269-81-C-0153

Approved for Public Release: Distribution Unlimited

Prepared For
NAVAL AIR DEVELOPMENT CENTER
Warminster, Pennsylvania

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1. REPORT NUMBER NADC-81108-60	2. GOVT ACCESSION NO. A125390	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) DEVELOPMENT OF A STABLE EPOXY RESIN SYSTEM FOR COMPOSITE REPAIR		5. TYPE OF REPORT & PERIOD COVERED Final, Sept. 22, 1981 to Nov. 15, 1982 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) JONAS WEISS		8. CONTRACT OR GRANT NUMBER(s) N62269-81-C-0153
9. PERFORMING ORGANIZATION NAME AND ADDRESS CIBA-GEIGY Corporation Plastics & Additives Division Ardsley, NY 10502		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 61153N; R02204-001; DG202
11. CONTROLLING OFFICE NAME AND ADDRESS Aircraft & Crew Systems Technology Directorate Naval Air Development Center (Code 6063) Warminster, Pennsylvania 18794		12. REPORT DATE March, 1983 13. NUMBER OF PAGES 23
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

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17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Composite Repair
Latent Epoxy
Latent Hardener
One-part Epoxy
Epoxy Prepreg

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Novel latent epoxy hardeners which show promise for the field repair of composite structures of naval aircraft have been developed and tested. Two hardeners, phthalamides of 1,2-diaminocyclohexane (DACP) and of m-xylylenediamine, were latent with multifunctional epoxy resins for 4 to 12 months and yield glass transition temperatures of 145°C and 130°C, respectively, when cured at 150°C. Fracture toughness (K_{Ic}) of a DACP/MY 720 molding is 2.27 kg/mm$^{3/2}$.

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20. Abstract (continued)

slightly higher than that of a standard diaminodiphenyl sulfone (DDS)/ MY 720 system.

Graphite prepreg was made using DACP/MY 720 and DDS/MY 720 matrix resins, and unidirectional, well-consolidated laminates were prepared at low pressure (100 psi). Short-beam-shear and tensile values for the phthalamide laminate range between 72% and 105% of the values determined for the DDS/MY 720 laminate. Dynamic mechanical analysis shows the Tg of the DACP laminate to be 130°C and to decrease by about 9°C with 1.1% water absorption on aging at 49°C and 95% relative humidity. This hot/wet conditioning reduces shear and tensile properties much more than for the DDS/MY 720 control laminate.

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FOREWORD

This report was prepared by CIBA-GEIGY Corporation, Ardsley, NY, under contract NADC N62269-81-C-0153, "Composite Repair System with Long-Term Latency". It is the final technical report for the contract for the period September 22, 1981 to November 15, 1982. The program was sponsored by the Naval Air Development Center, PA. It was performed under the supervision of Mr. Ron Trabocco, contract monitor for NADC.

Performance of this contract is under the direction of the Plastics and Additives Resins Department of CIBA-GEIGY Corporation. Dr. L. McAdams is Technical Director and Dr. J. Weiss is Project Leader and principle investigator.

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INTRODUCTION

This report describes the research under contract No. N62269-81-C-0153 to prepare and characterize a one-part resin system to be used in the field repair of graphite structural composites on naval aircraft.

The resin system should:

1. have long-term (at least six months) stability at ambient temperatures.
2. cure in one hour at 121-149°C (250-300°F) using a heating blanket and mechanical pump.
3. provide performance (e.g., high temperature properties and water resistance) approaching that of the resin used in the original advanced composite material. The basis for the matrix resin system of many of the composites for aircraft components is tetraglycidylated methylenedianiline (CIBA-GEIGY's MY 720) cured with bis-(p-aminophenyl)sulfone (Eporal or DDS). Because of the low reactivity of DDS at readily achieved temperatures, an accelerator, such as the complexed Lewis acid, BF_3•MEA is incorporated. Even with this accelerator, the MY 720/DDS system requires cure at high temperatures (177-200°C) to insure full reaction and development of composite properties.

Although a repair system based on the MY 720/DDS matrix could provide properties equivalent to the advanced composite substrate, such a system is unsuitable for field applications because of the high temperatures necessary to fully cure the system. Therefore, lower temperature curing systems were investigated.

