

NRL Report 6313

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Filament-Winding Plastics

Part 3 - Some Effects of Curing Agent Structure Upon Epoxide Plastic Physical Properties

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*Organic and Biological Chemistry Branch
Chemistry Division*

September 13, 1965



U.S. NAVAL RESEARCH LABORATORY
Washington, D.C.

PREVIOUS REPORTS IN THIS SERIES

"Part 1 - Molecular Structure and Tensile Properties,"
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"Part 2 - Role of the Resin in Glass-Fiber-Reinforced
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ABSTRACT

Plastics studies at NRL have attempted to relate the molecular structural characteristics of epoxide resins to tensile properties of the plastics obtained, with particular attention devoted to resin systems suitable for filament-winding applications. These materials may find use in the development of deep-diving submersible vehicles. The resins consist of an epoxide component and an amine curing agent, or coreactant, which react to form strong, tough plastics. Attention has been directed mainly toward amine curing agents and the effects of the structures of these components upon the resulting plastic properties.

Recent work has been concerned with finding an amine curing agent which gives optimum properties to the epoxy component. Basic requirements are that it be a liquid or low-melting solid or be soluble in the epoxide and that the reaction rate between the epoxide and the curing agent be slow enough to permit proper winding. Because of their slower reaction rate with epoxides, aromatic amines have been found preferable to aliphatic or alicyclic ones. Except in the case of *m*-aminobenzyl amine, physical properties of the system are not improved by extension of aliphatic side chains from the phenyl ring of the curing agents, and tensile properties of the plastic are diminished as the number of methylene groups increases. Of the combinations studied, one of the most suitable resins for wet winding is the system of *m*-aminobenzyl amine and the diglycidyl ether of Bisphenol A, because of its high elongation. The *cis-trans* bis(2,3-epoxy cyclopentyl) ether/*m*-aminobenzyl amine system is probably the strongest plastic available for wet winding.

PROBLEM STATUS

This is an interim report; work is continuing on the problem.

AUTHORIZATION

NRL Problem R05-24C
Project SPO WW-041

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the notch toughness of a composite produced with this plastic is also considerably greater (5). Part 2 was concerned with the effects of plastic variations upon the burst strengths of internally pressurized bottles (2). It was shown that the average level of burst strength was essentially independent of the plastic properties; however, the reliability of achieving burst within narrow design limits was noticeably determined by the toughness of the resin.

RESIN PROPERTY CONSIDERATIONS

Primary consideration in these studies has been devoted to amine curing agents for the epoxide components, and particularly to those which are suitable for the "wet-winding" process, as distinguished from "pregreg." To be suitable for this process there are several convenient characteristics which a curing agent should possess. It should be a liquid or low-melting solid ($mp < 80^{\circ}C$), or it should be readily soluble in the epoxide component. These characteristics facilitate easy blending of the components in the proper ratios. Also, the rate of reaction between the curing agent and epoxide should be sufficiently slow that ample time is available for the winding process. A number of amines produce good plastics with standard epoxides but have reaction rates which are too great for convenience. The reactions are generally very exothermic; consequently, there is an escalating temperature-rate effect which results in a sudden hardening of the plastic. Cooling procedures can be used to retard this process but may be relatively impractical in some applications.

Some of the newer epoxide components are difficult to utilize because they react slowly with amines and volatilize if curing is attempted at elevated temperatures. In addition to presenting health and fire hazards, this loss of material disturbs the stoichiometric ratios of the components and results in less than optimum plastic properties. This difficulty is encountered more frequently in the cases of alicyclic epoxides than in those of the glycidyl ether type.

IDENTIFICATION OF MATERIALS

Some of the materials used in this investigation were commercially available products, some were synthesized under NRL contract, and others were synthesized locally. Tables 1 and 2 list the materials with identifying symbols, chemical names, and the structural formulas of the major constituents. (In some subsequent parts of this report symbols will be used for brevity, instead of the names of chemical compounds.)

PREPARATION OF MATERIALS

In all cases the plastics were prepared by blending the amine and epoxide components in such ratios that there was a 1:1 equivalence of epoxide functional groups and amine hydrogens. It has been found in the case of m-phenylene diamine that the optimum resin properties are usually obtained when less than the stoichiometric amounts are used. Also, the nature of the cure schedule can affect the properties obtained. However, for the purposes of this study, the conditions were standardized in order to have a common basis of comparison. Since several of the materials were available only in small quantities, it has not been possible to optimize variables of composition and cure.

