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EPOXY RESIN DEVELOPMENT FOR COMPOSITE FIELD REPAIR.(U)
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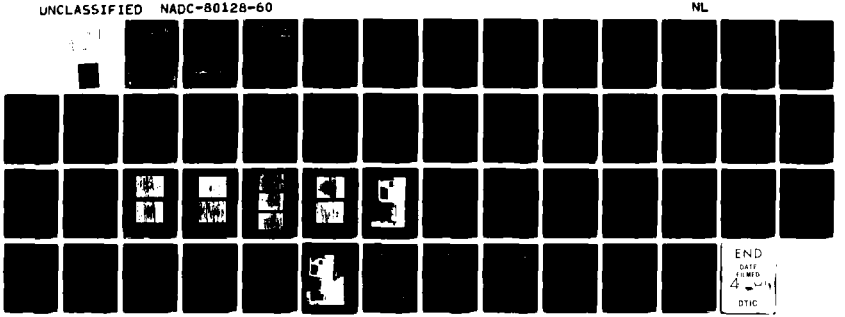

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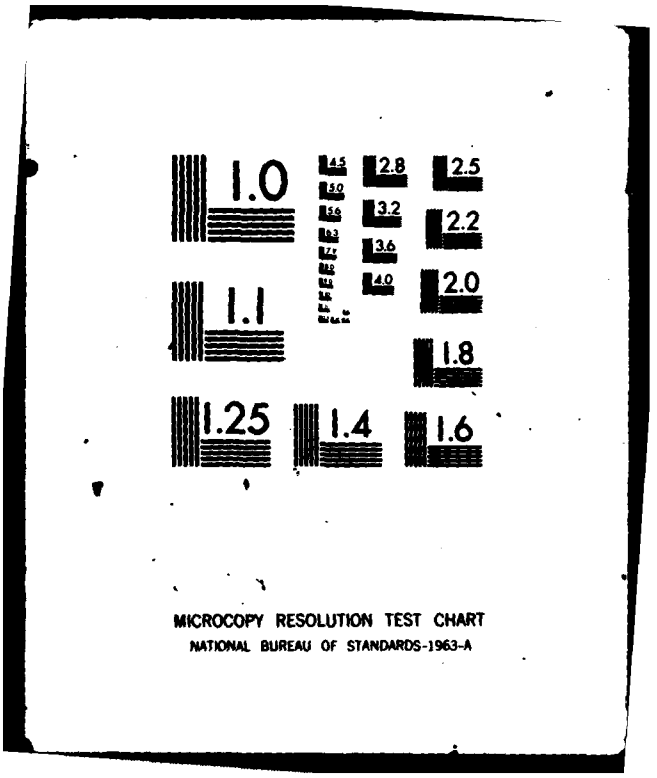
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EPOXY RESIN DEVELOPMENT FOR COMPOSITE FIELD REPAIR

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

An evaluation was conducted to determine the optimum polymer matrix material to be used for composite repair. The material must have room temperature storage and adequate strength at elevated temperatures. Mechanical property degradation due to moisture absorption was also a consideration. Differential scanning calorimetry and dynamic mechanical analyses were performed on the candidate resin systems. Short beam shear and flexural strengths were examined for each system. The most promising resin systems

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proved to be stable at room temperature in the B-stage condition. These systems incorporated a sterically hindered amine hardener that provided latency. The effects of cloth style on void content and shear properties were also examined.

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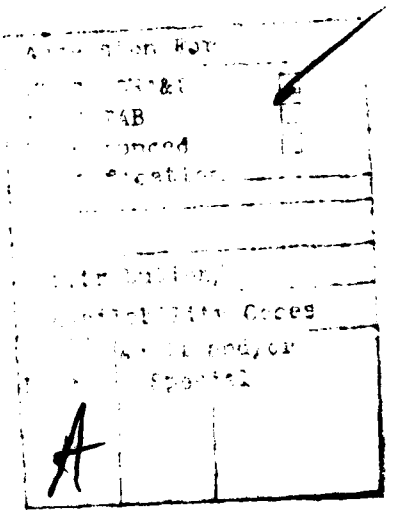

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INTRODUCTION

The use of graphite/epoxy as an aero-structural material has increased greatly over the last few years. The Navy, with the F-18 and AV-8B aircraft, uses graphite/epoxy to such an extent that it has become necessary to consider effective repair of these structures. Graphite/epoxy structures are usually fabricated by the application of heat and pressure in an autoclave. Repairs made with the use of an autoclave, where such procedures are possible, do not present any serious technical problems. Repairs made where an autoclave is not readily available, such as on an aircraft carrier, require materials that can be cured with vacuum pressure only to form high integrity laminates.

Storage of repair materials may also be a problem. For example, most resin systems when mixed must be used immediately or else, when B-staged, must be stored at low temperature (0°F, -18°C).

An ideal resin system for repair of graphite/epoxy structures should have the following characteristics:

(1) Ambient or low temperature cure;

(2) Low viscosity;

(3) Room temperature storage in the B-stage condition;

(4) Short time cure (1 hour or less);

(5) Elevated temperature and moisture resistance; and

(6) Vacuum pressure during cure.

Item (1) eliminates the problems associated with conventional heat sources such as heating blankets and hot air guns. Current technology, though, does not permit elevated temperature performance of room temperature cured epoxy resins. Therefore, a moderate temperature cure (200°F to 300°F; 93°C to 149°C) with glass transitions at or above the cure temperature is the next best approach.

Low viscosity is preferred to enable the resin to flow in between the fibers. Room temperature storage, either unmixed or mixed (in the B-stage), is a necessity for a repair system on the basis of supply and storage at the field level. A short time cure of 1 hour or less decreases the total repair time which decreases the down-time of the aircraft.

Elevated temperature and moisture resistance is best achieved by having a higher glass transistion temperature.

Current composite repair techniques include Grumman's F-14 boron/epoxy repair kit (Ref. D). This kit is intended for repair of the horizontal stabilizer which is constructed of titanium and boron/epoxy face sheets over an aluminum honeycomb core. Titanium foil is used with a film adhesive which must be stored at 0°F, -18°C.

Current techniques with graphite/epoxy includes precured patches that are adhesively bonded to the composite structure. The patches are precured and therefore limited to flat areas of the structure. One part of the adhesive system has a room temperature shelf life of only 3 months. This is clearly

unacceptable for field level repair where the distribution of materials can be very time-consuming.

Bolted or mechanically fastened repairs have been successfully demonstrated by McDonnell Douglas for graphite/epoxy laminates 3/16 and 1/2 inch thick with through-the-thickness hole damage (Ref. E). These repairs were developed for application on fuel cell composite wing surfaces using titanium alloy patches and backing plates. The problems with this type of repair is the damage to the composite caused by the bolt hole drilling and the aerodynamic incompatibility of the titanium patch. Also, a bolted repair system cannot be used on honeycomb structure.

The optimum situation for the field repair of composite structures is to use both methods of repair, bonded or bolted, depending on the location of the damage. Honeycomb, for example, requires bonded repair methods while thick, flat, non-honeycomb structures are suited to bolted repair.

These considerations led to the use of a resin system that is stable in the B-stage at room temperature. It consists of a difunctional epoxide resin with a tetrafunctional amine hardener. This system was developed by J. Rinde at the Lawrence Livermore Labs. (Ref. A). The resin is a diglycidyl ether of bisphenol-A. Its advantages consist of high purity and low viscosity. Other resins, such as polyglycidyl ether of orthocresol-formaldehyde novolac and resorcinol diglycidyl ether were used either as an additive or a substitute.

The hardeners used were 2, 5-dimethyl 2, 5-hexane diamine and menthane diamine. Both of these tetrafunctional hardeners are sterically hindered to prevent or delay the secondary amine hydrogen reaction.

Other systems and modifications are presently being explored. Attempts are being made to decrease the time and temperature of the cure and extend the storage life.

DER 332 - Diglycidyl ether of bisphenol - A

RDGE - Resorcinol diglycidyl ether

DMHDA - 2, 5-dimethyl 2, 5-hexane diamine

ECN 1299 - Polyglycidyl ether of orthocresol-formaldehyde novolac

NC 513 - Aromatic ether (epoxy reactive diluent)

MD - Menthane diamine

EXPERIMENTAL PROCEDURE

This investigation was conducted in two phases. Phase I included epoxy resin selection and evaluation by mechanical properties of a cured graphite cloth laminate. Phase II consisted of evaluation of various epoxy resin systems through thermal analysis techniques, specifically DSC (differential scanning calorimetry) and DMA (dynamic mechanical analysis). Also included in Phase II was a check on laminate quality using optical microscopy. Initially, all evaluation was based on mechanical properties. The aquisition of thermal analysis equipment added a second dimension to the resin evaluation.

Phase I

An industry and literature search led to the selection of candidate resin systems. Laminates were fabricated using these resin systems in conjunction with Hercules A-370-8H and A-370-5H graphite cloth. Their layup was a 9" x 9", quasi-isotropic type using $(\pm 45, 0, 90)_s$. The candidate resin systems were mixed, when necessary, and applied to the cloth. The resin and the curing agent was reacted at a stoichiometric ratio which was calcuated as follows:

$$\text{phr} = \frac{(A_H)\ (100)}{R_{EQ}}$$

phr = parts by weight of amine per 100 parts resin

A_H = Amine Hydrogen Equivalent Weight

R_{EQ} = Epoxide Equivalent Weight of Resin

The resin was distributed throughout the laminate by the use of a rolling pin. Excess resin was removed by vacuum bleeding before cure. Room temperature cures were run using a mylar bag with a 10 oz. fiberglass tooling cloth as a bleeder material. Higher temperature cures were run using vacuum pressure only. All laminates, with one exception, which employed either DMHDA or MD as the hardener were cured at 260°F for one hour. There were no elevated temperature post-cures.

Test specimens were cut from the laminates to evaluate the following mechanical properties:

(1) Short beam shear strength at room temperature, 220°F (104°C), and 220°F + moisture.

(2) Flexure strength at room temperature, 220°F, and 220°F + moisture.

All mechanical testing was done on an Instron test machine. Elevated temperature short beam shear tests were usually performed first to determine if further testing was justified. Data was collected and reduced for each laminate. The moisture content was obtained by placing test specimens in a humidity chamber set at 100% relative humidity, 140°F (60°C), for a minimum of 96 hours.

Phase II

Thermal analyses were conducted using DSC and DMA on cured and uncured resin systems. The cured laminates were sectioned, mounted, and polished to examine their cross-sections. A Buehler "Minimet" Automatic Polisher was used to complete the polishing.

Photomicrographs were taken of the near surface and center cross-sections to determine void content and resin distribution.

RESULTS

A list of all the resin systems evaluated is given in Table I. Shear and flexure data are presented in Table II for the systems that were initially screened. Tables III and IV show shear and flexure data for the more promising systems. The testing was done at room temperature, 220°F (104°C), and 220°F with moisture.

The mechanical properties improved as the number of voids in the laminates decreased. Figures 1 through 4 show photomicrographs (at 70X) of the laminates. Laminate quality improved with fabrication experience as shown by the decrease in the size and number of voids.

The tensile lap shear data for Arnox 3110 is presented in Table V. Arnox 3110 was evaluated in two ways, as an adhesive and as a laminating resin.

The percent decreases in shear and flexure strength from room temperature values are presented in Tables VI and VII. Table VIII shows the moisture absorption data obtained for the laminates. It can be seen that the specific type of weave, 8-harness or 5-harness, affected the moisture content.

Thermal analyses were conducted using a DuPont 1090 thermal analyser with DMA and DSC modules. Figures 6 and 7 show DSC plots of 332/DMHDA in the freshly mixed and B-staged conditions. Figures 8 and 9 show similar DSC plots for the same system with the ECN-1299 additive. Figures 10, 11, and 12 show the DSC plots of RDGE/MD with respect to time. Figure 10 depicts material in the freshly mixed condition; figure 11, in the B-stage (4 days later); and figure 12, an additional day at room temperature. Figures 13 and 14 show DSC plots of RDGE/DMHDA in the freshly mixed and B-staged conditions. Figures 15 and 16 show the same type of DSC plots for 332/MD. Figure 17 shows a DSC plot for Arnox 3110. The Arnox 3110 resin system is premixed and stable at room temperature in this condition.

The peak exotherms and melt endotherms are summarized in Table IX for the DSC plots.

Dynamic mechanical analyses (Fig. 18) were performed on cured laminate sections. Due to the dimensional restrictions on the DMA sample, the laminate thickness became the sample width by taking a very thin slice of the laminate. This meant there were no continuous fibers between supports. Thus, the matrix was evaluated with as little contribution from the fibers as possible. The DMA plots of 332/DMHDA 8-harness and 5-harness cloth laminates are shown in Figures 19 and 20. There were no significant differences between the 8-harness and 5-harness cloths. Figure 21 shows the DMA plot of 332/DMHDA with the ECN-1299 additive. The frequency and damping plots (modulus and tan δ) are shifted to the right which shows an increase in the glass transition temperature. Figures 22 and 23 show DMA plots of RDGE/DMHDA and Arnox 3110. The RDGE/DMHDA has a relatively low Tg. All of the Tg's and tan δ peaks are summarized in Table X.

DISCUSSION

Of the resin systems investigated the room temperature curing systems like Dow's D.E.R. 332/D.E.H.20 and CIBA's 6005/0500/HY956 flowed when tested at elevated temperature (220°F) and thus were not further evaluated (see Tables I and II). Shell's Epon 828/Z system, which was cured at 300°F, yielded acceptable

mechanical properties but was difficult to work with due to the danger of the Z catalyst. This would be especially true in a field or intermediate-level maintenance type of environment. The Apco resin system was much too viscous to be used as a laminating resin. Epoxylite 8130 did not have adequate strength even at room temperature. The Imidazoles were examined very briefly and show potential as adhesive-type curing agents. Because they were in the form of a powder, mixing was a problem. The Arnox 3110 resin did not have the mechanical strength either as a laminating resin or as an adhesive. (See Tables III, IV, and V). This material was fabricated early on in the program and evidenced voids in the cured resin. It is planned to obtain a second batch of this material to substantiate initial mechanical property results.