In the previous contract period, four new phthalamide-amine curing agents were synthesized and characterized. Latency of m-xylylenediamine with multifunctional epoxy resins was found to last as long as four to five months at ambient temperatures, glass transition temperatures ranged up to about 142°C and water absorption at equilibrium at 95% relative humidity and 71°C was a relatively low 5.5 weight percent. A major problem with the materials developed has been their lack of solubility in epoxy resins at curing temperatures (under low pressures). Cured samples did not consolidate and remained heterogeneous, unless cured under high pressure. The object of the work in this contract is to improve compatibility of the components at normal curing temperatures (up to 150°C) and at low pressures (under 100 psi) so that a practical system can be made. This is to be followed by the preparation of test samples and the determination of their physical properties, e.g., tensile and fracture properties, and dynamic mechanical analysis (to determine Tg and the effect of hot/wet aging on Tg). The fabrication and properties of graphite prepreg and laminate using multifunctional epoxy resins and the most promising latent hardener are investigated.

EXPERIMENTAL RESULTS AND DISCUSSION

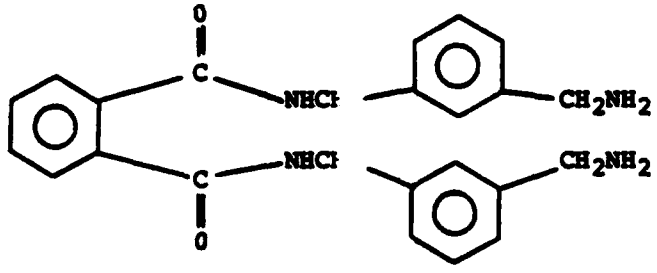
A. Compatibility Improvement

Additional batches of amine-phthalamide adducts were synthesized as described in the November, 1981, final report on contract No. N62269-80-C-0711.

Various techniques were investigated in order to prepare clear, homogeneous test samples of later phthalamide hardeners with multifunctional epoxy resins for the determination of their physical properties. Compression moldings of the mixed components gave consolidated samples, but these were inhomogeneous, especially at relatively low pressures.

1. Particle Size Reduction

The phthalamide of m-xylenediamine (MXDAP)* hardener described in the previous reports was ground in a Wiley mill and sieved to isolate material of a very fine particle size, below 75 microns. MXDAP (4.4 parts by weight, 100.6 grams/equivalent) was hand mixed with the multifunctional epoxy resins MY 720 (2.37 pbw) and 0510 (2.37 pbw) to yield a uniform paste. This was molded in a one-inch diameter mold at temperatures up to 150°C and at about 50 psi. The mixture cured to a hard mass, but was heterogeneous, with numerous particles evident. Particle size reduction alone is apparently not a solution to compatibility improvement of this system.



MXDAP

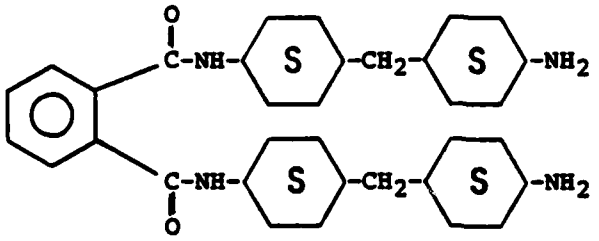
2. Epoxy Resin Substitution

A study was done to determine whether resin compatibility can be improved by substitution of standard bifunctional epoxy resins for part of the multifunctional resins. ARALDITE® 6010, a diglycidyl ether of bisphenol A, was mixed with MXDAP and molded at about 50 psi. An opaque molding resulted. A low viscosity cycloaliphatic epoxy resin, ARALDITE® CY 179, was also used to replace half of the MY 720 in a mixture of MY 720/0510/MXDAP/PACMP. The resulting sample was also heterogeneous after molding. Replacement of 20% of the MY 720 with a low melting solid epoxy resin, ARALDITE® GT 7014, did improve the clarity of the samples somewhat, but heterogeneities were still evident.

*Preparation and analysis described in the November, 1981 final report on Contract No. N62269-80-C-0711.

