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# CHARACTERIZATION OF POWDER COATED THERMOPLASTIC COMPOSITE

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**Thermoplastic powder resin preforms are a relatively new form of composite preform material. Powder preforms offer improved handelability as well as the possibility to produce composites from resins that are difficult to prepreg. The purpose of this project was to characterize thermoplastic powder coated material. A Poly ether ether ketone (PEEK) resin coated material supplied by BASF was studied. The physical properties of the preform were characterized through electron microscopy and thermal analysis. Electron microscopy was used to determine the size and distribution of the powder particles along with the amount of binder in the preform. Thermal analysis DSC and TGA were used to determine the melting point and the amount of binder in the preform. In addition the resin content was measured using acid digestion. The resulting understanding of the material properties will provide a basis for developing process models and implementing this material in a production process.**

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## LIST OF FIGURES

# INTRODUCTION

This report describes the characterization of powder polymer coated thermoplastic composite. Characterization of the material utilized thermal analysis and electron microscopy to determine the physical properties of the preform and to observe the size, shape and distribution of the particles. The powder preform material was supplied by BASF thermoplastic materials. The material consisted of celion G30-500 graphite fiber with Poly ether ether ketone (PEEK) resin. Two grades of PEEK resin were evaluated, 150P and 450P. The differences between theses two types of resin are 150P has a lower molecular weight and viscosity than the 450P grade in addition the 450P preform was produced on a research impregnation line and the 150P preform on a production impregnation line.

The demand for high strength light weight structures in aerospace, transportation and recreational applications has brought about an increased use of composite materials. Composites offer high specific strength and stiffness as well as resistance to hostile environments and the ability to be formed into many complex shapes. These properties make composites ideal materials for many structural applications.

Thermoset composites offer all of the required properties; however, their impact resistance, limited shelf life and long processing times make them less efficient than thermoplastic matrix composites in many applications. While the design database

for thermosets is much larger and the experience in production greater for thermosets, the use of thermoplastics is rapidly increasing in many areas due to their processing and mechanical property advantages.

Thermoplastics can be processed using cycle times on the order of minutes versus the hours required for thermoset materials. Thermoplastics offer the ability to be reprocessed if required, since no chemical bonds are formed during processing. Thermoplastics also offer improved impact resistance, damage tolerance and an infinite shelf life. Some of the drawbacks to the use of thermoplastic composites include lower compression strength, higher processing temperatures and pressures, and lack of experience in production and use.

Thermoplastic matrix composites have been available for many years; however the poor solvent resistance of the early thermoplastic polymers has limited or excluded their use in many applications The recent introduction of engineering thermoplastics such as Poly-ether-ether-ketone (PEEK), Poly-phenylene-sulfide (PPS), and Poly-aryl-ether-sulfone (RADEL X) have made available thermoplastic composites with good solvent resistance.

In addition to processing difficulties, thermoplastics are often difficult to work with in the fabrication stage. Thermoplastic prepregs have no tack, poor drape and their boardy nature makes layup difficult particularly on curved tools. Layup of dry boardy prepreg requires that plies be tacked together to

prevent them from moving and to maintain orientation. This is done by melting the matrix resin with a soldering iron or other localized heat source. In many cases large flat sheets are made with the required orientation and then formed in a secondary operation. This flat sheet forming process is limited by the amount fibers can move, thickness variations in corners and walls and wrinkles that result from the material being compressed and stretched.

**Thermoplastic Materials**

Poly-ether-ether-ketone has been available as a matrix for long fiber composite material since the early 1980s. This material known as APC-II was developed by ICI(1). APC-II has some advantages over thermosetting materials including its toughness, short processing time and reprocessability. These advantages are offset somewhat by the high processing temperatures of 382°C (720°F) to 399°C (750°F) and consolidation pressures of 0.689 MPa (100 psi). The prepreg is also difficult to work with due to its lack of tack and drape. Plies must be tacked together using heat to melt the resin in order that the plies of composite hold alignment. It is also difficult to conform to a curved tool since the material is very stiff and without tack. The problems associated with the processing of PEEK can be overcome by forming flat sheets and then using other traditional metal forming techniques such as superplastic forming and stamp forming to make the final geometry. The difficulty with these techniques is that the fibers can become misaligned during the flow in the mold,

thickness variations can occur and some complex shapes cannot be formed due to the stiffness of the fiber and the lack of extensibility of the fiber.

In an effort to eliminate some of the handling problems of thermoplastic composite hybrid fiber forms were developed(2). This material consists of thermoplastic fibers intermingled with graphite fibers to form a tow. This type of material is shown in Figure 1 The tow can then be made into unidirectional tape, woven into a fabric or a three dimensional structure. The commingled tow has drape and can be laid into a curved tool; however, commingled fibers have no tack and the processing is more difficult than for APC II. The increased difficulty is the result of higher pressures and temperatures that are required to force the resin through the fiber bed to wet the fibers. Processing of this type of material has been studied by Van West (3). In this work the consolidation of the fiber bed, impregnation of the tows and resin flow were modeled. Hybrid material was also studied by Kinard (4) who examined the processing using a statistical processing experiment where temperature, pressure and time were evaluated to determine which variables had the greatest effect on mechanical properties.

Powder resin coated preform is the newest form of thermoplastic matrix composite. Powder preform is available with thermoplastic as well as thermosetting resins. Powder preform is composed of fibers coated with a fine powder of resin(5,6). The powder size is in the order of 3 to 12 microns and is put on the

fibers in such a way as to uniformly coat each fiber. Figure 2 shows a cross section of the material. The resin is held on the fibers with a binder which makes the preform quite durable; the particles will not fall off the fibers during handling cutting or layup. This material has excellent drape and can be laid into complex tool configurations. The drape results from the resin particles and the fibers being able to move independently and not having to act as a full ply. The preform can also have tack. Tack results from the binder becoming moisturized. The tacky preform can be laid up into a complex shaped part and hold the contour and orientation of the fibers. The laminate will also hold together after it has been dried.

Powder resin coated preform offers the potential to eliminate the problems of handling thermoplastic prepreg. Powder resin preform has the same handling qualities as thermoset prepreg as well as the ability to hold its shape when it is removed from the layup tool. The processing of powder resin coated composites is not as severe as commingled tow and about the same as APC-II prepregs 399$^{o}$C (750$^{o}$F) and 1.37 MPa (200 psi)(7). The size and distribution of powder on the fibers require the resin to flow a small distance in order to wet all of the fibers. One of the questions that needs to be answered about the material is the amount of binder that is on the material, when the binder is removed from the prepreg and its effect on the quality of the laminate.

# BACKGROUND

## Powder Polymer Composite Development

Powder polymer coated fiber preform was developed to provide a means to produce composites from polymers that are difficult to prepreg due to high melt viscosity or the need to use high boiling point solvents which are difficult to remove. There are several forms of powder coated composites available which include sheathed materials, melt fused materials and binder materials (Figure 3). The material designation differentiates the material by the method used to hold the powder on the fiber. In a sheathed system powder polymer is impregnated into the fiber tow and then the tow is coated with molten polymer to hold the powder on the fibers. The hot melt system utilizes a fluidized bed or other system to impregnate the tow and then a heat source to melt the particles to fuse them onto the fiber. The binder system uses a polymer to bind the powder particles on the fibers. This system produces the most flexible and drapeable material; however, removal of the binder may be difficult. The binder type material supplied by BASF(8) was used in this study.

The general process for producing binder type of powder preform is described in the next section. The polymer is ground into a fine powder having a particle size less than 20 microns. The powdered resin is mixed with the binder in a solution. The fibers are separated to expose the largest number of fibers to the

powder. The fibers are run through the bath containing the resin powder and binder where they are coated with polymer. The rate that the fiber is pulled through the bath and the amount of powder in the bath determine the amount of resin in the composite. The width of the tape is determined by the number of tows pulled through the bath and how they are arranged in the drying step. The binder not only causes the powder to adhere to the fibers but can cause the tows to stick to one another. This effect is used to make wide tapes by placing many tows next to each other during drying. The dry material is then wound onto a roll.