The 332/DMHDA system with and without modification showed potential. These resin systems were more thoroughly investigated with hot-wet mechanical properties and thermal analyses. The RDGE/MD, 332/MD, and RDGE/DMHDA systems were also examined with thermal analysis (specifically DSC).

Important considerations for any potential repair system are the matrix dominated mechanical properties. One such property used for material characterization is short beam shear strength. Short beam shear properties are an excellent measure of overall laminate quality. They are a resin-dependent property and therefore very sensitive to temperature. Adequate elevated temperature shear properties require good fiber - resin adhesion and higher glass-transition temperature than the test temperature desired.

Good fiber-resin adhesion can be achieved or improved by decreasing the void content in the laminate. This was attempted in two ways. First, a 5-harness satin graphite cloth was used which is a more open weave of cloth than the 8-harness satin. (See Figure 1). Second, a reactive diluent, Cardolite NC-513 was used. This low viscosity fluid has one epoxide group per molecule and, therefore, combines chemically in any cured epoxy resin formulation. As little as 10% by weight reduces the viscosity of a liquid epoxy by 70%. Lower viscosity improved wetting and penetration into the laminate and also reduced air entrapment. Photomicrographs showed that the number and size of voids in the cross-section decreased with the addition of Cardolite NC-513. (See Figures 1 and 2). This addition, however, has also decreased the elevated temperature strength of the resin. This reduction in strength is due to the plasticizing nature of the additive. The failure mode changed at this point due to the influence of the additive. Instead of a typical shearing type of failure, the sample plastically deformed. This decreases the overall load bearing capability of the laminate by making it easier for the resin to flow.

The mechanical properties that were evaluated for the 332/DMHDA system seem to depend on the processing conditions. Because the graphite cloth was impregnated manually, penetration of the resin into the cross-weave of the cloth has not been optimized. It is believed that machine prepregging of this system would improve the mechanical properties. Efforts are presently underway to have this attempted.

The photomicrographs show the 5-harness cloth to have better resin penetration than the 8-harness (see Figure 1). The addition of NC-513 to 332/DMHDA improved penetration and flow of the resin but also decreased the mechanical strength of the composite. Both shear and flexure values decreased much more with temperature than the other resin systems (see Tables VI and VII). The

ECN 1299 additive did not show much of an effect with respect to the void content. The processing (hand laminating procedure) greatly improved with experience (see Figure 3). Consequently, the laminates fabricated toward the latter part of this investigation contained less voids and were of a higher quality than the laminates fabricated early in the study. Laminates using Arnox 3110 contained a higher void content than laminates of the other resin systems.

The addition of CIBA-Geigy's ECN-1299 to the 332/DMHDA system was made to improve the elevated temperature performance. ECN-1299 is a polyglycidyl ether of orthocresol-formaldehyde novolac with an epoxide equivalent weight of 225. It requires minimum chain extension and crosslinking reactions to reach a high molecular weight and crosslink density necessary for elevated temperature performance. Short beam shear and flexure data do not show any significant improvement of elevated temperature (See Tables III and IV). Laminate quality of 332/1299/DMHDA, as shown in Figure 3, improved to the extent that voids were no longer a major problem. As shown in Table VIII, the decrease in short beam shear strength from room temperature to 220°F (104°C) and 220°F plus moisture was about the same or less without the ECN-1299 additive. The same behavior is noted in flexure strength in Table IX.

The mechanical properties of the Arnox system did not decrease as much as the other resin systems with elevated temperatures and moisture (see Tables IV and VII). This system has relatively low strength to begin with (at room temperature) as is shown in Tables III and IV. Adhesive lap shear data using graphite/epoxy coupons also showed that the system has low strength (see Table V).

Moisture absorption data is shown in Table VIII. The 8-harness laminates absorbed twice as much moisture as the 5-harness laminates with the same resin system. In general, the 5-harness laminates had less voids (due to better resin penetration) than the tighter weave 8-harness. A greater void content provides a greater volume for absorbed moisture. The NC-513 additive seemed to cause an increase in moisture content over the base system. The Arnox 3110 laminate contained the highest void content of all (see Figure 4) and consequently absorbed the greatest amount of moisture.

A higher temperature cure of 300°F was attempted with the 332/1299/DMHDA system. No significant increase in mechanical properties was noted. There was no improvement even at elevated temperature. This laminate contained voids which could have adversely affected the mechanical properties.

The differential scanning calorimetry apparatus is shown in Figure 5. The 332/DMHDA system shows two exotherms when examined in the freshly mixed condition (see Figure 6). These exotherms correspond to the primary and secondary hydrogens on the amine groups in the hardener (2, 5-dimethyl, 2,5-hexane diamined). As shown in Figure 7 with the B-staged system, only one broad exotherm remains along with an endotherm. The single, broad exotherm represents the reaction of the secondary amine hydrogen and the endotherm represents the melt of the uncrosslinked polymer material. This same type of behavior is noted when ECN 1299 is added to the 332/DMHDA system. (See Figures 8 and 9). In this case, though, the melt endotherm is not quite as deep.

The RDGE/MD seemed to cure at room temperature with time (see Figures 10 and 11). The curing agent, menthane diamine or 1, 8-diamino-p-menthane did not pro-

vide the latency that was expected. As shown in Figure 12, the exotherm diminishes completely with an additional day at room temperature. No melt endotherm was observed with RDGE/MD after storing at room temperature in the B-stage. The RDGE/DMHDA did show a slight melt endotherm and latency (see Figures 13 and 14). With additional time at room temperature the melt endotherm decreased and the resin system approached the fully cured condition. The 332/MD system did not cure at room temperature. It remained stable with a melt endotherm and a curing exotherm (see Figures 15 and 16). Arnox 3110, with the advantages of room temperature stability in the mixed condition and fast cure, has a sharp exotherm at about 100°C (212°F) (see Figure 17).

With the exception of Arnox 3110 all of the systems using the resins, DER 332, RDGE, and ECN-1299 and the hardeners DMHDA and MD showed two DSC exotherms when examined in the freshly mixed condition.

The RDGE/MD resin system cured at room temperature as is shown by the absence of a curing exotherm after B-stating. The 332/DMHDA, 332/1299/DMHDA, and 332/MD resin systems did not advance to cure at room temperature. These systems melted, as shown by the endotherms (see Table IX), and cured as shown by the exotherms. They did this after being stored at room temperature in the B-stage. During this test all of the resin systems were left exposed to a laboratory environment. No efforts were taken to prevent moisture contamination which, to the extent it occurs, advances the cure. Considering these points, the above resin systems show promise as future composite repair materials. The dynamic mechanical analysis apparatus is shown in Figure 18. The dynamic mechanical analyzer yielded measurements of the glass transitions and tan δ peaks. The DMA plots show the resonant frequency and damping (energy dissipation) as a function of the sample temperature. The resonant frequency is directly proportional to the elastic modulus. The damping or energy dissipation corresponds to tan δ values. The DMA plot for 332/DMHDA, 8-harness fabric is shown in Figure 19. The dynamic glass transition and tan δ peak can be seen as 133°C and 150°C respectively. The same Tg and tan δ peak was found for the 5-harness fabric (332/DMHDA). (See Figure 20). Therefore, the type of weave of reinforcing cloth has no effect (as was expected) on the frequency and damping response of the sample. The addition of ENC 1299 was shown to increase the Tg and tan δ peak (see Figure 21). The drop-off in the frequency (modulus) curve is more sudden. This could mean that the modulus is less affected by temperature until the Tg is reached. Once the Tg is reached, a sharp transition occurs. The advantage to this type of behavior (as opposed to a more gradual change in slope of the curve) is a higher use-temperature.

RDGE/DMHDA has shown stability in the B-stage at room temperature. The Tg and tan δ peak for this material occur at a lower temperature than the other systems (see Figure 22). The plot for Arnox 3110, 8-harness fabric, Figure 23, gradually changes slope with a lesser decrease in frequency than the other systems. Table X lists all of the glass transition and tan δ peak temperatures. The Tg was taken as the intersection of the two tangent lines drawn to fit the straight line portions of the curve. This glass transition temperature is often taken as the use or service temperature. The tan δ peak, which is 20° to 30°C higher, is referred to as the dynamic Tg. Because crosslinking of the polymer restricts the mobility of the polymer segments, higher dynamic Tg's are associated with more efficient cross-linking.

CONCLUSIONS

1. The most promising resin systems based on the ground rules of short time, low temperature, vacuum pressure cure and long shelf life at room temperature in the B-stage were:

   (1) 332/DMHDA

   (2) 332/1299/DMHDA

   (3) RDGE/DMHDA

   (4) 332/MD

2. The ECN-1299 additive increased the glass transition temperature of the 332/DMDHA resin system.

3. There were less voids in the laminates with the 5-harness graphite cloth than with the 8-harness cloth.

4. The NC-513 additive lowered the viscosity of the 332/DMHDA resin system. This addition also caused a marked decrease in the elevated temperature mechanical properties.

5. The Arnox 3110 resin system evidenced low mechanical properties.

6. Dynamic mechanical analysis indicated glass transition temperatures near or above the cure temperatures.

RECOMMENDATIONS

Additional efforts in this area should be directed towards increasing the storage time of the B-staged resin. The advancement of the system should be studied as a function of storage time and environment. Moisture is thought to cause crosslinking in the system by allowing the secondary amine hydrogens to react. This effect along with how much flow occurs after time in the B-stage should be examined. During the initial part of this work, all resin formulations were mixed stoichiometrically. Varying the amounts of hardener above and below this point could improve the latency and mechanical properties, especially at elevated temperature.

Menthane diamine with more steric hindrance could increase the stability of the resin system and thus offer a longer storage time in the B-stage.

Small-scale repair simulations using the above mentioned materials with adhesives, if needed, are a natural follow-up to this work. A compatible adhesive must be found in this case with similar storage capabilities

A C K N O W L E D G E M E N T S

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TABLE I

RESIN SYSTEMS EVALUATED

RESIN	ADDITIVES	HARDENER	RESIN MANUFACTURER
Epon 828	---	Z	Shell
Epon 815	---	Z	Shell
Epoxylite 8130	---	8130	Epoxylite Inc.
Apco 5393	---	5393	Applied Plastics Inc.
Der 324	---	DEH20	Dow
Araldite 6005	Araldite 0500	HY956	CIBA-Geigy
Der 332	---	DMHDA	Dow
Der 332	---	BTDA/MA	Dow
Der 332	---	MI-1	Dow
Der 332	Cardolite NC-513	DMHDA	Dow
Der 332	ECN 1299	DMHDA	Dow
Der 332	---	2PZ-OK	Dow
Der 332	---	2PZ-OK	Dow
Der 332	---	2P4MHZ	Dow
Arnox 3110	---	---	General Electric
RDGE	---	Menthane Diamine	DuPont
Der 332	---	Menthane Diamine	Dow
RDGE	---	DMHDA	DuPont

TABLE II

SHORT BEAM SHEAR STRENGTH (PSI) OF INITIALLY SCREENED SYSTEMS

RESIN SYSTEM	ADDITIVE	ROOM TEMPERATURE	220°F (104°C)
Epon 828/Z	---	6531	---
Epon 815/Z	---	6663	1117
Der 324/DEH 20	---	3516	794
CIBA 6005/HY956	CIBA 0500	5247	1717
Der 332/DMHDA	---	5884	4266
Der 332/MI-1	---	4323	1187
Der 332/DMHDA	NC-513	6358	3587
Der 332/DMHDA	ECN 1299	6185	4199
Arnox 3110	---	4352	3854

TABLE III

SHORT BEAM SHEAR STRENGTH (PSI) AT ROOM TEMPERATURE 220°F (104°C), AND 220°F + MOISTURE OF CANDIDATE SYSTEMS

RESIN SYSTEM	CLOTH TYPE	ROOM TEMPERATURE	220°F	220°F + MOISTURE
Der 332/DMHDA	8-Harness	6424	4511	3839
Der 332/DMHDA	5-Harness	6772	4982	4648
Der 332/NC-513/DMHDA	8-Harness	6358	3587	1479
Der 332/ECN 1299/DMHDA	8-Harness	6185	4199	3070
Der 332/ECN 1299/DMHDA	5-Harness	7363	5026	4369
Arnox 3110	8-Harness	4352	3854	3423

TABLE IV

FLEXURE STRENGTH (PSI) AT ROOM TEMPERATURE, 220°F (104°C), AND 220°F + MOISTURE OF CANDIDATE SYSTEMS

RESIN SYSTEMS	CLOTH TYPE	ROOM TEMPERATURE	220°F	220°F + MOISTURE
Der 332/DMHDA	8-Harness	67,244	52,020	47,047
Der 322/DMHDA	5-Harness	83,312	55,027	41,068
Der 322/NC-513/DMHDA	8-Harness	79,325	31,623	14,114
Der 322/ECN 1299/DMHDA	8-Harness	74,011	49,735	29,197
Der 322/ECN 1299/DMHDA	5-Harness	73,863	44,162	41,606
Arnox 3110	8-Harness	54,233	38,855	31,529

TABLE V

ARNOX 3110 LAP SHEAR STRENGTH

TYPE OF COUPONS	TENSILE LAP SHEAR STRENGTH (PSI)
Graphite/Epoxy	939
	763
	882
	961
	643