3. Combinations of Hardeners

A mixture of phthalamide hardeners may exhibit a depressed melting point, and thereby dissolve more readily in epoxy resins before hardening. PACMP (the phthalamide of duPont's PACM-20)* was mixed with MXDAP in a 1:1 molar ratio. This was molded with MY 720/0510, with MY 720/0510/6010, with MY 720/6010, with MY 720/0510/CY 179, and with ARALDITE® 6010. Heterogeneous moldings resulted from all of these mixtures.



PACMP

4. Addition of Solvents

Clear films or uniform powders containing phthalamide hardener and epoxy resin were prepared by first dissolving each component in an appropriate solvent. The phthalamides were dissolved in methanol up to a concentration of 33% by weight. The epoxy resin was dissolved in acetone (or alternatively in 2-butanone or methylene chloride) at 26 wt. % solids. Mixtures of these solutions gave clear, brown solutions.

a. m-Xylylenediamine Phthalamide (MXDAP) + MY 720 + 0510

The MXDAP hardener was dissolved in anhydrous methanol and epoxy resins MY 720 and 0510 were dissolved in acetone. The two solutions were mixed in a stoichiometric ratio (using 4 amino hydrogen equivalents per mole MXDAP) to yield a clear solution. The solution was sprayed onto teflon-coated aluminum plates and residual solvent was stripped at 0.1 mm mercury and ambient temperature. A yellow powder melting at 90-100°C was obtained.

A clear, brown disc was molded from this powder at about 100 psi; its Tg was 110°C. DSC analysis showed a heat of reaction of 17.7 kcal/epoxy equivalent centered at about 130°C; this is 89% of the value expected for non-reacted material. However, Δ H fell to 9.2 kcal/equivalent within one week, showing that the material is advancing significantly. A few percent of solvent was found to be retained in the mix, which could account for the low Tg as well as the advancement. The addition of n-hexane to the acetone/methanol solution did not aid in stripping off the last remnants of solvent.

*Preparation and analysis described in the November, 1981 final report on Contract No. N62269-80-C-0711.

b. MXDAP + MY 720

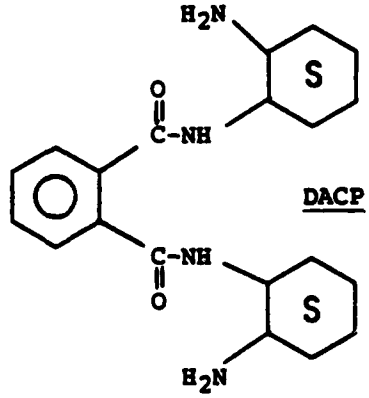
The elimination of 0510 resin (triglycidyl p-aminophenol) from the previous formulation facilitates the preparation of a dry solvent-free powder, melting at 62-67°C. Two different batches prepared from methanol/acetone gave heats of reaction of 19.7 and 17.2 kcal/epoxy equivalent. However, the mixture was not stable at ambient temperatures, since these values fell to 10.7 and 12.5 kcal/equivalent after seven days.

c. PACM-20 Phthalamide (PACMP) + MY 720

PACMP was dissolved in anhydrous methanol and mixed with a stoichiometric amount of MY 720 in acetone (using 4-amino hydrogen equivalents per mole of PACMP). Spraying and overnight evaporation yielded an almost white powder melting at 60-65°C. The heat of reaction was determined (via DSC) to be 7.0 kcal/epoxy equivalent, less than half of the value obtained in the absence of solvent treatment. Apparently, the components react during preparation of this powder.

d. 1,2-Diaminocyclohexane Phthalamide (DACP) + MY 720

A total of 340 grams of the phthalamide of 1,2-diaminocyclohexane (DACP) was prepared in two batches, according to the procedure described in the final report on Contract No. N62269-80-C-0711. The material melts at 75-85°C and IR analysis shows no contamination with imide or with amine salt.



DACP

Attempts to make a molding powder out of DACP, MY 720 and the trifunctional epoxy resin 0510 were unsuccessful because solvent could not be completely removed from the gummy solid formed without heating, which would start the cure reaction.

The phthalamide was successfully converted to a powder by omitting the 0510 and using a stoichiometrically equivalent amount of MY 720. This was done by making a 33 wt. % solution of DACP in methanol and mixing with a 26 wt. % solution of MY 720 in acetone. The combined solution was sprayed onto teflon-coated

plates and evacuated to remove solvent. The resulting pale yellow material was ground to a fine powder with a mortar and pestle prior to molding. Its melting range is 77-81°C.