Thermoplastics as well as thermoset resins can be used in this process. Matrix materials produced include Poly-ether-ether-ketone (PEEK), Poly-phynlene-sulfide (PPS), Poly-ether-sulfone (HTA), Poly-ether-imide (PEI), Thermoplastic polyimide (LaRC TPI) and Polyimide (PMR-15). The technique can be used for any polymer that is available as a fine powder. The powder coated preform provides the handling characteristics of a thermoset prepreg. It possesses tack and drape and can be formed into a complex tool. The binder holds the resin particles onto the fiber while the material is being handled and holds the plies together when it is laid up.

The advantage of this material form over hot melt prepregs is that this material is more flexible and has enough tack to hold plies together during layup. The flexibility is the result of the physical state of the prepreg; fibers are not bonded into a full width tape as they are in hot melt systems. The ability of each

fiber to move independently provides the preform enough drape to form over the most complex tools. The tack is the result of the binder which is soluble in water. The binder can be moisturized and dried a number of times to aid in the layup of a part. One of the drawbacks of the material form is that the preform is very thick compared to melt impregnated preforms since the particles prevent the fibers from nesting together.

# EXPERIMENTAL METHODS

In this section the experimental methods used to characterize the powder resin preform are described. A description of the test fixtures and methods is provided in order to aid in the understanding of the results presented in the next section.

## Powder Preform Physical Property Evaluation

### Scanning Electron Microscopy

The powder preform was first characterized by Scanning Electron Microscopy (SEM). In this study several pieces of the preform were mounted on an SEM stage, sputtered with gold and placed in the scanning electron microscope. The fibers and resin particles on the surface of the preform were examined. Several

samples were also made from preform that was pulled apart in order to observe the fibers and resin in the center of the ply. This was done to determine the distribution of powder throughout the ply. Micrographs at magnifications from 100X to 2000X were taken to show particle size, distribution, fiber orientation and binder content.

Optical microscopy was performed on several samples of the consolidated material in order to determine overall laminate quality, resin distribution and if any voids were present.

**Resin Content**

The resin content of the preform and processed laminates was determined by acid digestion ASTM (D3410). Samples were placed in hot sulfuric acid to dissolve and remove the PEEK resin from the fibers. After the resin was removed the fibers were dried and weighed to determine the fiber volume fraction. The resin content of laminates fabricated for mechanical testing was also measured using acid digestion technique.

**Thermal Analysis**

In order to determine the amount of binder in the preform Thermal Gravimetric Analysis (TGA) was preformed using a DuPont 952 TGA and a 1090 Thermal Analyzer. Samples were heated from 40$^{o}$C (104$^{o}$F) up to 400$^{o}$C (752$^{o}$F) in nitrogen. The weight percent lost was determined and used to estimate the binder content. A sample of APC-II was also analyzed in order to determine the weight loss of the PEEK resin and fiber. In addition to TGA,

Differential Scanning Calorimetry (DSC) was run on samples of the preform and APC-II to determine the amount of heat absorbed by the material as it was heated to the processing temperature. This testing was done using a DuPont 982 DSC and the 1090 Thermal Analyzer

**Mechanical Test Laminates**

Laminates for mechanical testing were processed in the high temperature autoclave using the following procedure. A steel tool with integral vacuum ports was used. The laminate was placed on a sheet of polyimide film coated with Freecoat FRP. A steel picture frame was placed around the laminate and covered with a ply of release coated polyimide film. The laminate was then covered with 4 plies of 120 fiberglass breather. A vacuum bag was formed around the laminate with Airtech A800-G3 vacuum sealing tape, a sheet of polyimide film was placed over the laminate and pressed into the tape to form the bag. A steel picture frame the same size as the vacuum sealing tape was placed over the bag. Clamps were placed around the picture frame to press it down into the tape and provide the required vacuum seal. Vacuum was drawn in the bag and checked for five minutes with less than 1 inch of mercury loss. Four thermocouples were placed around the panel to monitor the temperature of the laminate and to control the autoclave. Each laminate was processed at 382$^{o}$C (720$^{o}$F) and under pressures of 0, 0.14 (10), 0.34 (50), 0.69 (100) and 1.38 (200) MPa (psi). The laminates were cooled at approximately 10 degrees a minute to 149$^{o}$C (300$^{o}$F). The laminates were removed from the

autoclave and nondestructively inspected by C-scan. After inspection test specimens were cut from the laminates. Five 90° tensile specimens and five short beam shear specimens were cut from the panel.

Short beam shear specimens were tested in the an Instron test machine. Three point shear test ASTM D2344 was performed on the specimens. The shear strength was calculated for each specimen. The average shear strength for each processing condition was determined. 90° tension tests ASTM D3039 were also performed on the laminates. The average tensile strength was determined for each processing condition. All testing was performed at room temperature on five specimens from each laminate. Failure surfaces of the 90° tensile specimens were observed in the SEM. The interface between the resin and the fiber was characterized to determine the amount of adhesion between the resin and fiber.

## RESULTS AND DISCUSSION

In this section the results of experiments performed on the powder resin coated material will be presented. The results of thermal analysis, resin content, microscopy, and mechanical testing will be discussed.

**Thermal Analysis**

Thermal Gravimetric Analysis (TGA) results show that the powder resin preform lost approximately 1.6% of the material weight when heated from 40°C (100°F) to 400°C (750°F). A plot of weight versus temperature, Figure 4, shows that the weight loss was constant over the temperature range of 40°C (100°F) to 240°C (465°F). The sample lost 0.4% of its weight over this range. From 240°C (465°F) to 400°C (750°F) the sample lost an additional 1.2%. The rate of weight loss increases above 320°C (608°F). The weight loss is attributed to the loss of binder from the composite. The weight loss was compared to a sample of APC-II which was run under the same conditions Figure 5. The APC-II sample shows very little weight loss over the temperature range. The difference between Figure 4 and 5 is the result of the binder being driven out of the powder coated composite.

Differential scanning calorimetry (DSC) was run on the sample of powder resin preform. The results of the temperature scan from 25°C (75°F) to 400°C (750°F)(Figure 6) show small transitions at approximately 160°C (320°F) and 200°C (392°F) . The major melt endotherm is at approximately 300°C (572°F). The small transition at 160°C (320°F) is due to recrystalization of the polymer(9). The small transition at 200°C (392°F) may be the result of binder being removed from the sample. The results are again compared to APC-II in Figure 7 the DSC scan for APC-II shows only the melting endotherm at 300°C (572°F).

## Resin Content

Resin contents were run on the preform material and the consolidated laminates. The resin content of the 150 grade preform was 36% and the 450 grade 33%. The resin contents the consolidated laminates were 32.8% and 29.8% for the 450 grade processed at 0.689 MPa (100 psi) and 1.378 MPa (200 psi) respectively and 34.5% and 30.8% for the 150 grade consolidated at 0.689 (100 psi) and 1.378 MPa (200 psi) respectively. The resin contents were in the order that would be expected for the processing conditions and the original resin contents. Some of the difference in the resin contents from the preform to the consolidated laminate can be attributed to the weight of the binder which was measured as part of the resin content in the preform.

## Electron Microscopy

Electron micrographs of the 150 grade powder material are shown in Figure 8. The micrographs at 100X and 200X show the powder was well distributed on the fibers and all fibers had some particles on them. The alignment of the fibers was seen to vary significantly. In the 100X micrograph several fibers were seen to bend away from parallel. The 200X micrograph also shows the fibers to be non parallel with fibers tending to cross several neighboring fibers. The micrographs in Figure 9 taken at 500X and 1000X show the particles to be between 2 and 20 microns with most of the particles in the 10 micron range. In these micrographs the particles were equiaxied and fairly smooth. There was no evidence

of the binder in the photographs and the fibers do not show damage from prepregging. The micrograph in Figure 10 was taken at 1900X and shows the fibers are not damaged and the particles do not appear to have binder on them. The particles were 10 microns and relatively smooth.