AVERAGE = 837.6

TABLE VI

PERCENT CHANGE IN SHORT BEAM SHEAR STRENGTH

RESIN SYSTEM	CLOTH TYPE	R.T.→220°F	R.T.→220°F + H_2O
332/DMHDA	8H	-29.7%	-40.2%
332/DMHDA	5H	-35.5%	-37.3%
332/513/DMHDA	8H	-43.5%	-76.7%
332/1299/DMHDA	8H	-32.1%	-50.3%
332/1299/DMHDA	5H	-31.7%	-46.0%
Arnox 3110	8H	-11%	-21.3%

TABLE VII

PERCENT CHANGE IN FLEXURE STRENGTH

RESIN SYSTEM	CLOTH TYPE	R.T.→220°F	R.T.→220°F + H_2O
332/DMHDA	8H	-22.6%	-30.0%
332/DMHDA	5H	-20.0%	-43.6%
332/513/DMHDA	8H	-60.1%	-82.2%
332/1299/DMHDA	8H	-32.8%	-60.5%
332/1299/DMHDA	5H	-40.2%	-43.6%
Arnox 3110	8H	-28.3%	-41.8%

TABLE VIII

MOISTURE ABSORPTION DATA

RESIN FORMULATION	CLOTH TYPE	% INCREASE	TIME (DAYS)
332/DMHDA	8H	.83	7
332/DMHDA	5H	.31	7
332/1299/DMHDA	8H	1.06	7
332/1299/DMHDA	5H	.48	7
332/513/DMHDA	8H	1.54	8
Arnox 3110 A	8H	1.64	8

TABLE IX

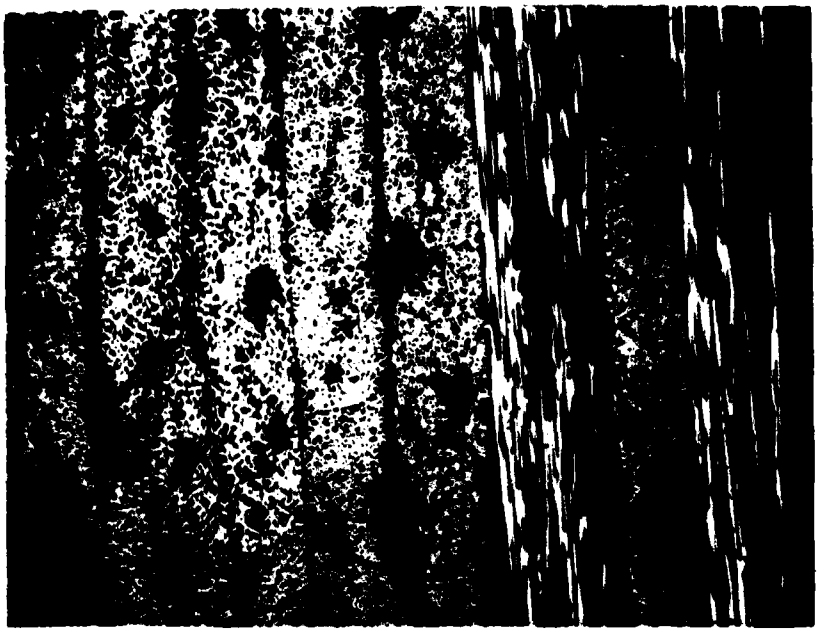
RESIN FORMULATIONS EVALUATED BY DIFFERENTIAL SCANNING CALORIMETRY

FORMULATION	PEAK EXOTHERM TEMPERATURE (°C)	MELT ENDOTHERM (°C)
332/DMHDA (Freshly mixed)	104,147	
332/1299/DMHDA (Freshly mixed)	100,144	
RDGE/MD (Freshly mixed)	87,134	
RDGE/DMHDA (Freshly mixed)	83,123	
332/MD (Freshly mixed)	109,161	
332/DMHDA (B-stage)	133	50
332/1299/DMHDA (B-stage)	138	52
RDGE/MD (B-stage)	---	--
RDGE/DMHDA (B-stage)	114	60
332/MD (B-stage)	154	42

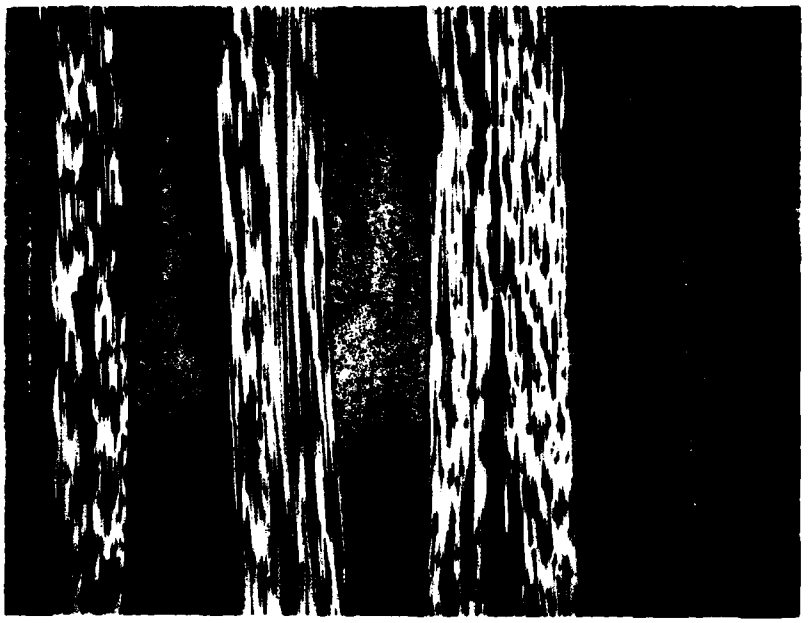
TABLE X

RESIN FORMULATIONS EVALUATED BY DYNAMIC MECHANICAL ANALYSIS

FORMULATION	T_g(°C)	TAN δ PEAK(°C)
332/DMDHA, 8H	133	150
332/DMHDA, 5H	133	150
332/1299/DMHDA, 5H	144	160
Arnox 3110, 8H	140	171
RDGE/DMHDA, 5H	106	124

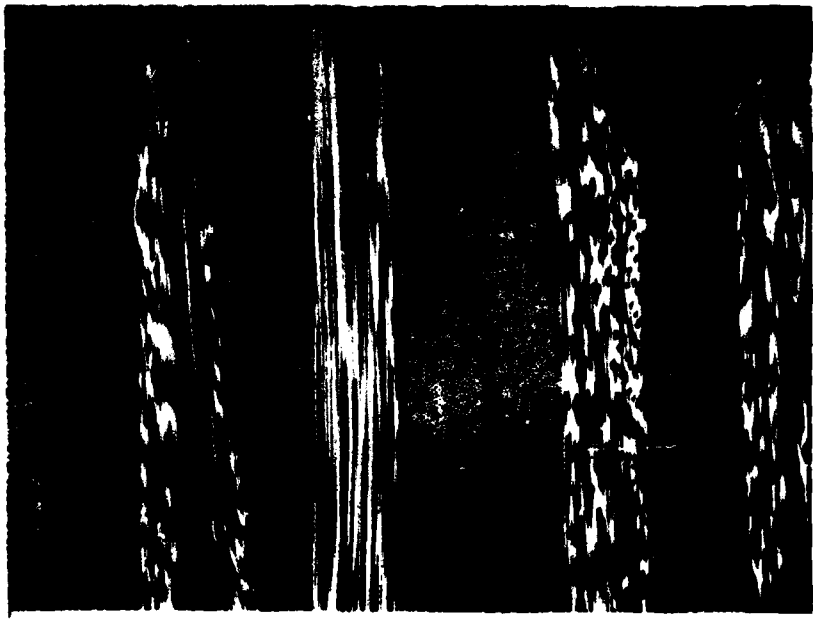


332/DMHDA; 8-HARNESS SATIN WEAVE



332/DMHDA, 5-HARNESS SATIN WEAVE

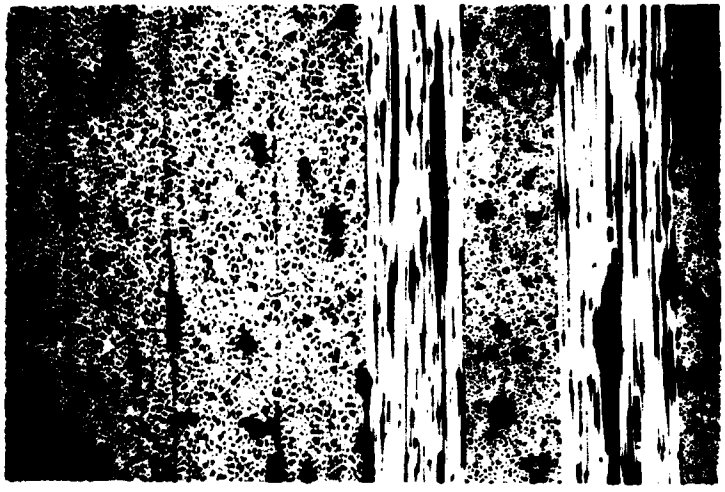
FIGURE 1. A VOID CONTENT COMPARISON OF THE 8-HARNESS AND 5-HARNESS GRAPHITE CLOTH USING THE 332/DMHDA RESIN SYSTEM.



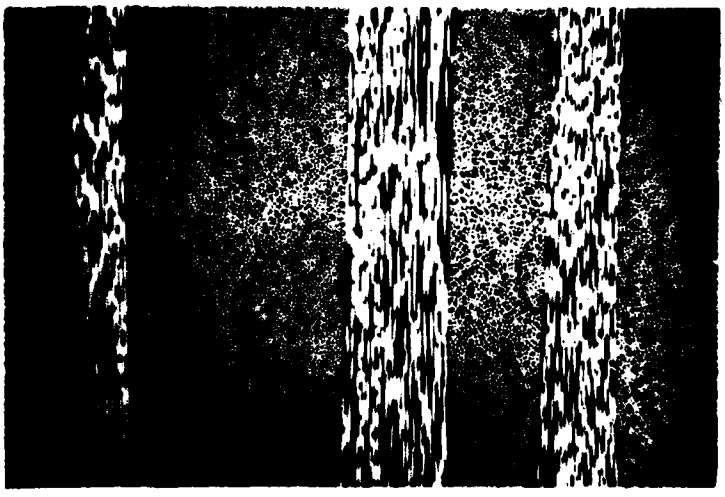
332/513/DMHDA, 8-HARNESS

332/1299/DMHDA, 8-HARNESS

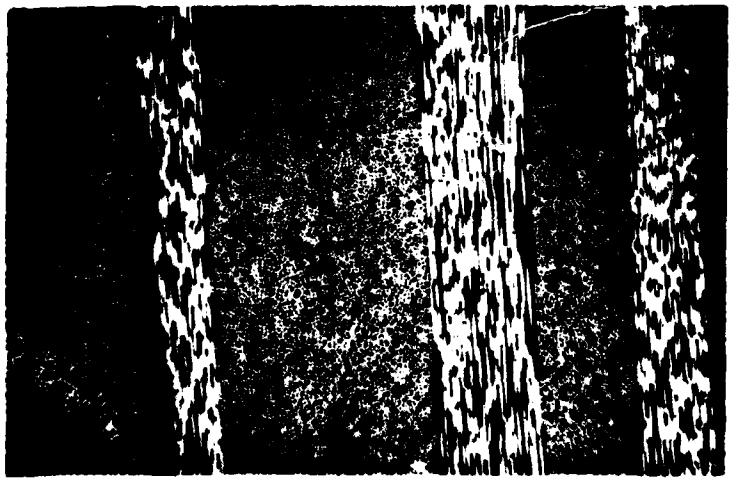
FIGURE 2. A RESIN FLOW COMPARISON OF THE NC-513 AND ECN 1299 ADDITIVES TO THE 332/DMHDA RESIN SYSTEM.



332/1299/DMHDA, 5-HARNESS

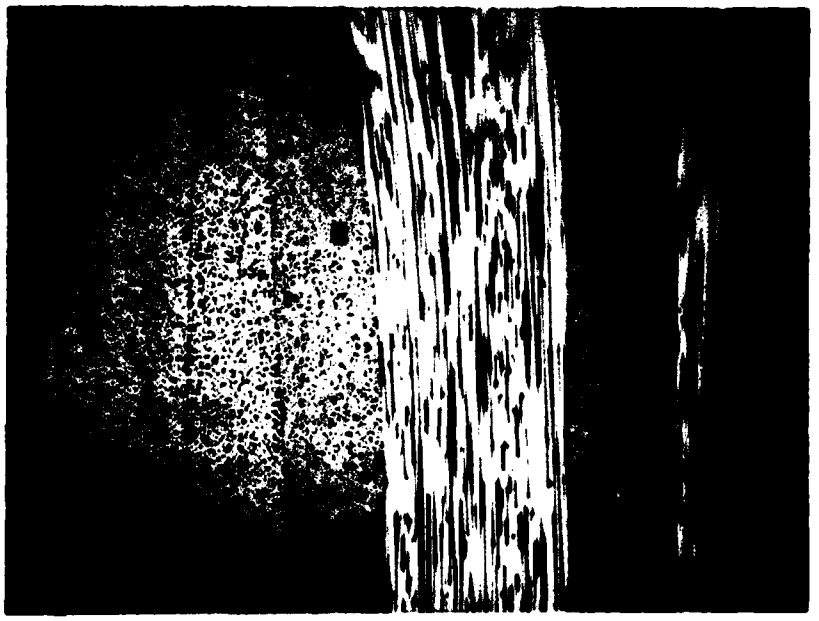


332/1299/DMHDA, 5-HARNESS



332/1299/DMHDA, 5-HARNESS

FIGURE 3. A COMPARISON OF THE LAMINATE QUALITY (RESIN DISTRIBUTION) IMPROVEMENTS WITH EXPERIENCE.