The solutions of resin and amine were blended, outgassed in a vacuum, poured into molds, and cured. Standard technique was to allow the mixed components to react to "B" stage overnight at $55^{\circ}C$, cure at 80° for 1 hour, and finally, cure at 170° for 3 hours. The molds were made of steel, with typical dumbbell specimens. After removal from the mold,

Table 1
Amine Structures

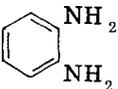
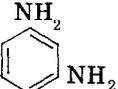
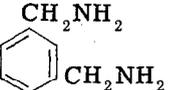
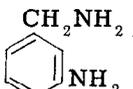
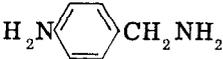
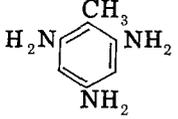
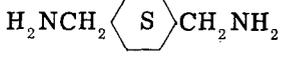
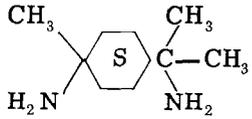
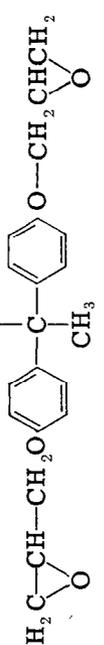
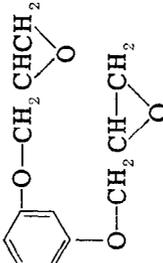
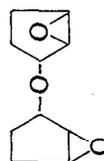
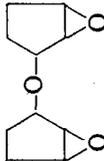
Symbol	Chemical Name	Formula	Source
OPDA	o-Phenylene diamine		Commercial
MPDA	m-Phenylene diamine		Commercial
PPDA	p-Phenylene diamine		Commercial
MXDA	m-Xylylene diamine		Commercial
MABA	m-Aminobenzyl amine		Synthesis (NRL)
PABA	p-Aminobenzyl amine		Synthesis (NRL)
PAPEA	p-Aminophenethyl amine		Synthesis (NRL)
TAT	2,4,6-Triaminotoluene		Synthesis (NRL)
CHMA	1,4-Cyclohexane bis (methylamine)		Commercial
MDA	Menthane diamine		Commercial

Table 2
Epoxide Structures

Symbol	Chemical Name	Formula	Source
DGEA	Diglycidyl ether of Bisphenol A		Commercial
RDGE	Resorcinol diglycidyl ether		Commercial
ERLA-0400	cis-trans Bis(2,3-epoxy cyclopentyl) ether		Contract (Union Carbide)
ERRA-0300	cis & trans Bis(2,3-epoxy cyclopentyl) ether		Contract (Union Carbide)

the specimens were machined to constant thickness, polished, measured, and placed in a standard tensile testing machine. Data were taken for the average values of essentially flawless specimens. (Specimens containing obvious flaws were discarded.)

EFFECTS OF VARIOUS AMINES ON TENSILE PROPERTIES

Ortho/Meta/Para Variations of Phenylene Diamines

Of the three phenylene diamines—ortho, meta, and para—the meta isomer has the lowest melting point (102°, 63°, and 140° respectively) (6). Because of this, its ease of synthesis, and the generally good properties it exhibits, m-phenylene diamine has been extensively used in filament-winding applications. With some blending difficulty, the ortho isomer may also be used. However, the high melting point and relative insolubility of the para isomer at room temperatures severely limit its usefulness in this application.

From a structural standpoint, it would appear that there would be much steric congestion in the vicinity of the amino groups of o-phenylene diamine and that the phenylene group would project to the side of any polymer chains formed. It would not be surprising, therefore, to find that epoxides cured with the ortho isomer would be weaker and less extensible than those cured with the meta or para isomers. If its physical properties would permit, the strongest plastics should result from the para isomer, because of its symmetrical structure and favorable steric situation. The tensile properties of the plastics resulting from reactions of these amines with DGEA are listed in Table 3. The tensile strength and modulus of the ortho isomer are surprisingly high; however, its ultimate elongation is only about one half that of the meta isomer. The net result indicates that extensively crosslinked, three-dimensional structures are obtained with any of the systems; but the ability of the network to become distorted without rupture is more limited in the case of the ortho isomer. Some para structure in the system did not improve the properties of the meta plastic.