The 450 grade PEEK resin material is shown in Figures 11,and 12. The 100X micrograph (Figure 11) again showed well distributed resin particles. The fibers appear to be better aligned in this sample than in the 150 material. The 200X, micrograph Figure 11, shows the fibers to cross in several locations and the particles to be well bonded to the surface. The 500X and 1000X micrographs in Figure 12 again showed good resin distribution and the particles to be between 2 and 20 microns with the average about 10 microns. The powder particles were rougher than the 150 grade particle but still spherical in shape. The 1000X micrograph showed some sign of binder or coating on the surface of the powder and the fiber. This is the only sample that showed the binder. The coating is visible on the fiber that runs from lower left corner to upper right corner. The 2000X micrograph, Figure 13, showed the equiaxed particles that look similar to the 150 grade particles. There was no evidence of the binder in this micrograph. All samples had an equal distribution of resin particle sizes. The number and distribution of particles did not vary from the edge to the center of the ply.

### Mechanical Testing

The bond produced between the resin and the fiber was characterized by short beam shear and 90° tensile testing. Laminates made using vacuum to 1.378 MPa (200 psi) were tested. The results are presented in Figure 14 and 15 for shear and 90° tensile tests. The short beam shear results show increased strength from vacuum to 0.689 MPa (100 psi) and then a leveling off above 0.689 MPa (100 psi). The 90° tensile results show a similar trend. This data may not be as reliable as the shear data since the test specimens were susceptible to cracking during handling and machining. The failed specimens were observed in the SEM to determine the degree of resin adhesion. The micrographs show that the resin adhered well to all specimens. The main difference between the samples was the number of voids in the laminate.

## CONCLUSIONS AND RECOMMENDATIONS

### Summary of Results

SEM characterization of the powder preform sample showed that the powder particles were well distributed on the fibers. The powder particles had a diameter of between 2 and 20 microns. Binder was observed on one of the 450 grade samples. The binder appeared as a rough coating on some of the fibers in the preform.

There was no evidence of the binder on the 150 grade material or on any of the other 450 grade samples. This sample was produced on a developmental coating line not the production coating equipment which may account for extra binder being on the preform. Another observation made with the SEM was that the fibers in the preform were not straight. Groups of fibers deviated from parallel in all samples. The amount of deviation varied from sample to sample and within each sample. Some of the fibers were several degrees out of parallel and crossed several neighboring fibers. This crossing over of the fibers could have a significant effect on the rate and degree of consolidation of the preform and on the mechanical properties of the consolidated laminate. The powder resin preform was much easier to handle and lay up than hot melt type prepregs. The preform was more flexible, and the tack that was developed by the binder made ply orientation easy to maintain. The binder gives the preform tack and allows fibers to move so that the material can be laid up on curved tools. The consolidation can be carried out in a similar process to hot melt thermoplastics. One problem with the material is that the plies of preform were very thick. This was the result of the resin powder preventing the fibers from packing together. The thickness of the plies resulted in very thick laminates which were difficult to bag and tool.

Resin content of the preform was determined to be 36 and 32 percent for the 150 and 450 grade material. The resin content decreased slightly on consolidation. Higher consolidation pressure caused more of a decrease in resin content.

Thermal analysis was used to determine the amount of binder in the preform and the temperature where it is removed. TGA showed a 1.6 percent weight loss over the processing range. The weight loss was rapid from approximately 240°C (465°F) to 400°C (750°F). TGA of APC-II did not show any weight loss. This weight loss of the powder resin sample is attributed to the binder being removed. DSC showed a small change in heat flow at approximately 240°C (465°F) for the powder material. The change in heat flow may be attributable to the binder being removed since it was not visible on the APC-II tests. The amount of binder in the preform is rather high. The amount that is removed and the effects it has on the composite properties must be investigated further.

For consistent high quality interface to develop in the PEEK powder preform processing at greater than 0.689 MPa (100 psi) and 382°C (720°F) is required. Short beam shear and 90° tensile testing showed an increase in strength as pressure was increased to 0.689 MPa (100 psi). Pressures above 0.689 MPa (100 psi) had little effect. The effect of the binder was not evident in the SBS tests; however the 90° tensile tests show low values for strength. This result is thought to be the caused by the binder reacting with the polymer and decreasing its tensile strength.

## Recommendations for Future Work

Further research on powder coated thermoplastics should investigate the following areas: binder removal and its effect on resin properties; consolidation of thick preforms and fiber misalignment and methods to control alignment in the preform

production. This research would provide a stronger basis for the use of powder coated thermoplastic composites. The need to understand the effect of binder on the resin and its effects on the properties of the composite must be addressed in order to understand the effects on the laminate mechanical properties.

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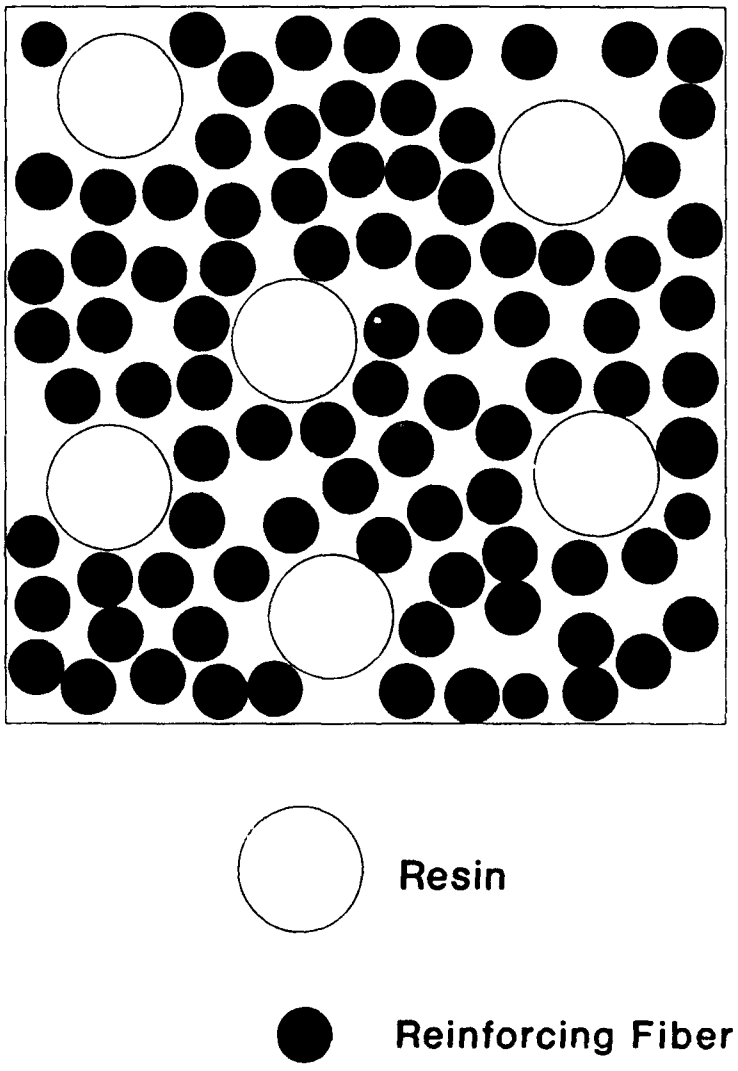