332/DMHDA, 5-HARNESS

ARNOX 3110, 8-HARNESS

FIGURE 4. A VOID CONTENT COMPARISON OF 332/DMHDA AND ARNOX 3110.

Figure 5. DU PONT DIFFERENTIAL SCANNING CALORIMETER WITH 1090 THERMAL ANALYZER.

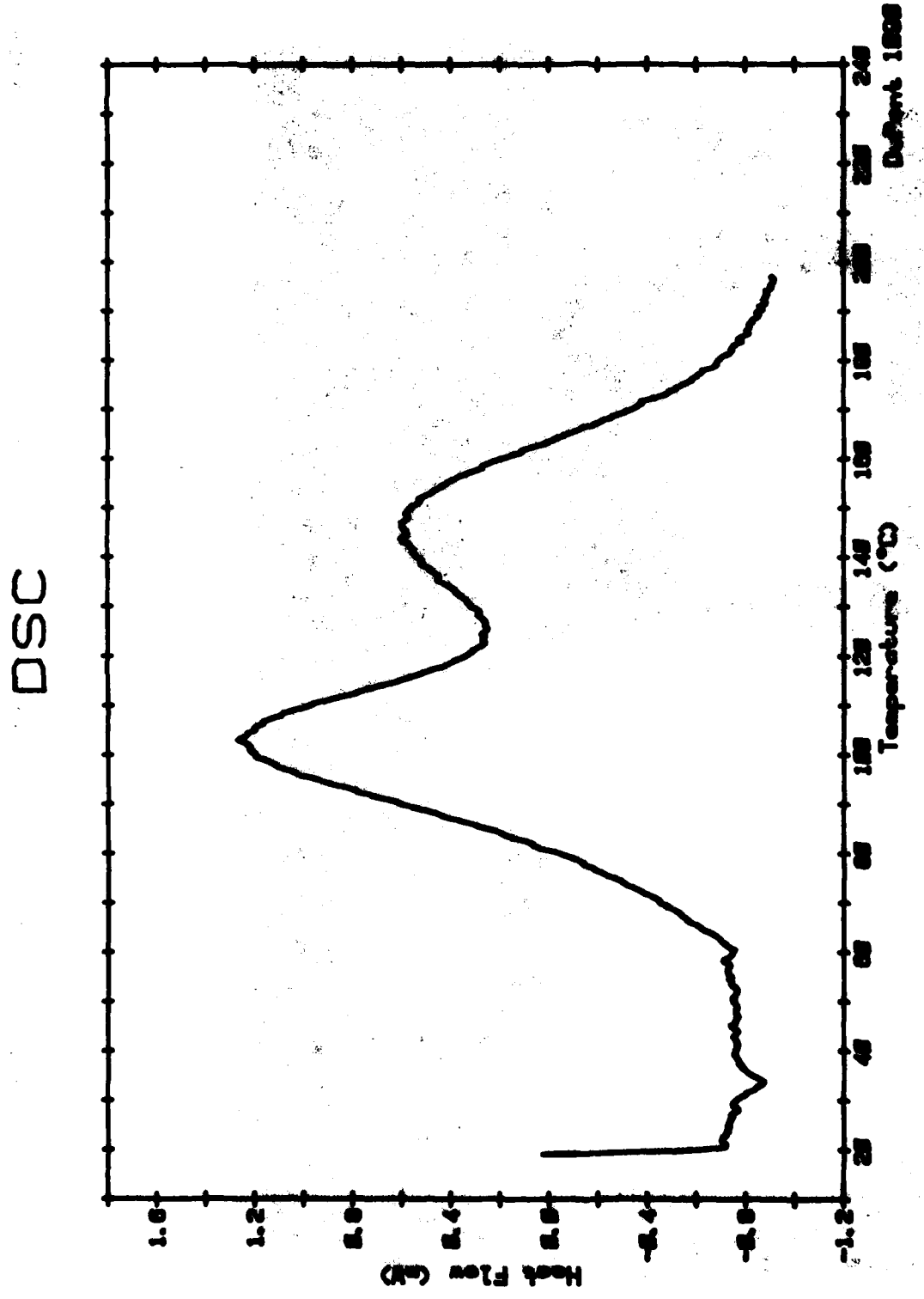


Figure 6. DSC PLOT OF 332/DMHDA IN THE FRESHLY MIXED CONDITION

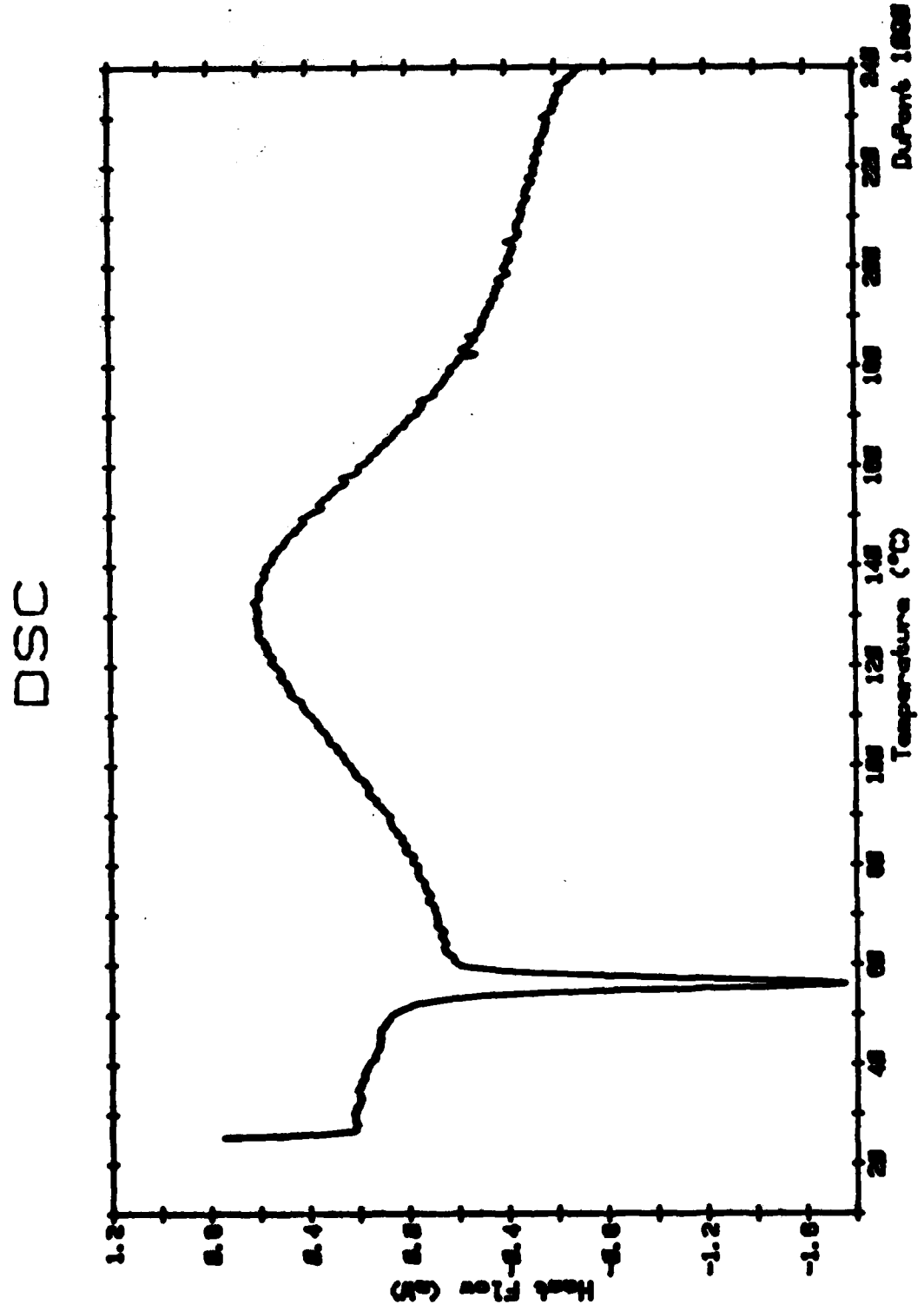


Figure 7. DSC PLOT OF 332/DMHDA IN THE B-STAGED CONDITION