Table 3
Tensile Properties and Ortho/Meta/Para Variations
of the Phenylene Diamines with DGEA

Amine	Tensile (psi)	Elongation (%)	Modulus (psi)
OPDA	11,000	4.0	442,000
MPDA	12,800	7.5	463,000
80% MPDA/20% PPDA*	12,200	6.4	430,000

*Above 20%, PPDA precipitates readily from the uncured plastic.

Another indication of meta/para effects was obtained by curing DGEA with the aminobenzyl amines (Table 4). In this case both MABA and PABA have physical properties such that they could be easily incorporated into the epoxide systems (7). This MABA system is one of the most suitable resins for use in wet winding presently available because of its high elongation. The extra elongation results from the ability of the plastic to display an actual yield point in the stress-strain curve, as opposed to the majority of epoxide plastics, which display brittle rupture while the curve is rising sharply. The results obtained with the para isomer are those that would generally be expected from the use of

Table 4
Tensile Properties and Meta/Para Variations
of Aminobenzyl Amines with DGEA

Amine	Tensile (psi)	Elongation (%)	Modulus (psi)
MABA	12,200	10.5	410,000
PABA	13,200	8.1	463,000

a more symmetrical structural unit—higher tensile, higher modulus, and decreased elongation. It may be assumed that the extents of the reactions were about the same in either case and that the density of actual crosslink points was about the same. Consequently, the differences in properties of the two plastics must have resulted from secondary forces which could come into play more freely in the case of the more symmetrical para isomer. Such a secondary force may have been the association of the hydroxyl groups which are generated in the epoxide-amine reaction.

Displacement of Amino Groups from the Ring by Methylene Groups

It is standard practice in polymer chemistry to increase the flexibility of plastics by the introduction of flexible chains within the polymer structures. The increase in elongation obtained with filament-winding resins by the use of this technique is not usually very significant, and an appreciable loss in tensile properties occurs. It was of interest, therefore, to determine the effect upon tensile properties of the filament-winding plastics which occurs when short displacements of the amino groups from the aromatic rings are made. Such displacements eliminate the moderating effect of the ring upon amine reactivities, however, and the resulting compounds tend to behave more like aliphatic amines than aromatic amines in reactions with epoxides. This means that in filament-winding applications the resins generally have to be cooled to avoid rapid reactions and high exotherms. Table 5 lists a number of systems in which the amino groups are progressively displaced from the ring by methylene groups and the effects upon tensile properties which occur.

Table 5
Tensile Properties and the Displacement of Amino Groups
from the Aromatic Ring

Epoxide	Amine	Tensile (psi)	Elongation (%)	Modulus (psi)
DGEA	MPDA	12,800	7.5	463,000
DGEA	MABA	12,200	10.5	410,000
DGEA	PABA	13,200	8.1	460,000
DGEA	MXDA	10,600	6.7	436,000
DGEA	PAPEA	9,900	7.6	340,000
ERLA 0400	MPDA	17,600	4.6	684,000
ERLA 0400	MABA	20,100	5.1	766,000
ERLA 0400	MXDA	15,600	6.3	552,000
ERLA 0400	PAPEA	14,300	3.1	658,000
ERRA 0300	MPDA	18,400	5.0	700,000
ERRA 0300	MABA	17,500	5.3	654,000
ERRA 0300	MXDA	14,500	4.6	572,000
RDGE	MPDA	16,900	8.1	666,000
RDGE	MXDA	15,300	6.1	665,000

It can be seen from these results that, generally, the tensile properties of the plastics were diminished as the number of methylene groups increased. One outstanding exception to this occurs in the first displacement, that is, the conversion from *m*-phenylene diamine to *m*-aminobenzyl amine. As already noted, in the DGEA system there is a considerable increase in elongation. The 0400 system is particularly notable, since there were increases in tensile, modulus, and elongation all occurring at once. The 0400/MABA system is the only one evaluated which exhibited a tensile strength in excess of 20,000 psi. This, combined with a modulus of 766,000 psi, probably means that this is the strongest plastic available for use in the wet-winding process.

In each case in which two methylene groups are inserted between the ring and the amino groups, there is a substantial decrease in tensile strength and in modulus, and usually in elongation also. Loss in tensile strength is particularly evident in the PAPEA systems, in which one of the amino groups is displaced from the ring by two methylene groups. This variation in structure has, in effect, reduced the properties of the plastics to the levels obtained with the purely aliphatic amines (8). It is also evident that the shorter coupled epoxides—ERLA 0400, ERRA 0300, and RDGE—produce appreciably stronger plastics with any given amine curing agent than does the DGEA system.