Translucent discs of 1 1/8-inch and 2 1/4-inch diameter were molded from this powder at 34 psi and 150°C. The material consolidates, but small bubbles and particles can be seen on microscopic observation. At 750 psi, no bubbles are seen and inhomogeneities are mostly on the surface.

Compatibilization of the components using a mixed solvent was also used to make graphite prepreg with DCAP/MY 720 matrix resin.

B. Stoichiometry of the MXDAP/Epoxy Reaction

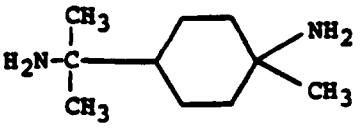
Differential scanning calorimetry showed that a significantly higher heat of reaction was obtained when a stoichiometry of four amino hydrogen equivalents/mole of MXDAP was used than when six equivalents/mole was used. To optimize the MXDAP/epoxy resin ratio further. MXDAP was added in ratios based on stoichiometries of three and five amino hydrogen equivalents per mole and the reaction with MY 720 plus 0510 studied via DSC. The results are shown in Table I. The stoichiometry of four amino hydrogen equivalents/mole gives the highest heat of reaction as well as the lowest temperature of reaction. The ratio based on this value appears to be the optimum for the complete low-temperature cure of this system.

TABLE I: STOICHIOMETRY OF THE MXDAP/EPOXY REACTION

Amino Hydrogen Equiv./Mole MXDAP	Center of 1st DSC Peak	1stΔH (Kcal/mole)
3	118°C	54.9
4	114°C	69.8
5	133°C	38.3
6	117°C	36.9

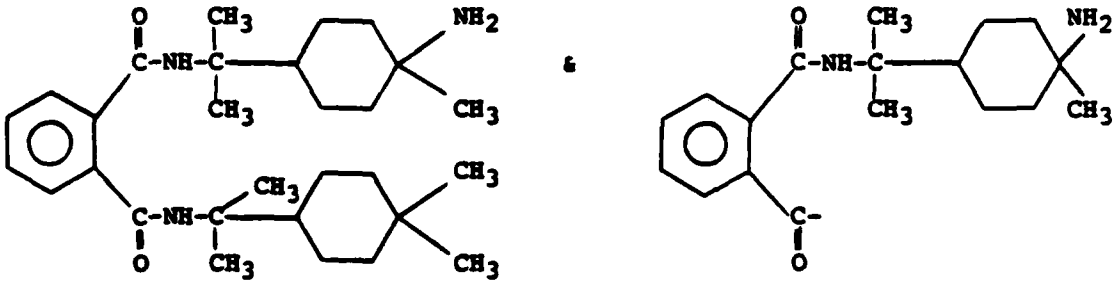
C. Synthesis and evaluation of 1,8-Diamino-p-Menthane Phthalamide (DAMP)

A new, potentially useful latent hardener was synthesized from the hindered amine, 1-8-diamino-p-menthane (DAM)



DAM

The amine was reacted with phthalic anhydride in dioxane solution at 105°C, according to the technique used in making MXDAP. A yellow oil was formed, which was transformed into a yellow powder (M.P. 133-145°C, 67% yield) on washing successively with hot heptane, toluene, hexane, and ethyl ether. Infrared analysis shows that the material is an amide, with no imide groups. NMR analysis indicates that the material is a mixture of 2:1 and 1:1 amine:anhydride adducts of the structures shown below:



DAMP

A mixture of DAMP with MY 720 and 0510 was latent for four days at about 21°C, but hardened after one week. This is probably due to the presence of some unreacted amine, which was found using thin layer chromatography. The heat of reaction with MY 720 plus 0510 was only 9.3 kcal/epoxy equivalent, centered at 130°C. Tg for this system was low (85°C).

D. Latency of Phthalamide/Epoxy Mixtures

Mixtures of PACMP with MY 720, and with MY 720 plus 0510, were not appreciably latent at room temperature for more than a few weeks; they were very hard after three months. Similarly, mixtures of DAMP, and of the phthalamide of 1,3-bis(aminomethyl)cyclohexane with a combination of MY 720 and 0510 got noticeably harder in a few weeks. Differential scanning colorimetry (DSC) was used to evaluate the stability of the other phthalamide/epoxy mixtures stored at room temperature. The results are given in Table II.