**Figure 1 Cross section of commingled material.**

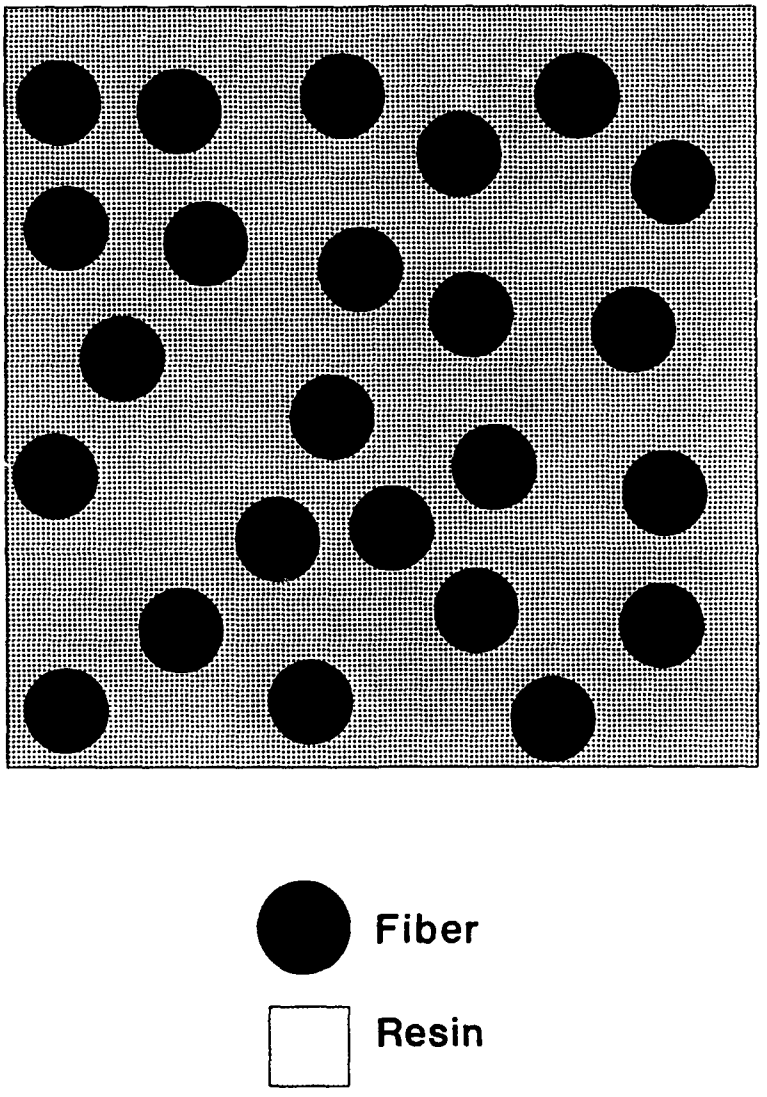



Figure 2 Cross section of powder coated material.

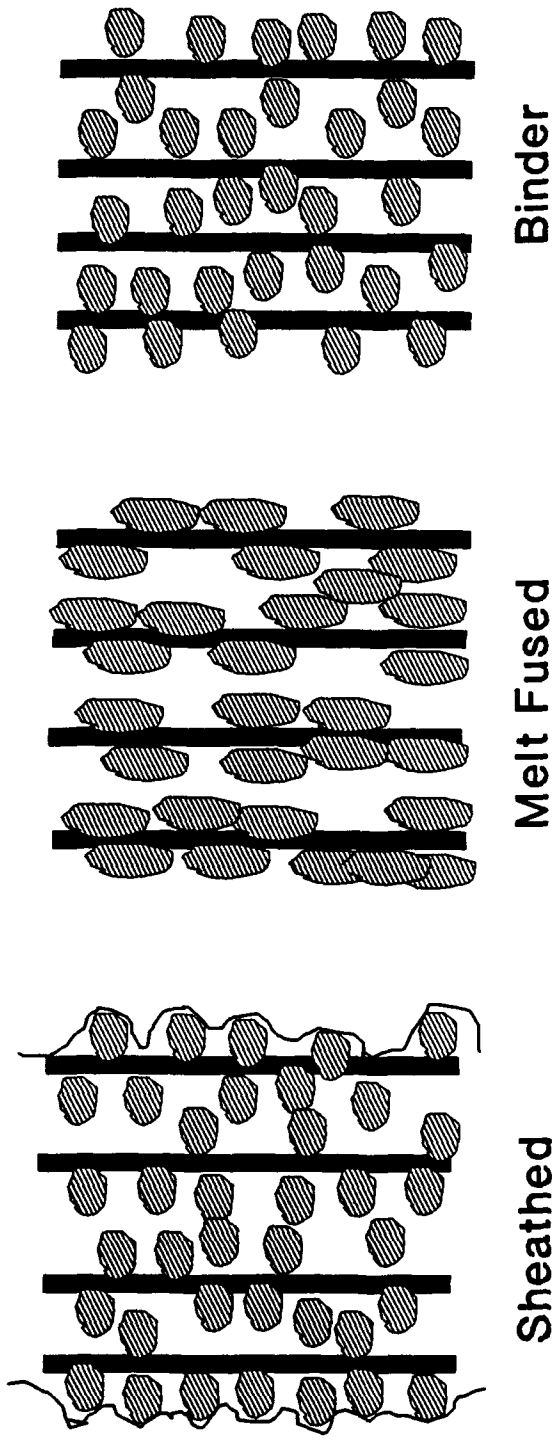



Figure 3 Types of powder coated preforms.