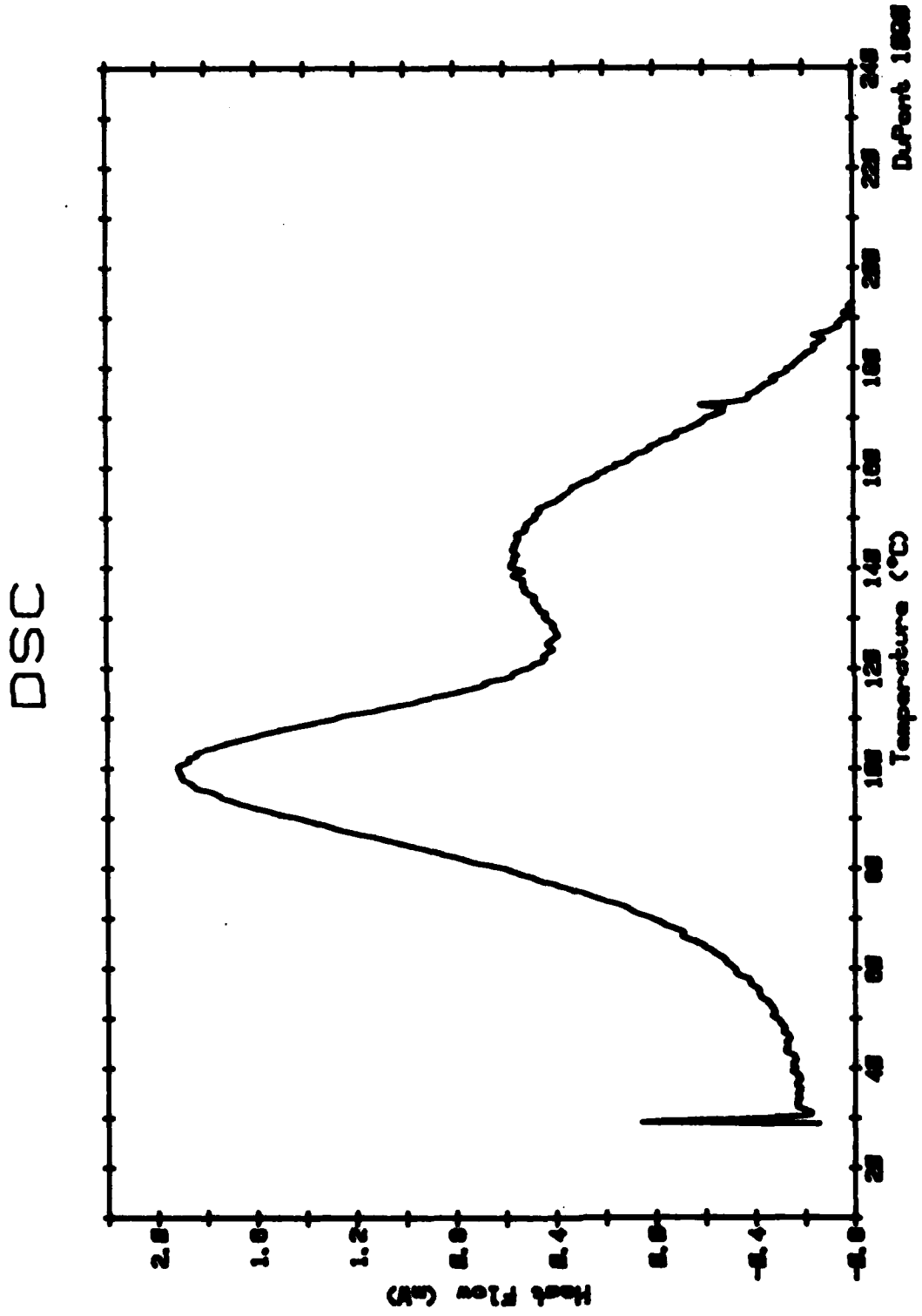


Figure 8. DSC PLOT OF 332/1299/DMHDA IN THE FRESHLY MIXED CONDITION

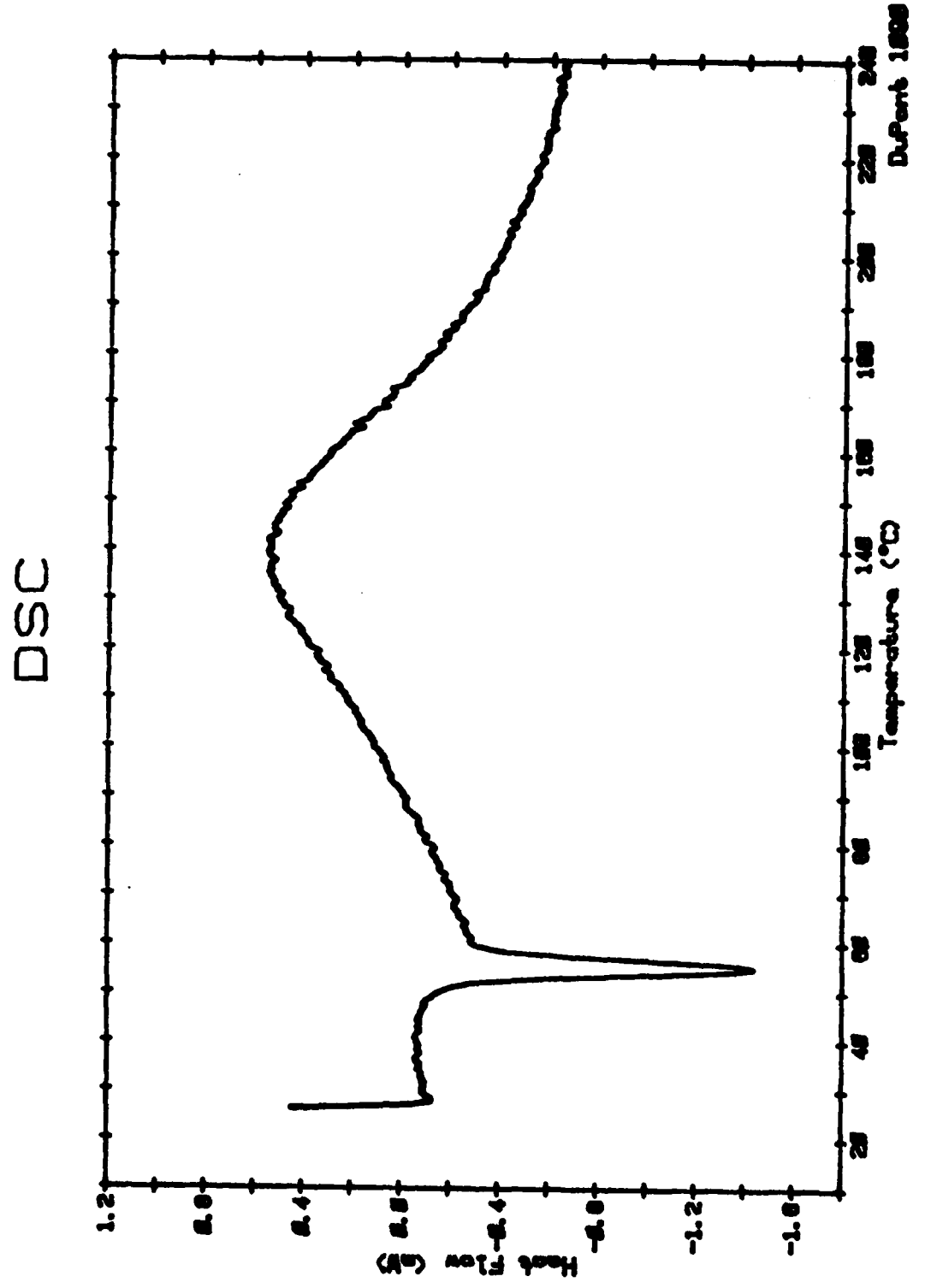


Figure 9. DSC PLOT OF 332/1299/DMHDA IN THE B-STAGED CONDITION

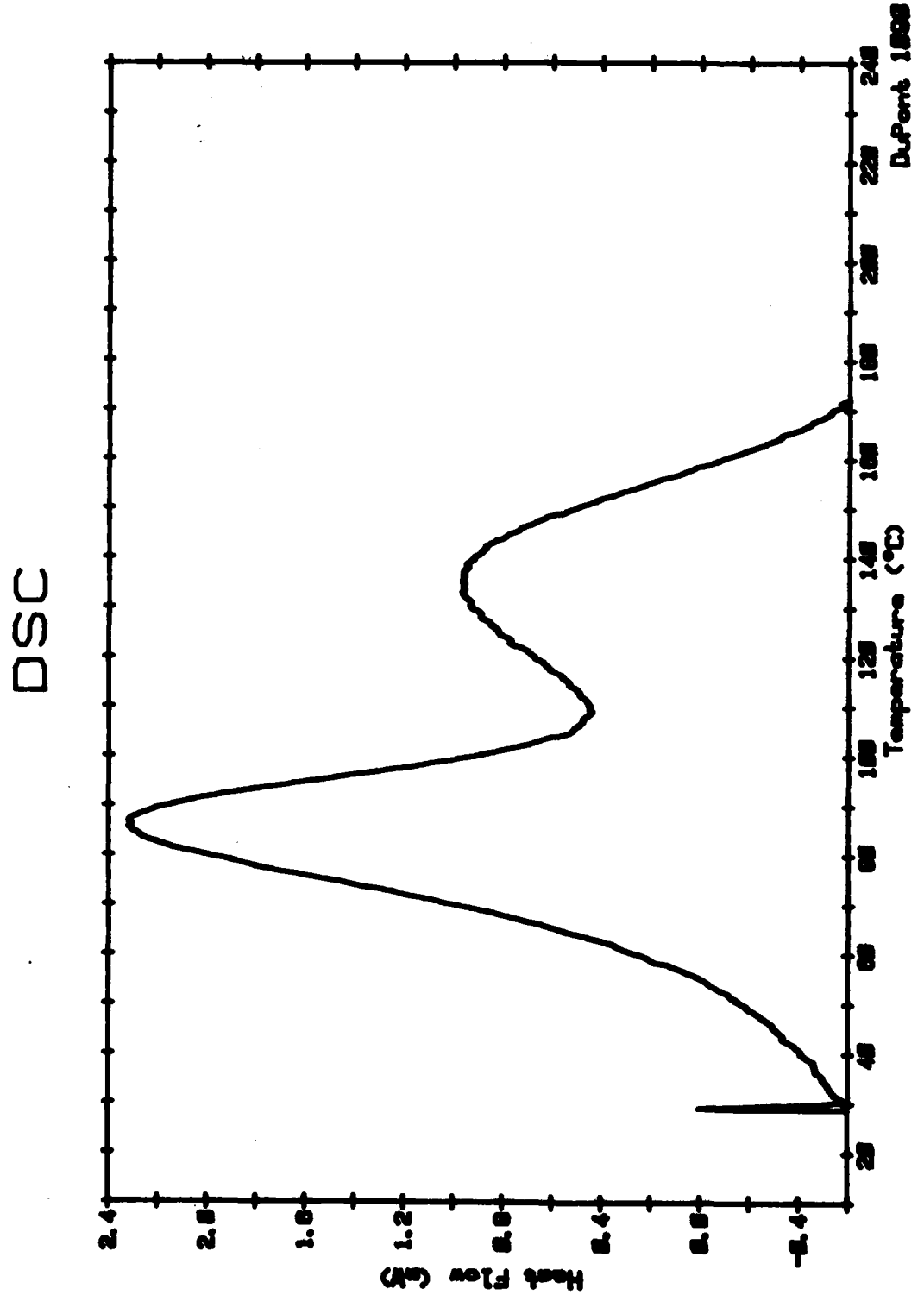


Figure 10 DSC PLOT OF RDGE/MD IN THE FRESHLY MIXED CONDITION

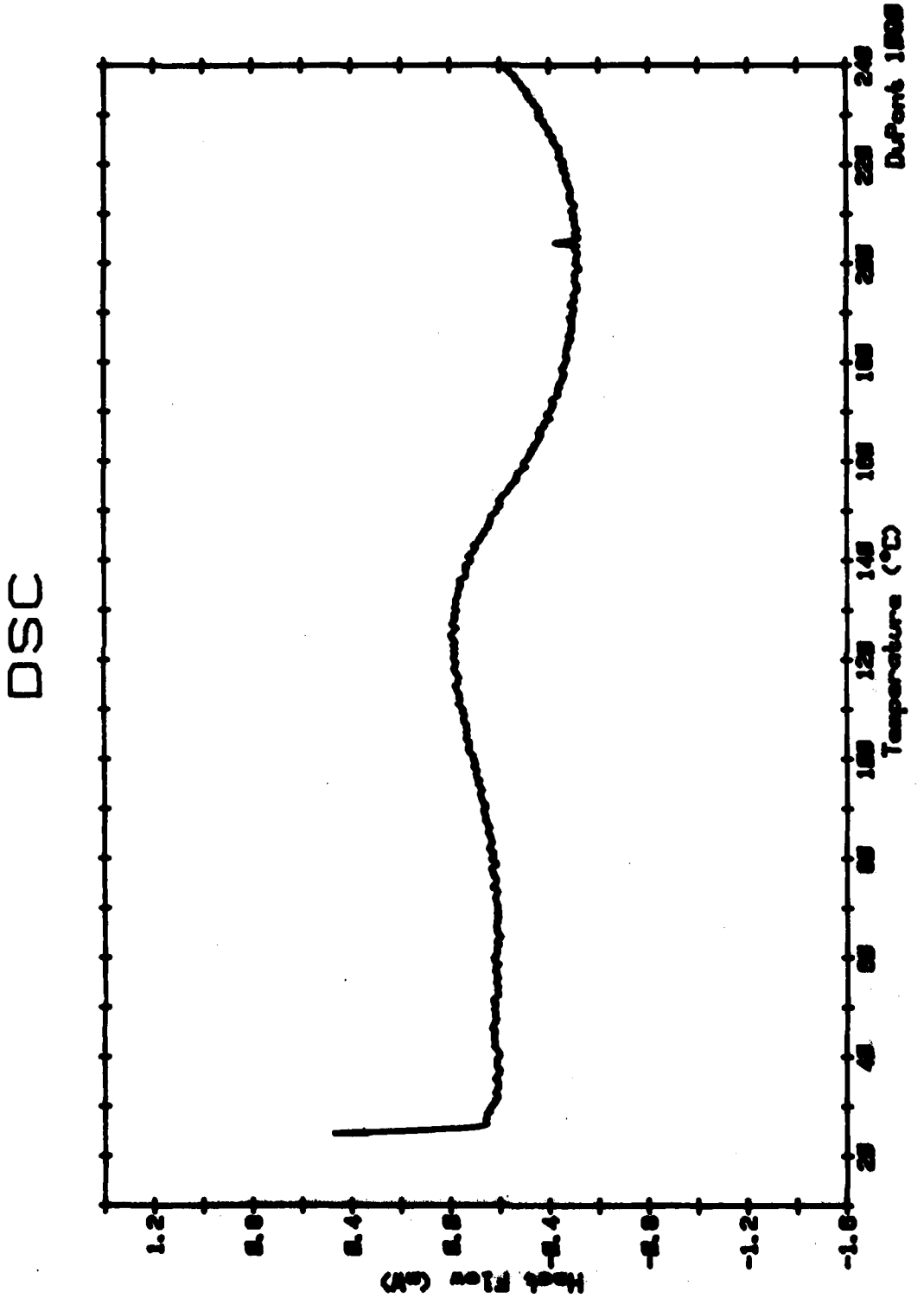


Figure 11. DSC PLOT OF RDGE/MD IN THE B-STAGED CONDITION.