The most stable formation is apparently MXDAP/MY 720; it retains 83% of its original reactivity after one year. However, this material is also the most difficult to process. MXDAP/MY 720/0510 can be molded, and retains 84% of its original activity after 4 months. DACP/MY 720 mixtures from solution show no decrease in heats of reaction after 3 months and retain 90% of their original value after 6 months. Other formulations containing PACMP, DAMP, or BACP become hard within 3 months of storage. Apparently, traces of retained solvent make the compounds reactive, in contrast to dry-blended mixtures, which were previously found to be stable for as long as five months.

TABLE II: HEATS OF REACTION OF HARDENER/EPOXY MIXTURES WITH TIME

	Δ H (kcal / mole)				
COMPONENTS	ORIGINAL	3 mos.	4 mos.	6 mos.	1 Year
DACP / MY 720	61.4	62.4	-	55.0	-
MXDAP / MY 720 / 0510	69.8	-	58.7	37.9	-
MXDAP / MY 720	86.4	-	-	-	72.0

E. Preparation of Prepreg and Laminate Test Samples

Unidirectional prepreg was prepared by pulling Thornel 300 graphite fiber (Union Carbide grade WYP 15, finish UC 314) through a solution of epoxy resin and hardener, and winding on a 12-foot circumference drum at 12 tows/inch. These prepregs were cut off the drum, dried overnight at ambient conditions, trimmed to a convenient size, and stored in a freezer until used to make laminates. No B-staging was done before molding. Table III lists the materials used and the resin contents obtained for the phthalamide system and for the control (all parts are by weight). The DACP-containing prepreg had considerably less tack and drape than the DDS-containing control.

TABLE III: PREPREG FORMULATIONS AND RESIN CONTENT

Resin	MY 720	MY 720
Hardener	DDS (47 phr)	DACP (68 phr)
Catalyst	BF_3•MEA (1 phr)	---
Solvent(s)	Acetone	Acetone/Methanol (2.5/1)
Solids Content of Solution	38%	26%
Volatiles in Prepreg	1.7%	3.1%
Prepreg Fiber Content	62.4%	63.7%
Prepreg Resin Content	37.6%	36.3%

Zero degree, 8-inch by 8-inch laminates were molded at 100 psi. Twelve-ply laminates were made for short-beam-shear tests and dynamic mechanical analysis; seven plies were used to make laminates for tensile tests. The DDS/MY 720 control was molded at 120°C for 1 hour, 177°C for 1 1/2 hours, and 200°C for 2 hours. The DACP/MY 720 laminate was molded at 120°C for 1/2 hour and 150°C for 1 1/2 hours. The phthalamide laminate had less flow than the control, but both laminates were well consolidated and appeared uniform, with fibers fully coated.

F. Test Results

1. Molded DACP/MY 720: DMA Results, Tg, and the Effect of Hot/Wet Aging

The glass transition temperature of small samples of DACP/MY 720 molded at 34 psi and 150°C was found to be 145°C, using thermomechanical analysis and also via differential scanning calorimetry. However, these methods could not be used to determine the Tg of water-saturated samples with reasonable accuracy, so dynamic mechanical analysis, using a duPont 981 DMA, was used for this purpose.

Compression-molded DACP/MY 720 was cut into rectangular specimens of dimensions 25 X 10 X 1.8mm. These were aged at 71°C and 95% relative humidity. The gain in weight seemed to stabilize at 4.2%

after three weeks. DMA at 5°C/min. was used to determine Tg, elastic modulus and loss modulus (at 25 Hz) of conditioned and non-conditioned samples. The averages of these data are given in the following table. Tg of the non-conditioned samples (from the peak of the damping curve) was 165°C, 15° above the final cure temperature and about 20° above the Tg previously found via DSC and thermomechanical analyses. On conditioning, DMA shows a drop in Tg of 34°, to 131°C. Changes in elastic modulus and in loss modulus due to hot/wet aging were not significant (within the method's experimental error of 10%). However, both moduli drop off as the Tg is approached.