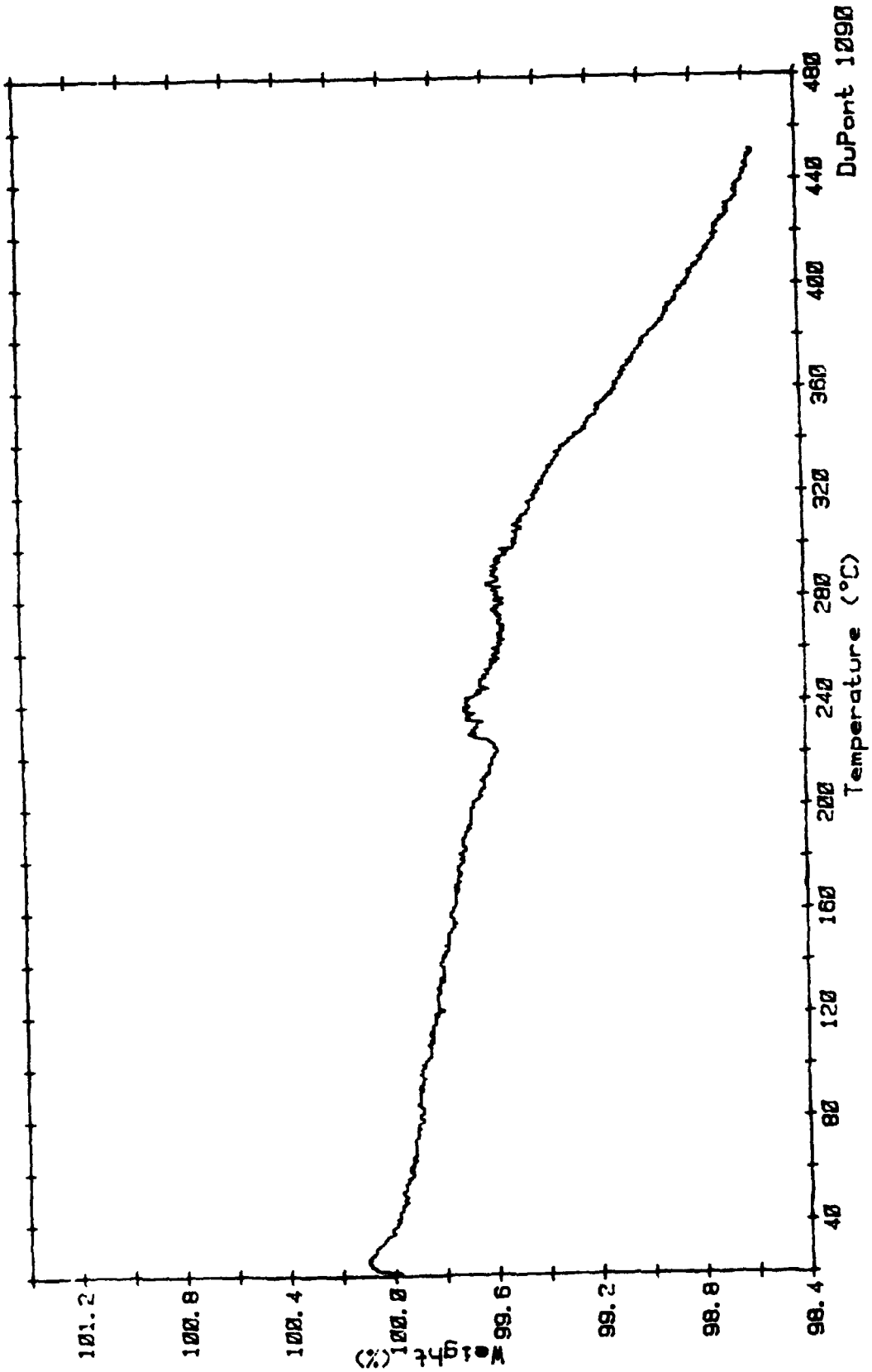



Figure 4 TGA of PEEK 150 powder preform.

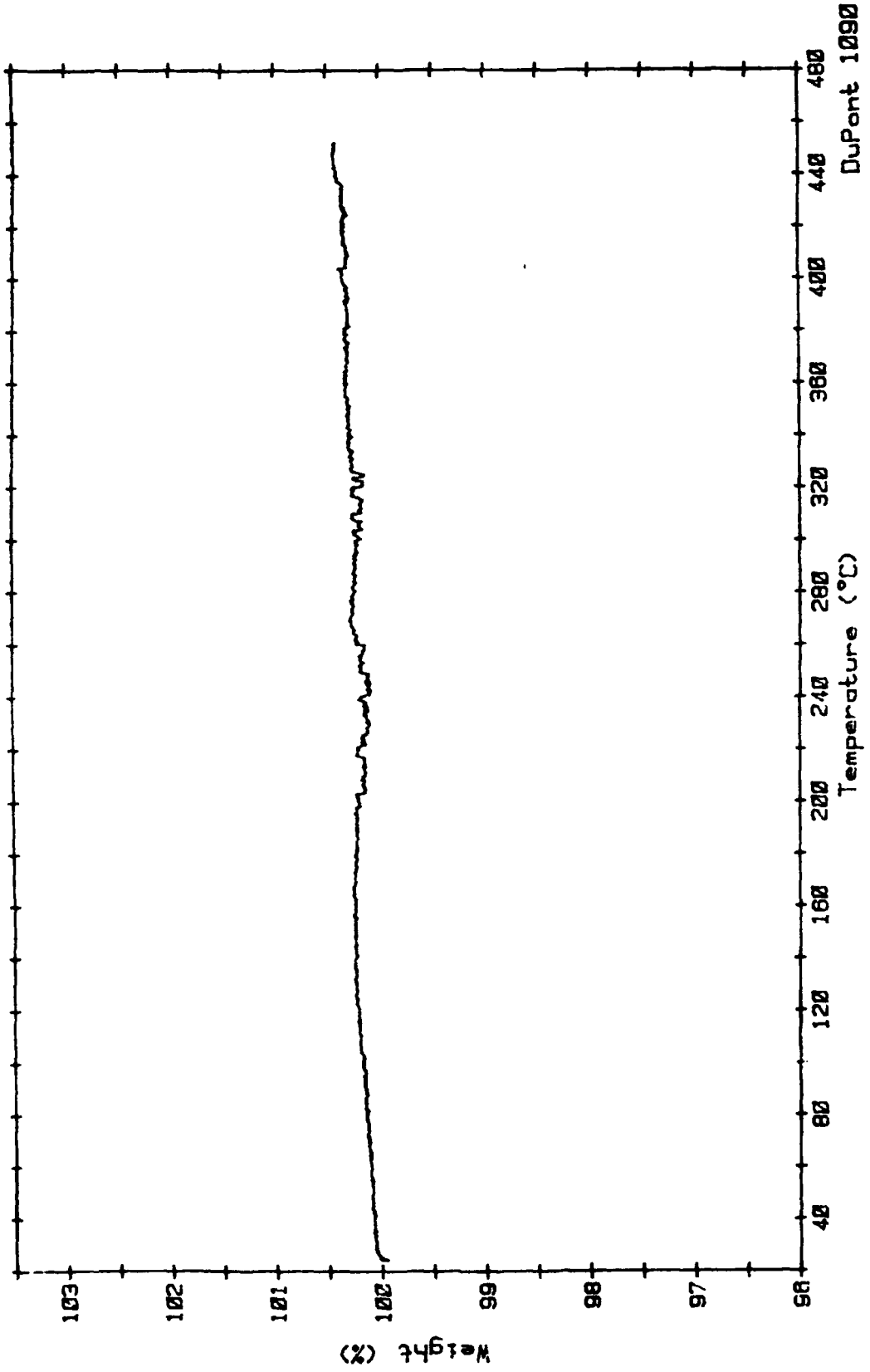



Figure 5 TGA of APC-II.