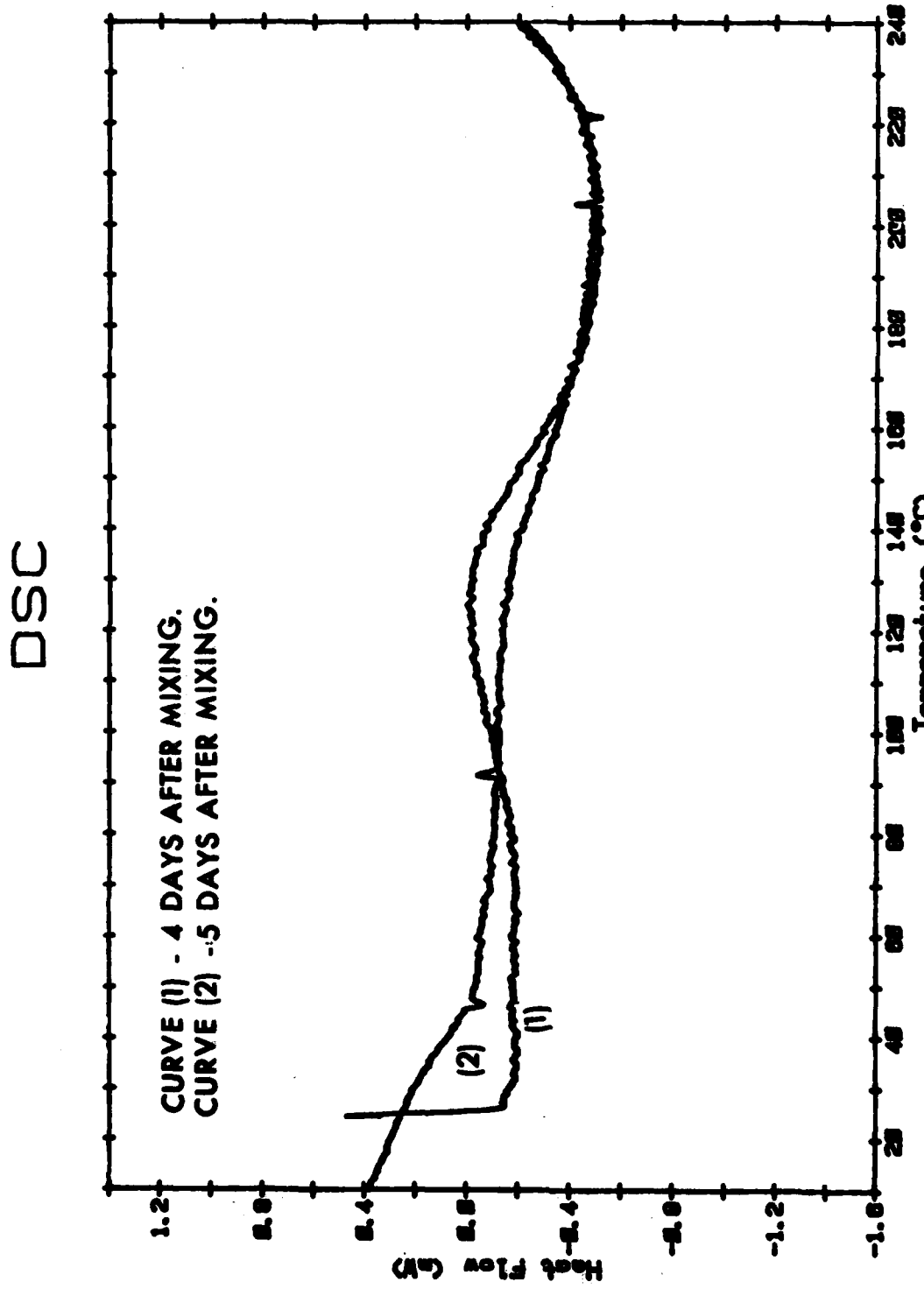


Figure 12. DSC PLOTS OF RDGE/MD INNTHE B-STAGED CONDITION SHOWING ADVANCEMENT WITH TIME

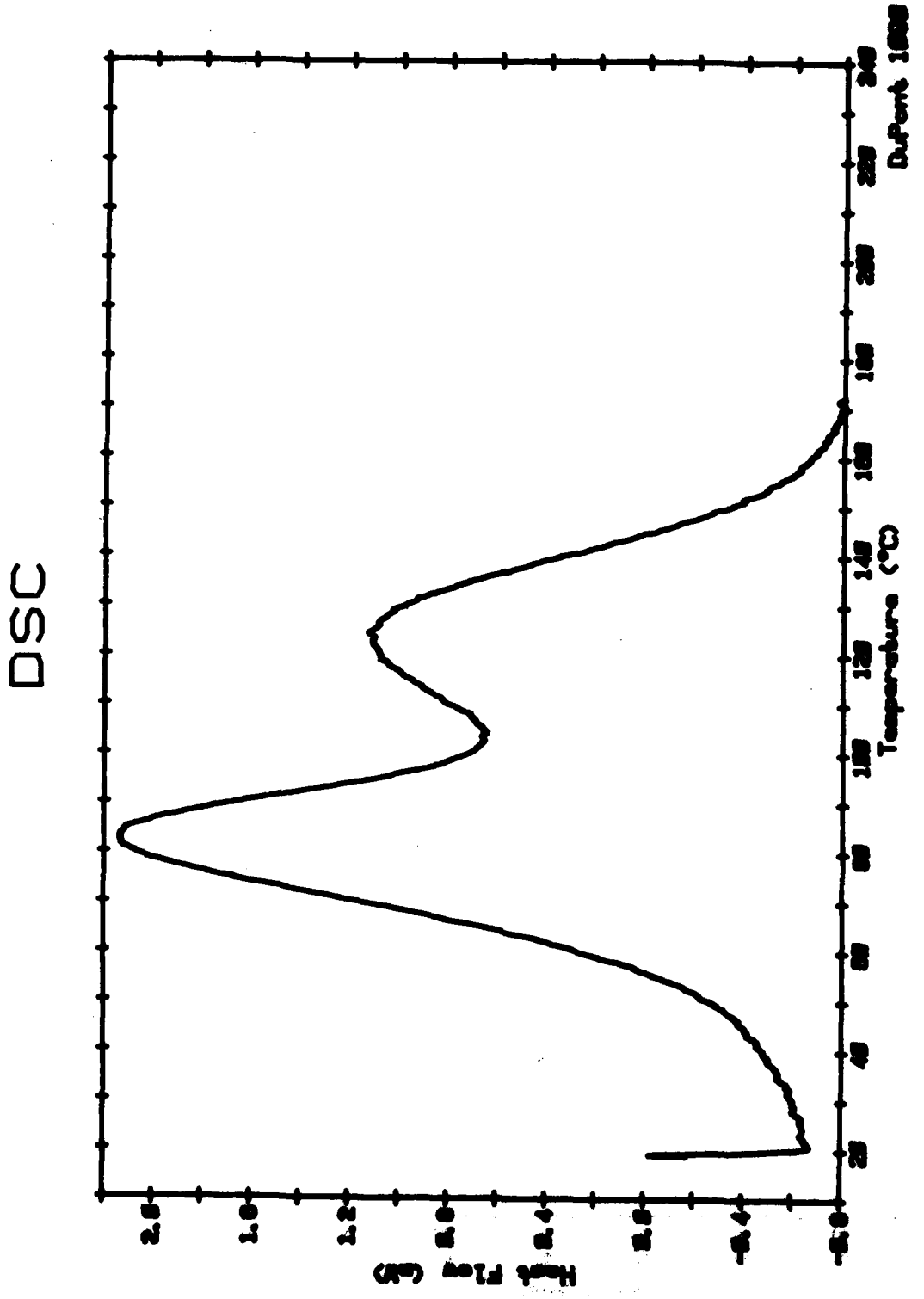


Figure 13. DSC PLOT OF RDGE/DMHDA IN THE FRESHLY MIXED CONDITION

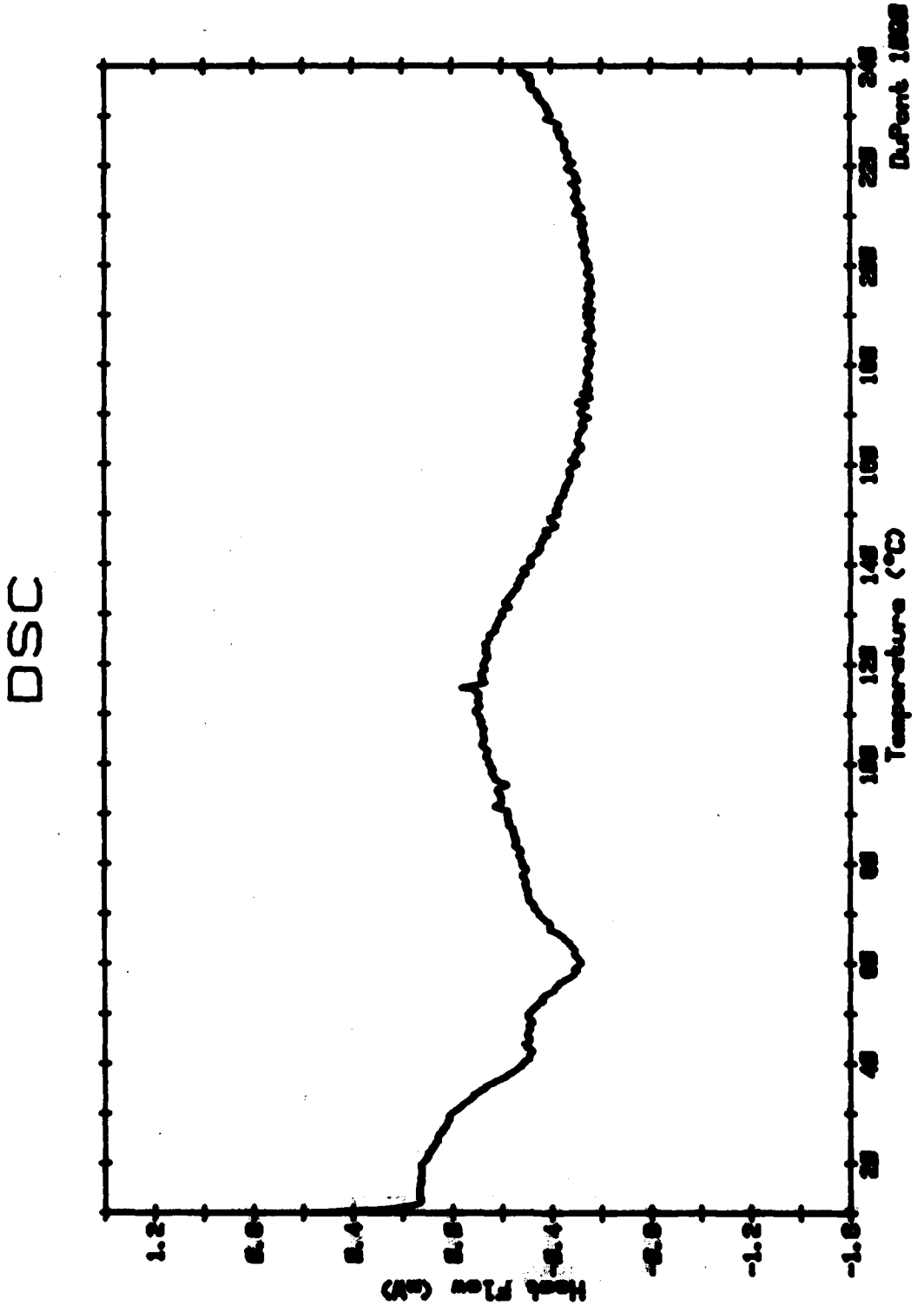


Figure 14. DSC PLOT OF RDGE/DMHDA IN THE B-STAGED CONDITION.