Cyclohexyl Amine Systems

Since the benzene nucleus is commonly taken to be a flat, rigid ring, it was thought that a comparison of results would be interesting if the benzene ring were replaced with a puckered cyclohexyl ring. Amines of types exactly analogous to the phenylene systems previously considered were not available; however, two commercial amines of close similarity were on hand. These two, 1,4-cyclohexane bis(methylamine) and menthane diamine, are both liquids which are suitable for filament winding. The former contains two aliphatic amine groups attached to terminal carbon atoms; consequently, it has a reaction behavior much like a straight-chain aliphatic amine. Pot life is short, and care must be taken to prevent a sudden exothermic reaction from occurring. On the other hand, MDA has two amino groups which are connected to tertiary carbon atoms. The reactivities of these amino groups are substantially reduced, and sudden exothermic reactions occur less readily. The reaction characteristics, and particularly the rates, are more typical of the aromatic than the aliphatic amines. The tensile properties obtained with these amines are shown in Table 6.

Table 6
Tensile Properties of DGEA Plastics
Cured with Alicyclic Amines

Amine	Tensile (psi)	Elongation (%)	Modulus (psi)
CHMA	6,900	3.2	360,000
MDA	10,400	3.6	448,000

The most noteworthy effect of this variation is that the systems are low-elongation plastics. This suggests that the symmetry and rigidity of the phenylene ring system play a substantial role in determining the properties of a plastic produced from DGEA. However, it is interesting that the slower reacting MDA produced a plastic which was considerably stronger than that produced by the more rapid CHMA. It is probable that the slower system had a better opportunity to develop a tight network before the structure became immobile.

EFFECTS OF AMINE STRUCTURE ON COMPRESSIVE PROPERTIES

The compressive properties of several of the systems previously discussed, plus those of DGEA and the highly functional 2,4,6-triamino toluene (TAT), are given in Table 7. In general, it can be seen that the compressive properties of these systems parallel the tensile properties. The "weakest" system was produced with CHMA, and the "strongest" systems result from the use of the dicyclopentyl epoxides. The functionality of TAT is 50% greater than that of MPDA, and this higher functionality resulted in a moderate increase in the compressive properties. The highest compressive modulus was obtained with the ERLA 0400/MABA system, but the other properties were slightly lower than for the MPDA system. All of the properties of the MDA system are considerably higher than those of the CHMA system, and MDA compares favorably with MPDA in compression.

Table 7
Compressive Properties of Several Amine-Epoxy Systems

System	Ultimate Compressive Stress (psi)	Yield Stress (psi)	Ultimate Strain (%)	Compressive Modulus (psi)
DGEA/MPDA	43,200	18,620	44.2	300,600
DGEA/TAT	46,900	20,960	41.6	324,280
DGEA/CHMA	28,900	15,000	40.8	256,000
DGEA/MDA	40,200	19,200	45.9	309,400
ERLA 0400/MPDA	56,900	28,600	46.5	415,000
ERLA 0400/MABA	51,000	26,000	46.1	425,000

CONCLUSIONS

It is apparent from these results that the physical properties of filament-winding plastics are not readily improved by the extension of aliphatic side chains from the phenyl ring of amine curing agents. The one exception is *m*-aminobenzyl amine, in which an amino group is displaced by only one methylene group. Considerable effort has gone into the use of commercial flexibilizers for epoxides, and results in these cases have been essentially the same—small gains in flexibility were accompanied by rather large losses in tensile strength and modulus (8).

Substantial improvements in both tensile and compressive properties are attained by the use of shorter coupled epoxides of the ERLA 0400 type. If the practical difficulties encountered in winding operations with these materials can be overcome without loss in their superior properties, then improved filament-winding systems should result. Of particular concern, at present, is the volatility of these epoxides during the curing cycles.

Aliphatic and alicyclic curing agents behave similarly in the epoxide systems, and in general, they do not result in improvements over aromatic systems of the same functionality. Currently, work is in progress to produce epoxy plastics with considerably greater elongation without excessive loss of tensile and compressive properties.

ACKNOWLEDGMENT

A.G. Sands and R.C. Clark prepared and tested the tensile and compressive samples.

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14. KEY WORDS	LINK A		LINK B		LINK C	
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Plastics Filament-winding plastics Epoxide resins Plastic properties Aromatic amines Curing agents Wet winding						

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