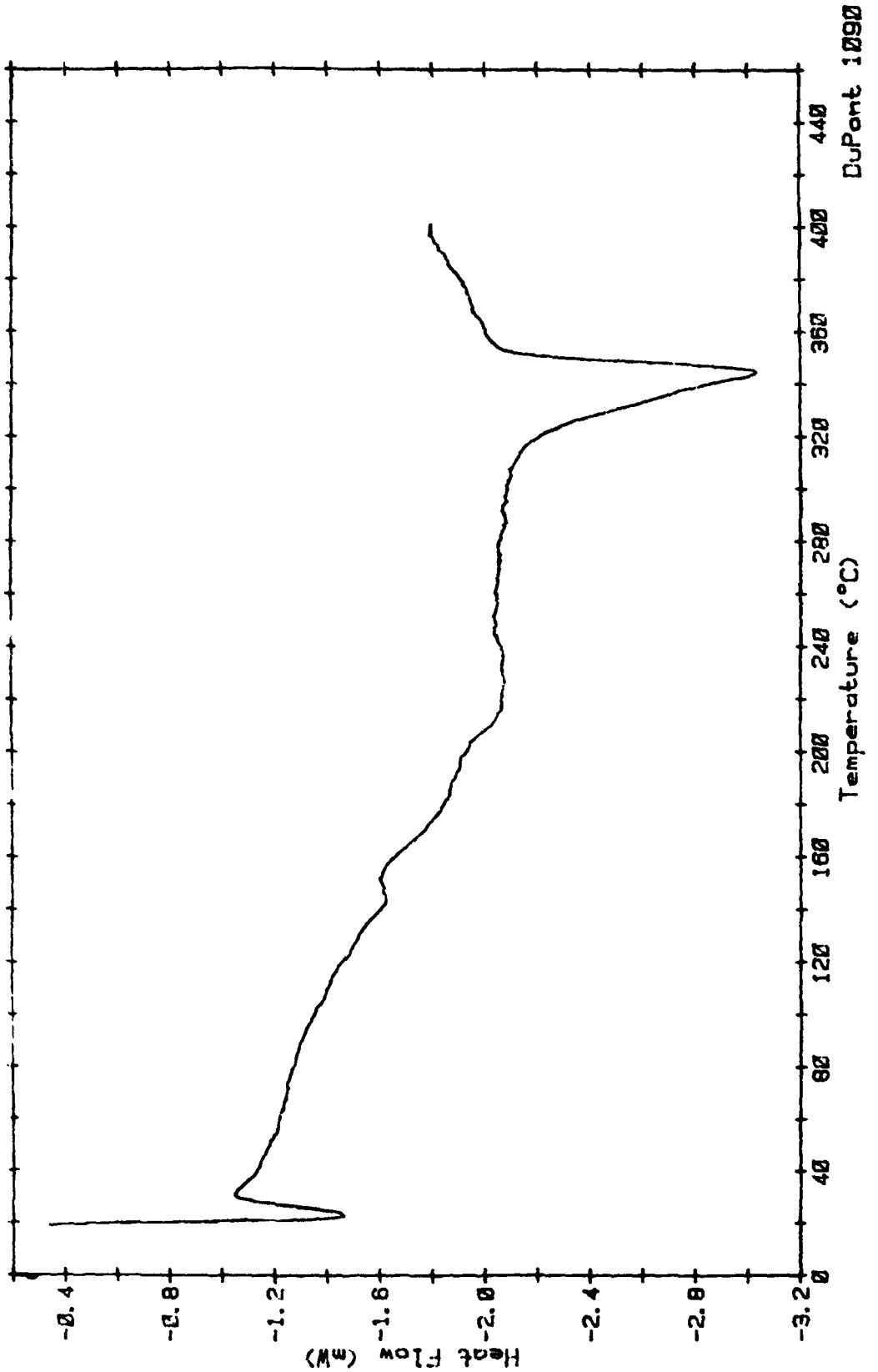



Figure 6 DSC of PEEK 150 powder preform.

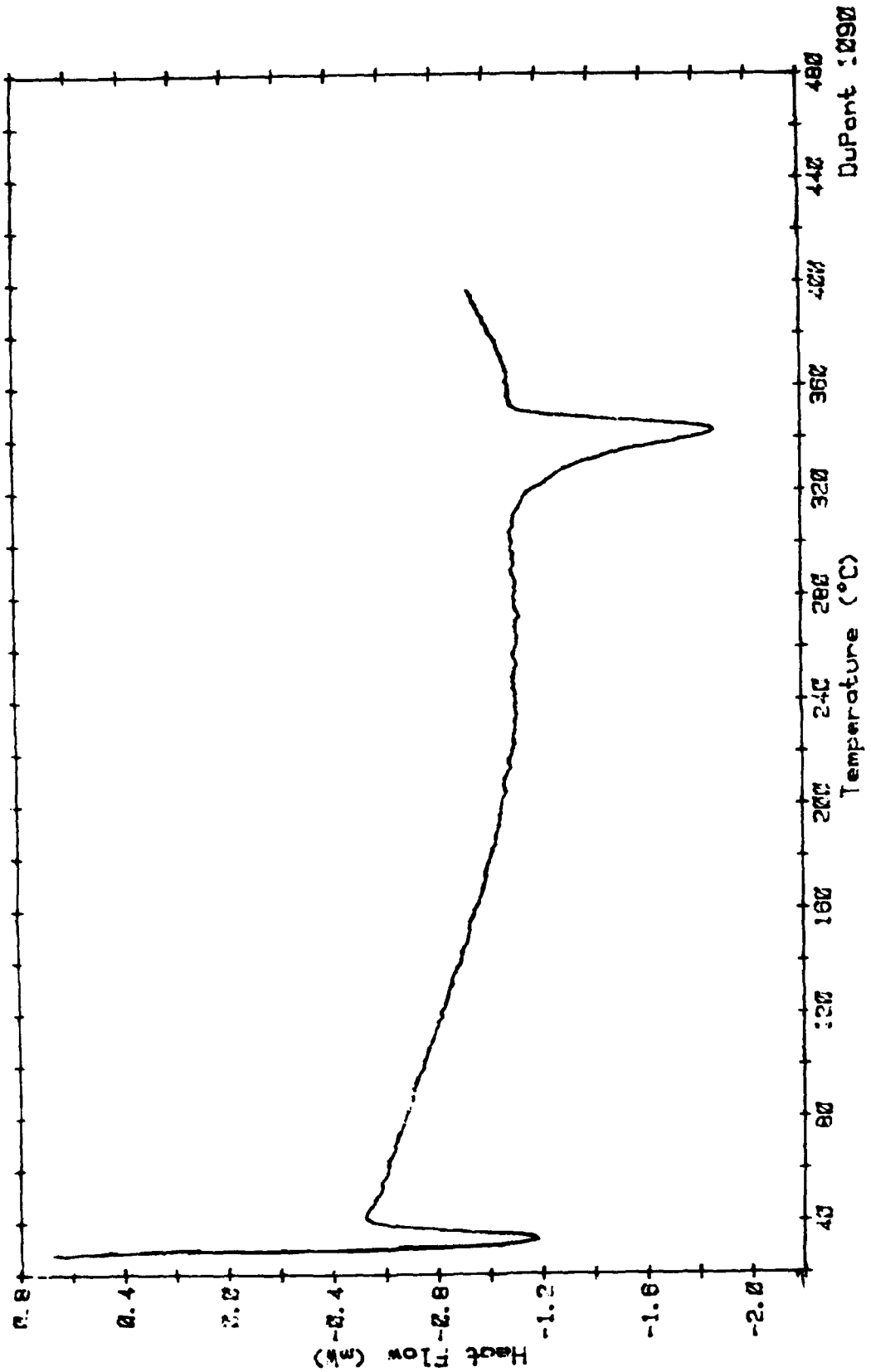
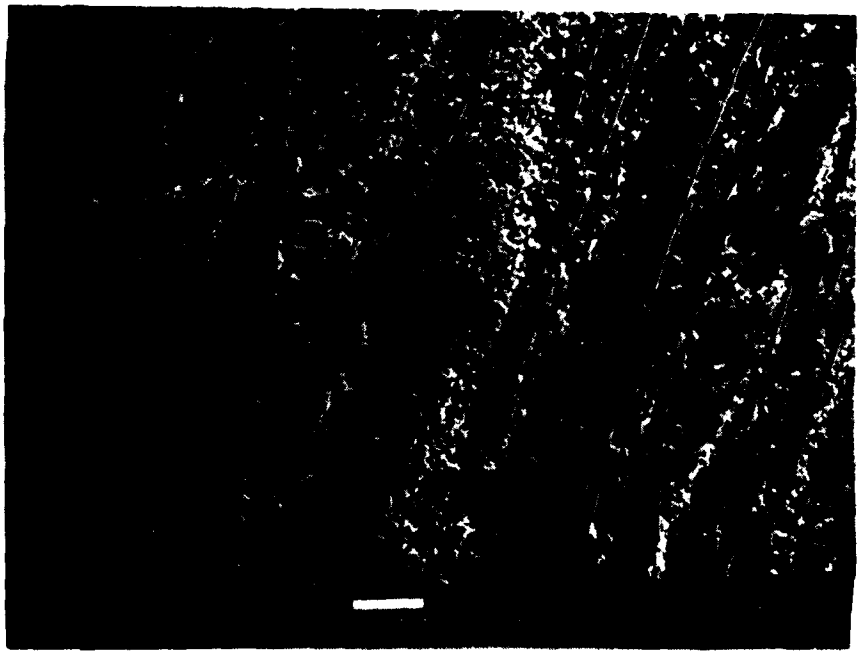
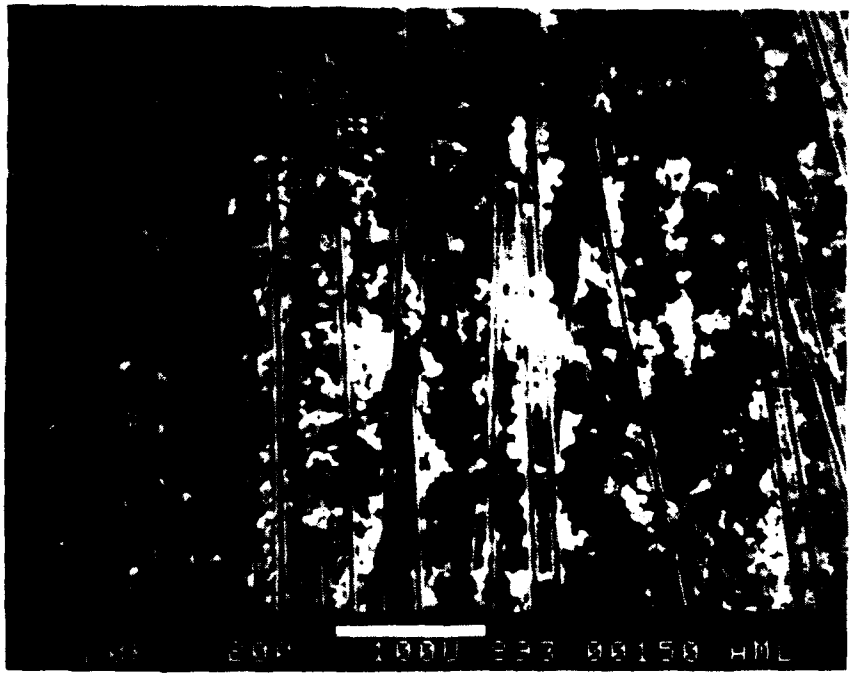