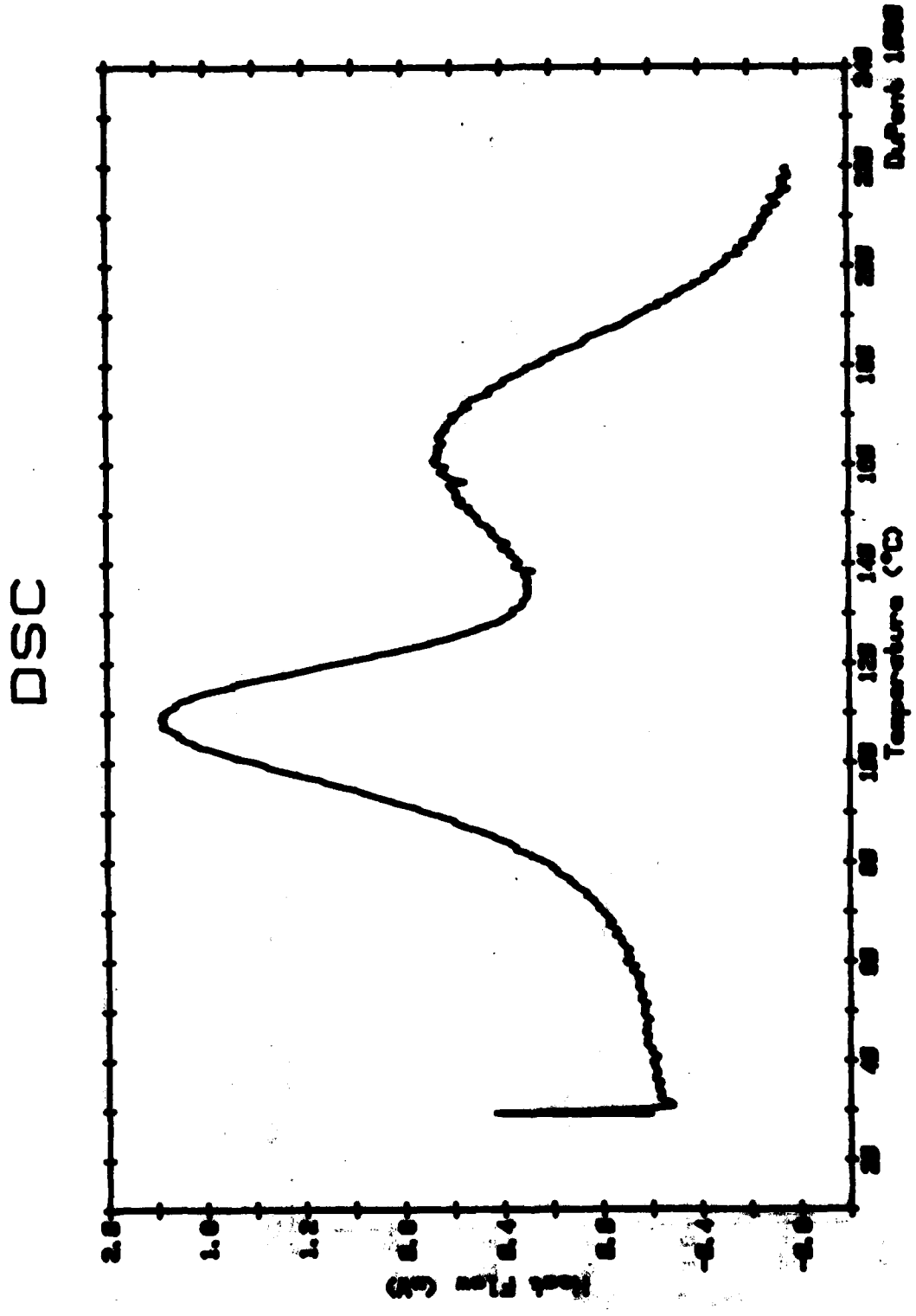


Figure 16. DSC PLOT OF 332/MD IN THE FRESHLY MIXED CONDITION

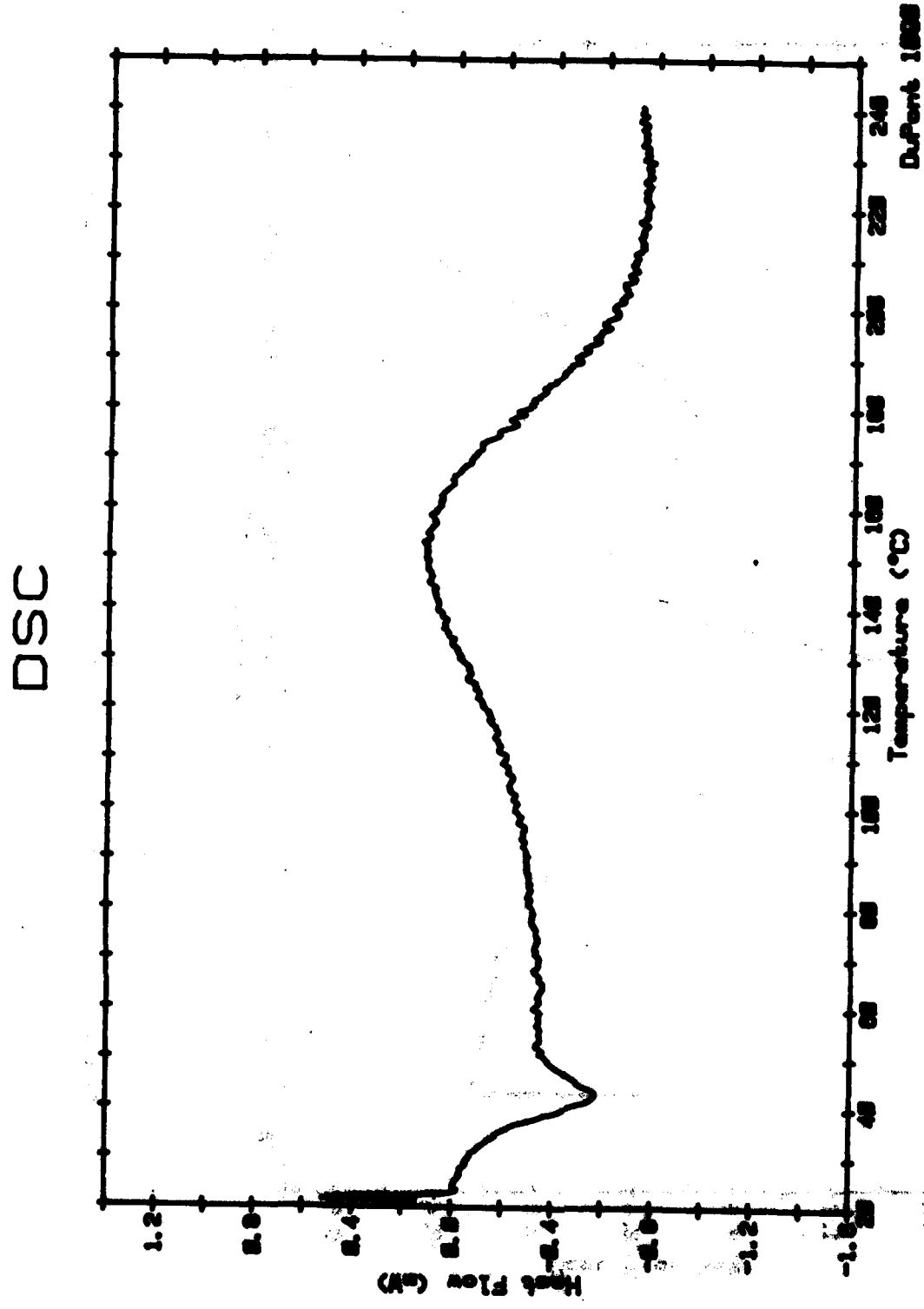


Figure 16. DSC PLOT OF 382/MD IN THE B-STAGED CONDITION.

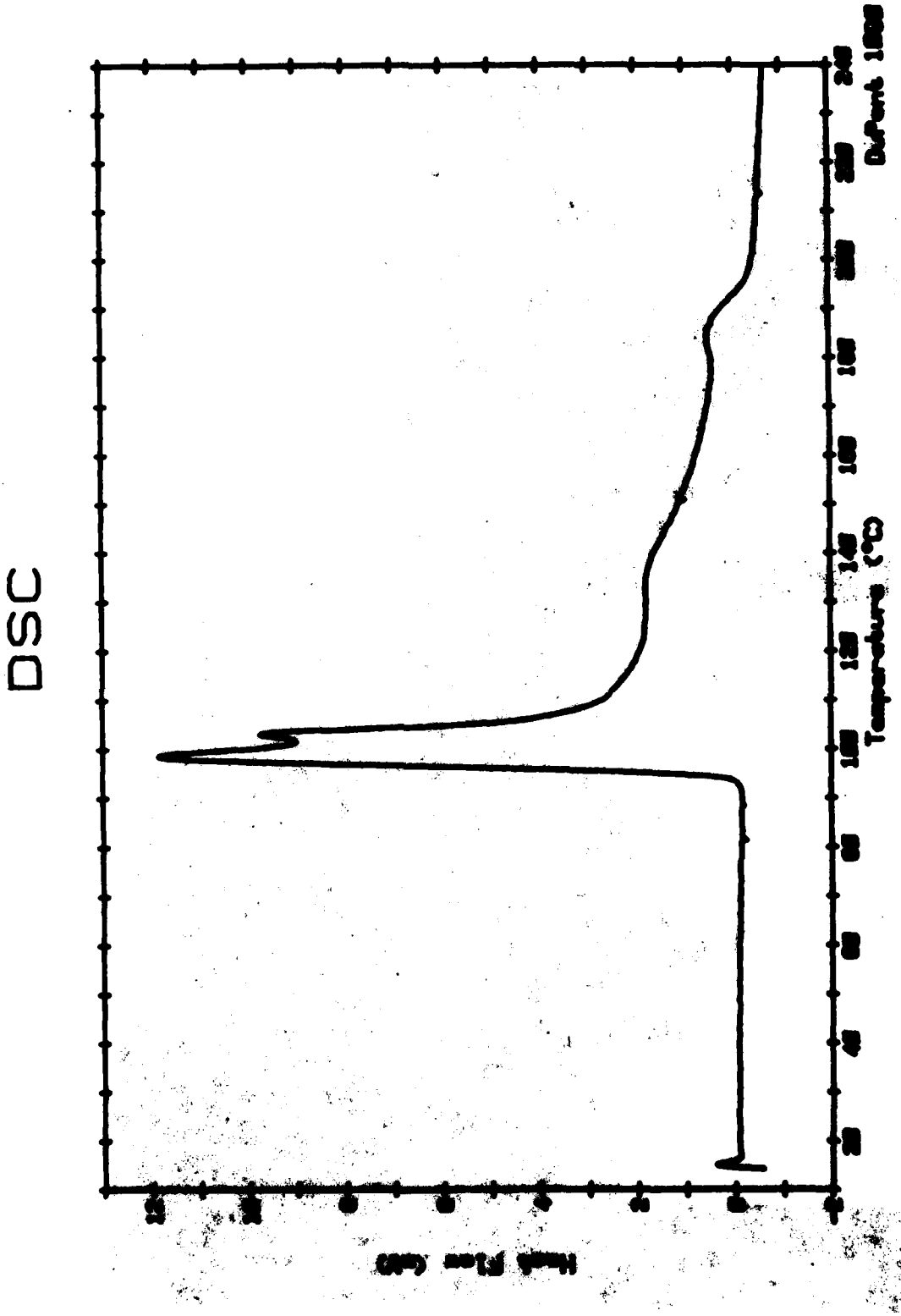


Figure 17. DSC PLOT OF ARNOX 3110.

Figure 18 DU PONT DYNAMIC MECHANICAL ANALYZER WITH 1090 THERMAL ANALYZER.

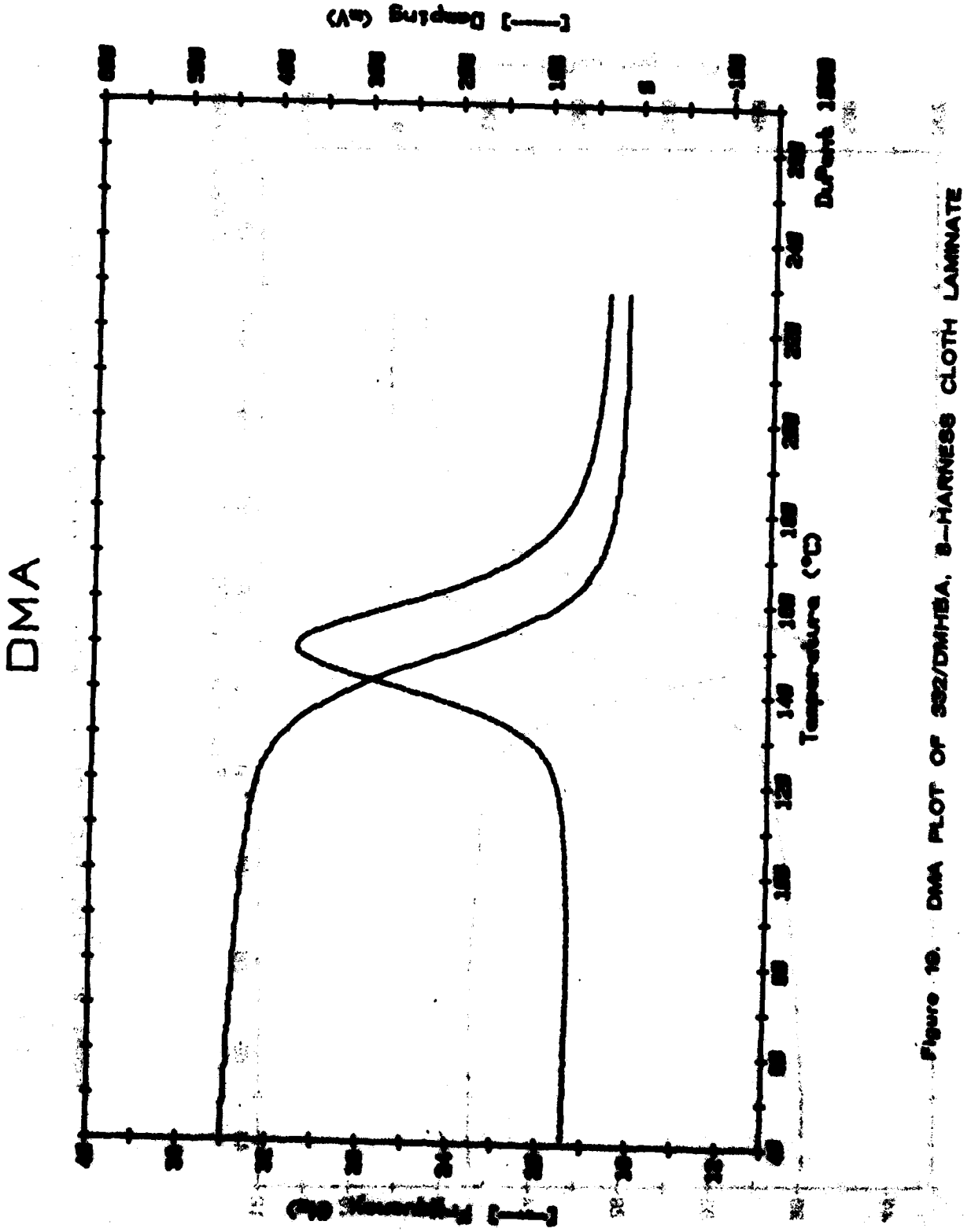


Figure 19. DMA PLOT OF 982/DMHBA, 8-HARNESS CLOTH LAMINATE

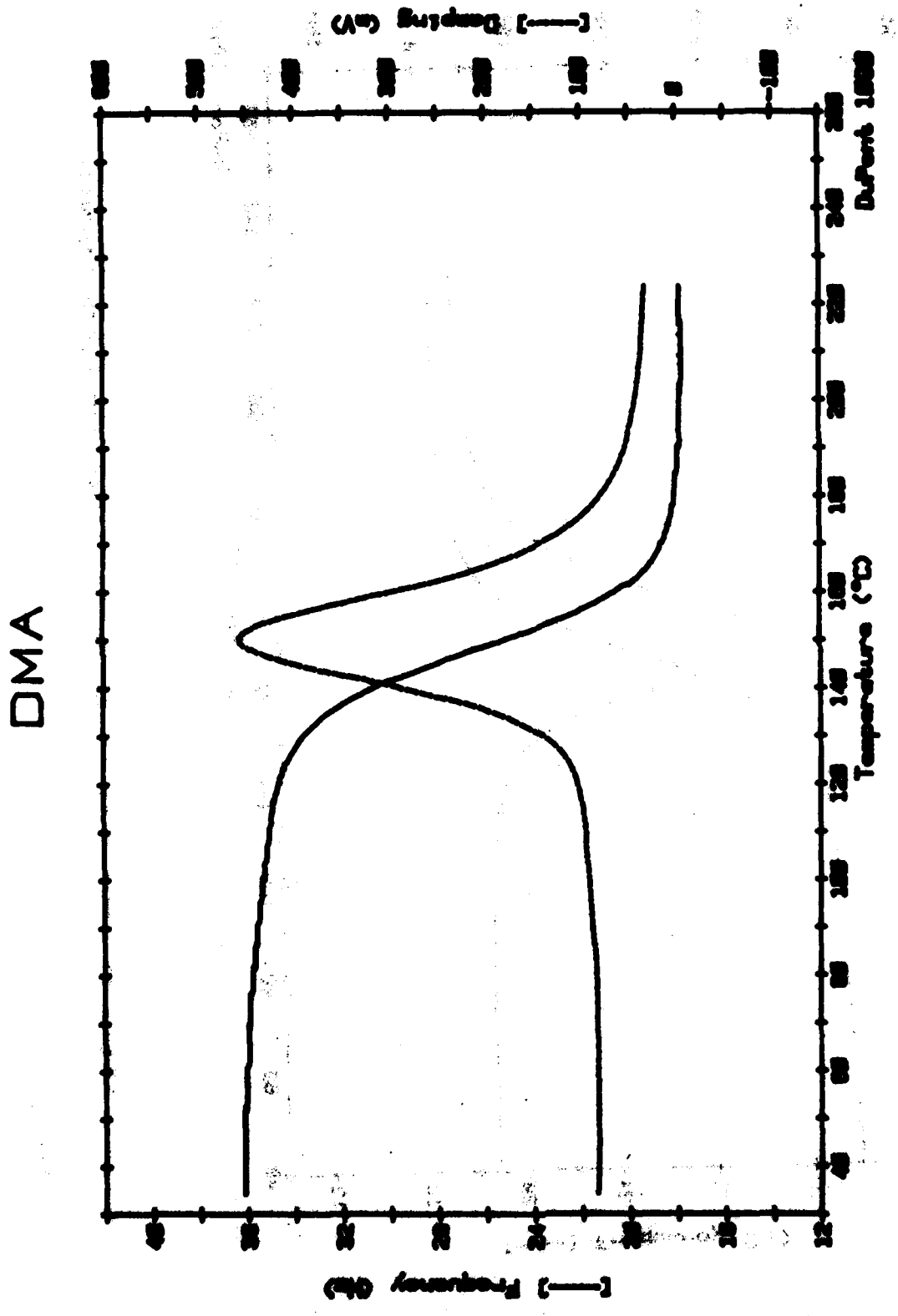


Figure 20. DMA PLOT OF 332/DMHDA, 5-HARNESS CLOTH LAMINATE.