TABLE IV: DMA VALUES FOR MOLDED DACP/MY 720

	NON-CONDITIONED	CONDITIONED
Tg (°C)	165	131
Elastic Modulus @ 25°C (GPa)	3.3	3.2
Elastic Modulus @ 100°	2.7	2.3
Elastic Modulus @ 140°	2.1	0.64
Elastic Modulus @ 165°	0.93	0.25
Loss Modulus @ 25°C (tan δ)	0.029	0.029
Loss Modulus @ 100°	0.030	0.047
Loss Modulus @ 140°	0.064	0.291
Loss Modulus @ 165°	0.245	0.171

2. Fracture Toughness of Resin Systems

Six 2 1/4-inch diameter, 1/4-inch thick discs of DACP and MY 720 were molded at 750 to 1000 psi and cut for a compact tensile fracture toughness test (figure 1). They were translucent and bubble-free, but contained some inhomogeneous particles. Various modifications of the heating schedule failed to improve the sample appearance. Eporal/MY 720 discs were made to control samples for this test. The average critical stress intensity factor (K_{Ic}) for DACP/MY 720 was found to be 2.27 kg/mm$^{3/2}$, with a standard deviation of 0.16. This is very similar to the value found for Eporal/MY 720, which was 1.92 kg/mm$^{3/2}$ (S.D. = 0.58).

Discs for fracture toughness were also molded (at 6000 psi) from m-xylylenediamine phthalamide (MXDAP) and MY 720. Moldings were translucent, but more inhomogeneous than with DACP/MY 720. Several processing modifications were tried to mold more homogeneous samples, but the resulting discs were not suitable for testing.

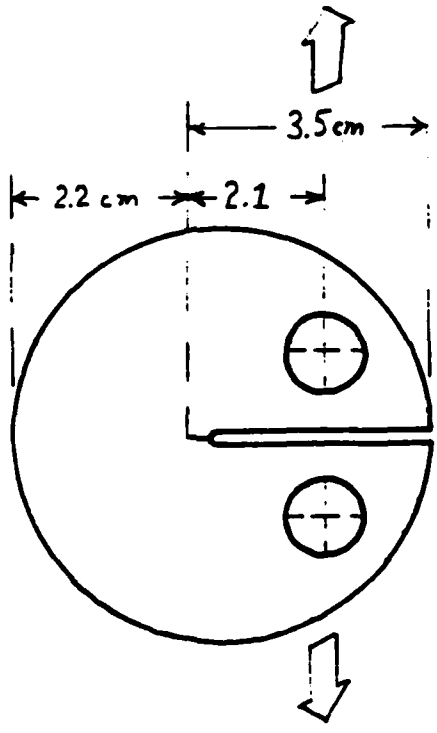


FIGURE 1: COMPACT TENSILE TEST SAMPLE

3. Hot/Wet Aging of Laminates

Samples for testing were divided into two groups; one was kept at ambient laboratory conditions and the other kept in a chamber at 49°C and about 95% relative humidity until its weight remains constant (about 3 weeks). DACP/MY 720/Thornel 300 laminates gained a total of 1.1% of their weight at equilibrium; the DDS/MY 720/Thornel 300 laminates picked up 0.85% of water.

4. Short-Beam-Shear Strengths of Laminates

Twelve-ply, 0.14 cm-thick laminates were tested as shown in figure 2, at a crosshead speed of 0.05 inch/minute (ASTM No. D2344). Hot/wet aged samples were tested rapidly (in about one minute) to minimize drying, at 104°C (220°F). Average values, from measurements of between 5 and 15 individual samples, are given in Table V. No tensile breaks were noted on microscopic examination after the tests. Apparently, all failures were in the shear mode, except for the conditioned DACP/MY 720 laminates, which failed by thermoplastic deformation at the 104°C test temperature. The horizontal shear strength of the dry DACP/MY 720 samples tested at room temperature was 82.8 MPa (12,000 psi), virtually the same as that of the DDS/ MY 720 control. On conditioning, both materials show much lower strengths, with the DACP/MY 720 deforming at 21.9 MPa (3,170 psi).