Figure 7 DSC of APC-II.

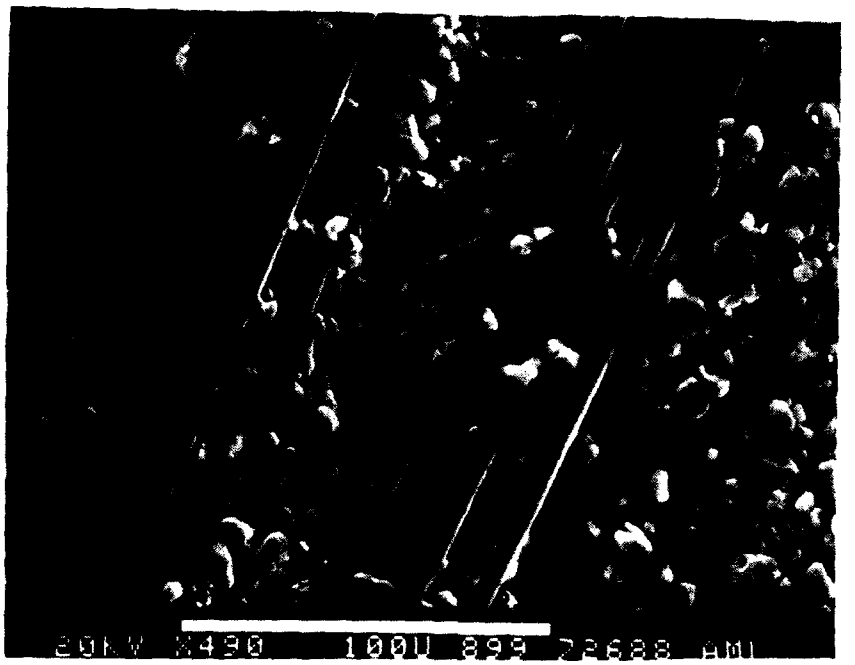


100 X



200 X

Figure 8 Electron micrographs of PEEK 150 powder preform.



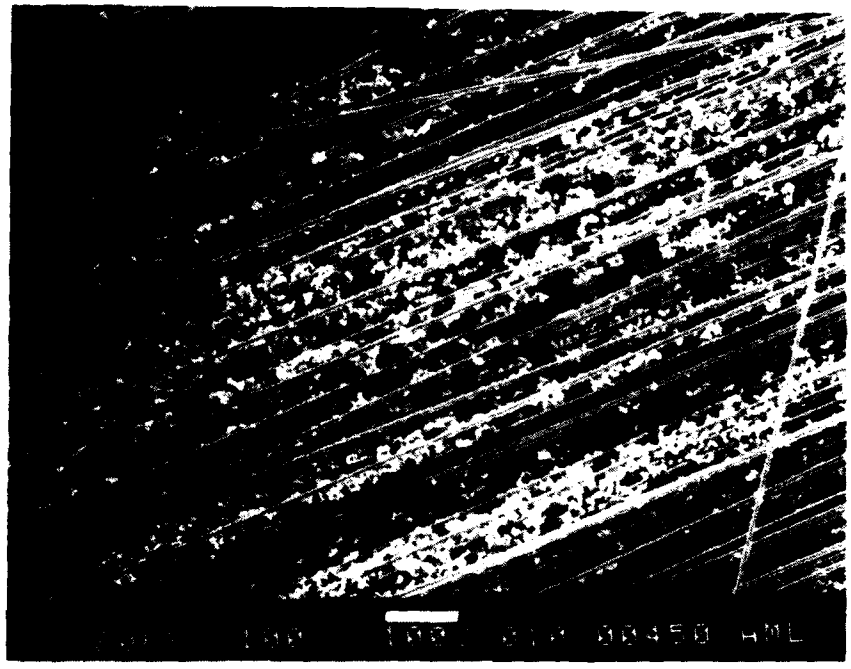
490 X

1000 X

Figure 9 Electron micrographs of PEEK 150 powder preform.


1900 X

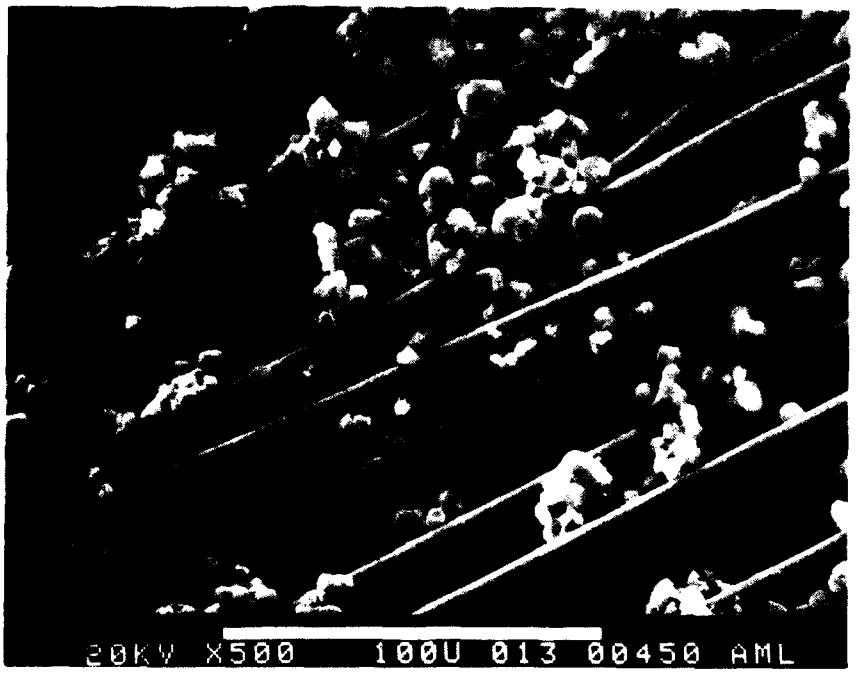
Figure 10 Electron micrograph of PEEK 150 powder preform.



100 X


200 X

Figure 11 Electron micrographs of PEEK 450 powder preform.




500 X


1000 X

Figure 12 Electron micrographs of PEEK 450 powder preform.


2000 X

Figure 13 Electron micrograph of PEEK 450 powder preform.