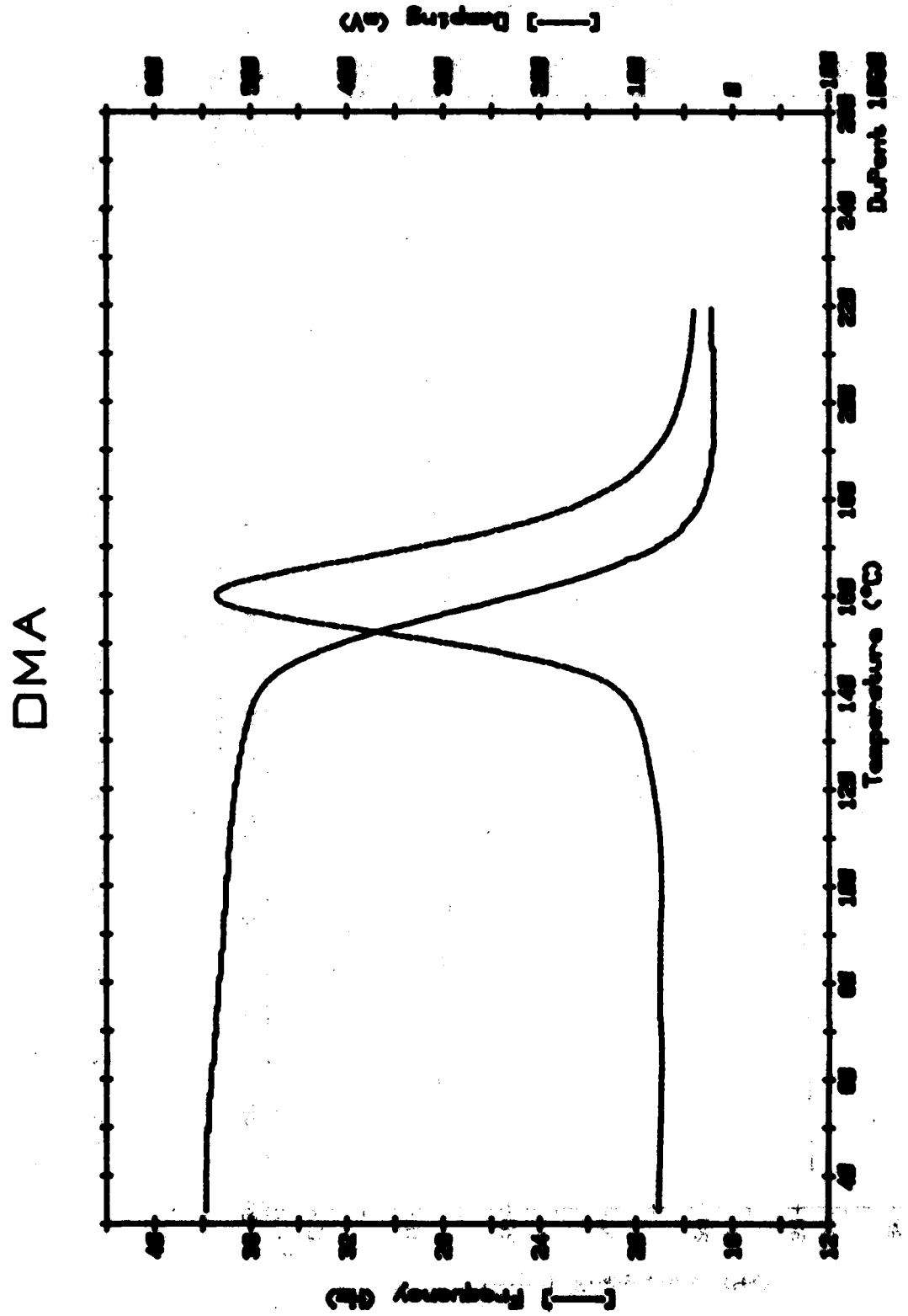


Figure 21. DMA PLOT OF 332/1299/DMHDA, 5-HARNESS CLOTH LAMINATE.

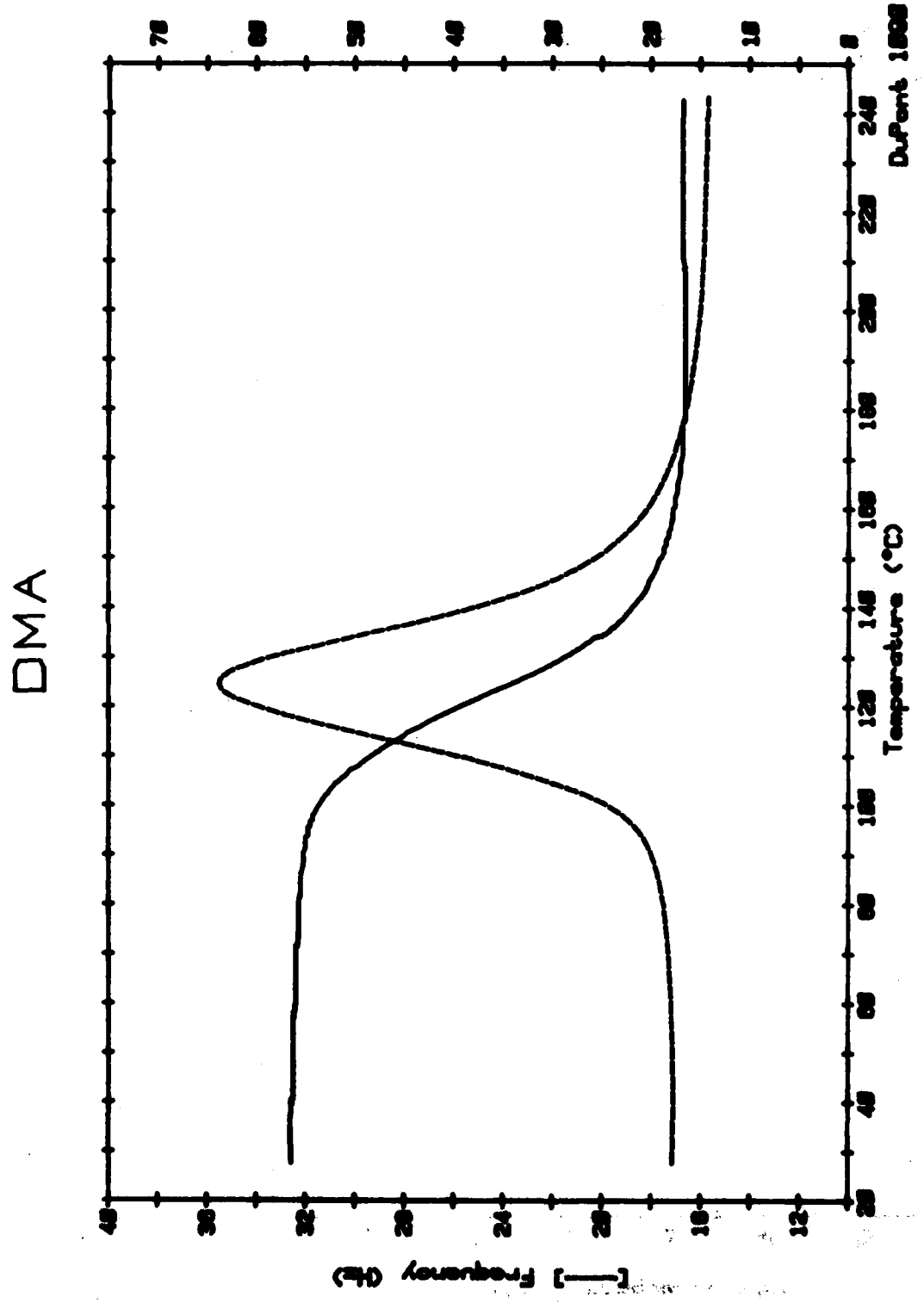


Figure 22 DMA PLOT OF RDGE/DMHDA, 8-HARNESS CLOTH LAMINATE

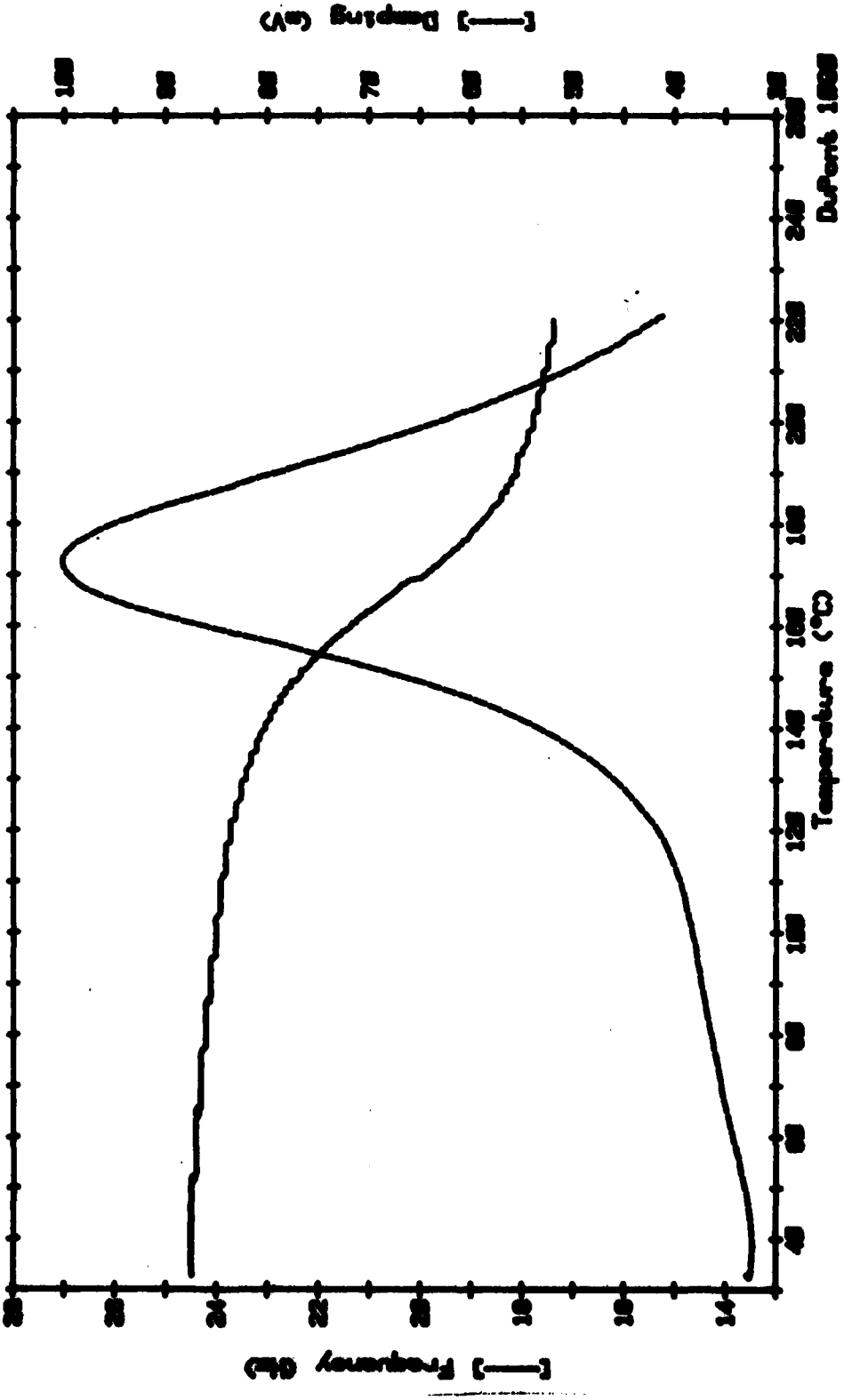


Figure 23. DMA PLOT OF ARNOX 3110, 5-HARNESS CLOTH LAMINATE.

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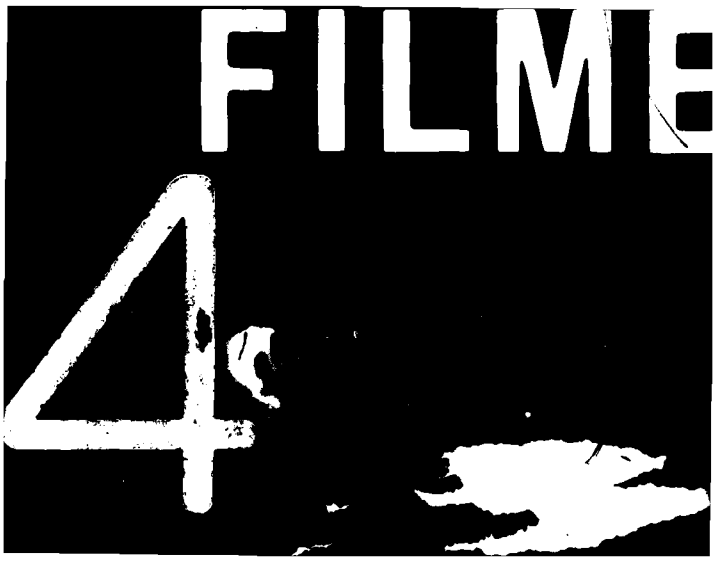
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