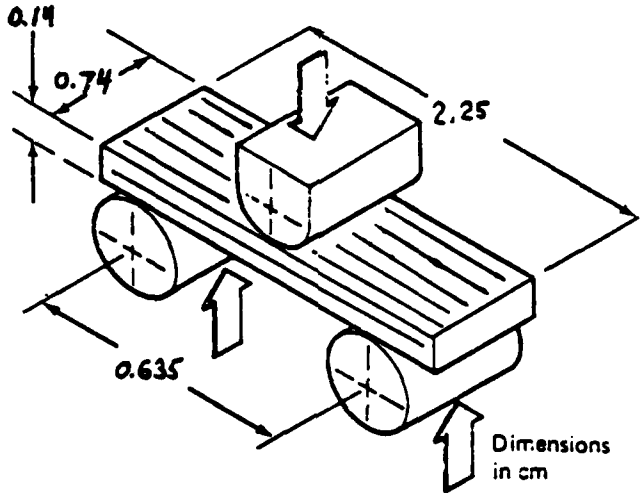


FIGURE 2: SHORT-BEAM-SHEAR TEST SPECIMEN

TABLE V: SHORT-BEAM-SHEAR STRENGTHS OF LAMINATES

	NOT CONDITIONED		HOT/WET CONDITIONED*	
	DACP/MY 720	DDS/MY 720	DACP/MY 720	DDS/MY 720
Sheer Strength (MPa)	82.8	78.5	21.9♦	48.6
Standard Deviation	4.51	3.72	3.63	2.25

* Tests run @ 104°C

♦ Thermoplastic deformation failure

5. Tensile Properties of Laminates

Seven-ply, 0.10 cm-thick laminates were tested in the transverse direction, as shown in Figure 3, at a crosshead speed of 0.05 inch/minute. Hot/wet aged samples were tested rapidly (in about two minutes) to minimize drying, at 104°C (220°F) and at room temperature. Average values, mostly from measurements of six individual samples, are given in Table VI.

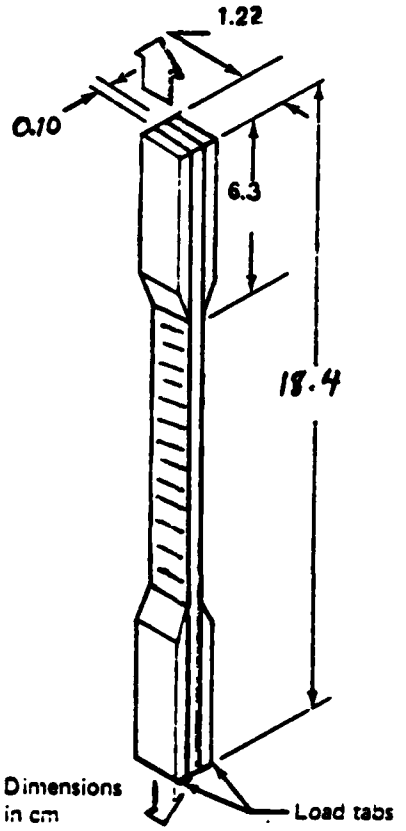


FIGURE 3: TENSILE TEST SPECIMEN

TABLE VI: TENSILE TEST VALUES OF LAMINATES

	NOT CONDITIONED		HOT/WET CONDITIONED		
	DACP/MY 720	DDS/MY 720	DACP/MY 720	DACP/MY 720	DDS/MY 720
Temperature of Test	23°C	23°C	104°C	23°C	104°C
Ultimate Stress (MPa)	29.5	40.9	5.13	10.8	32.6
Standard Deviation	4.37	6.44	-	0.92	3.93
Modulus (MPa)	8,640	10,880	-	1,950	3,573
Standard Deviation	763	1,220	-	166	777
Elongation (%)	0.34	0.37	-	0.11	0.27
Standard Deviation	0.04	0.06	-	0.01	0.03