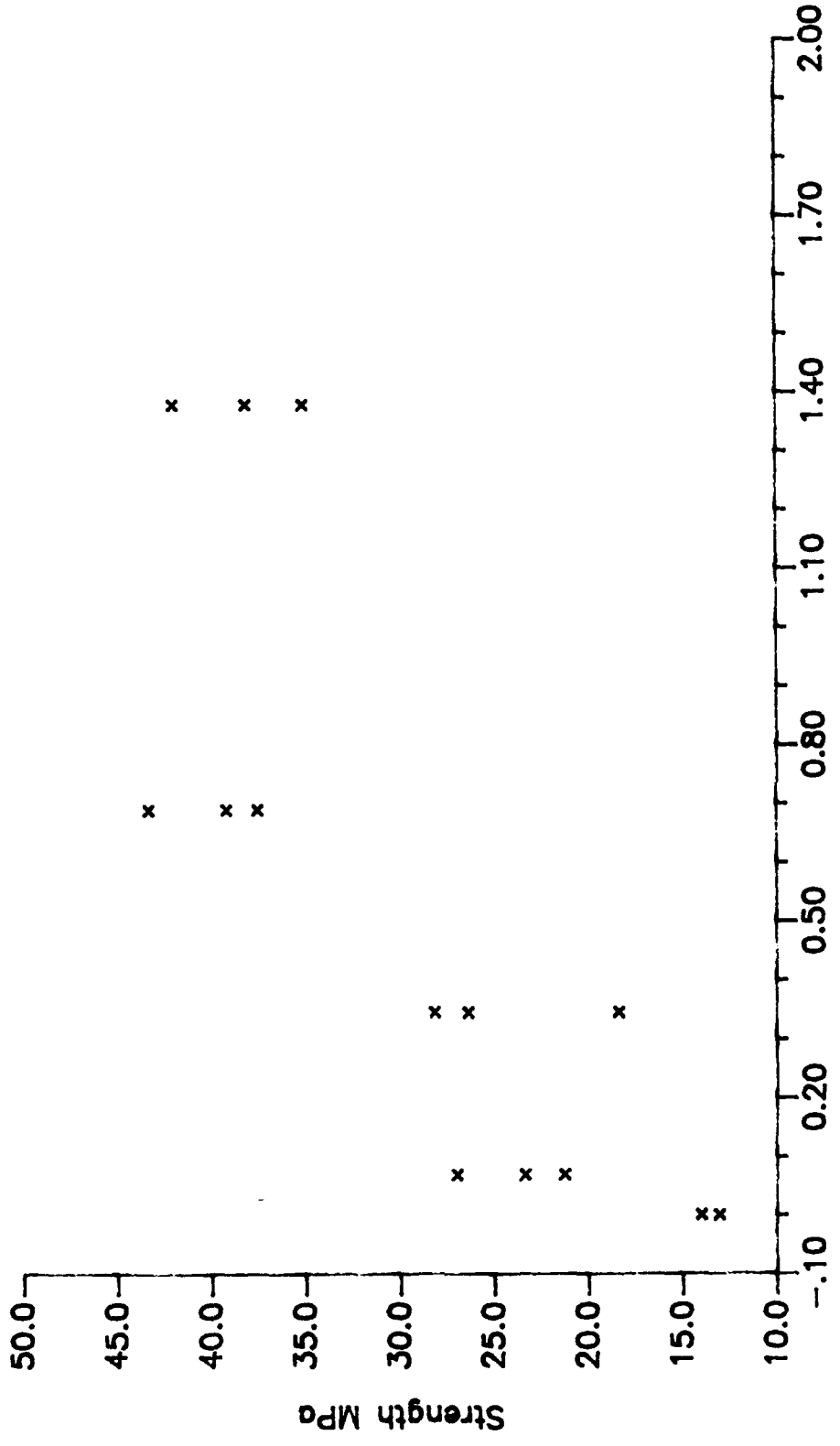



Figure 14 Effect of consolidation pressure on transverse tensile strength of powder preform laminates.

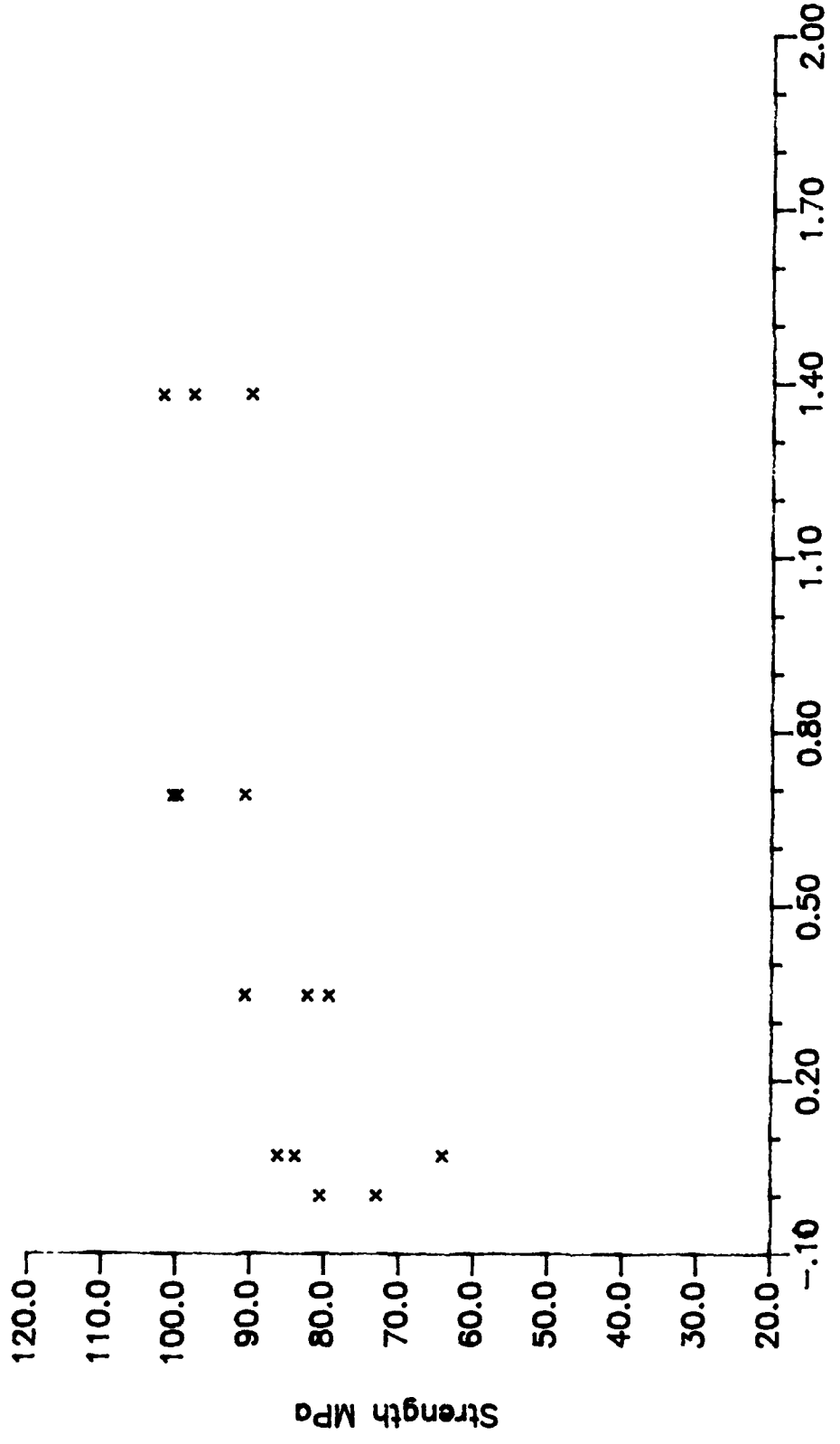



Figure 15 Effect of consolidation pressure on interlaminer shear strength of powder preform laminates.

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