TABLE VI: DMA VALUES FOR LAMINATES

	NOT CONDITIONED		HOT/WET CONDITIONED	
	DACP/MY 720	DDS/MY 720	DACP/MY 720	DDS/MY 720
Tg (5°/min.)	130°C	261°C	121°C	220, 266°C
Tg (15°/min.)	143°C	265°C	134°C	227°C
E elastic - 25°C	4.0 GPa	4.8 GPa	-	-
E elastic - 100°C	3.0 GPa	-	-	-
E elastic - 120°C	2.1 GPa	-	-	-
E elastic - 140°C	1.0 GPa	4.1 GPa	-	-
E elastic - 160°C	0.47 GPa	-	-	-
E elastic - 200°C	-	4.7 GPa	-	-
E elastic - 230°C	-	4.4 GPa	-	-
E elastic - 240°C	-	4.0 GPa	-	-
E elastic - 260°C	-	2.9 GPa	-	-
E elastic - 280°C	-	2.0 GPa	-	-
tan δ - 25°C	0.022	0.007	0.017	0.005
tan δ - 100°C	0.044	-	0.048	-
tan δ - 120°C	0.122	-	0.196	-
tan δ - 140°C	0.216	0.010	0.299	-
tan δ - 160°C	0.197	-	0.124	-
tan δ - 200°C	-	0.016	-	0.016
tan δ - 230°C	-	0.030	-	0.023
tan δ - 240°C	-	0.046	-	0.025
tan δ - 260°C	-	0.113	-	0.034
tan δ - 280°C	-	0.129	-	0.041

Laminates with DACP/MY 720 matrix resin have a tensile strength of 29.5 MPa (4,280 psi), 72% of the value of the DDS-cured laminate. The modulus was 8,640 MPa (1,254,000 psi), 79% of the control and the elongation was also slightly lower.

DACP/MY 720 laminates conditioned at 49°C and 95% relative humidity (containing an equilibrium amount of 1.1% water) showed a marked drop in tensile properties. The 104°C test temperature seemed to be above the onset of softening, so that tests were also run at ambient temperature (23°C). These values, 10.8 MPa (1560 psi) ultimate stress and 1,950 MPa (283,000 psi) modulus, were also much lower than that of the control.

6. Dynamic Mechanical Analysis of Laminates

The results of dynamic mechanical analysis of dry and of hot/wet aged laminates are shown in Table VII. Despite the drastic reduction in shear and tensile results of aged samples reported above, DMA showed the Tg of the DACP/MY 720 matrix laminate to be only slightly affected by the conditioning; at a heating rate of 5°/minute, dry and wet samples exhibited Tg's of 130° and 121°C, respectively. A 15°/minute rate was also used to reduce the possibility of drying out during the test, and Tg's of 143° and 134°C were found for non-conditioned and conditioned materials, respectively. The change in slope of the tensile modulus curve also decreases about 10°C on conditioning, from 110° to 100°C. Aged DDS/MY 720 matrix laminates exhibited two Tg's; apparently, the lower one for watersaturated resin is followed by a Tg for the dried-out sample. This phenomenon is not observed at the faster heating rate, where only the lower Tg is shown.

CONCLUSIONS

Latent phthalamide hardeners which were synthesized can be made compatible with multifunctional epoxy resins by dissolving the hardener in methanol and mixing with an acetone solution of the epoxy resin. Solvent can be removed by spraying, or by the customary drying of a prepreg.

The phthalamides of MXDAP and DACP are stable with MY 720 for at least 12 months and 6 months respectively. Other phthalamides prepared, DAMP, PACMP and BACP, have insufficient latency in epoxy formulations.

Glass transition temperatures for systems cured at 150°C are about 130°C for MXDAP/MY 720/0510, 145°C for DACP/MY 720 and 130°C for DACP/MY 720 laminate.

Prepreg was prepared from DACP, MY 720 and graphite fiber, and converted to a uniform, well-consolidated laminate at 100 psi and 150°C. At ambient conditions, the laminate has physical properties approching that of a higher-temperature cured DDS/MY 720 laminate. On hot/wet aging, 1.1% water is absorbed and shear and tensile properties are lowered considerably, especially at an elevated test temperature. However, DMA shows that Tg and tensile modulus are only decreased by about 9°C on conditioning at 49°C and 95% relative humidity.

RECOMMENDATIONS

While the feasibility of making a latent, one-component, multifunctional epoxy system curing at 150°C has been demonstrated, work to establish its practicality for composite aircraft repair is still needed. This would entail:

1. Scale-up of the synthesis of the phthalamide hardener and production of prepreg.

2. Investigation of the consolidation and curing of the prepreg using a vacuum-bagging technique and a heating blanket.

3. Testing of repair patches under more realistic conditions, e.g., determining the effect of heat and humidity cycling.

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