

Characterization of Coatings of Poly (hexamethylene adipamide) Deposited on Carbon Fibers by Interfacial Polymerization Techniques

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SYNOPSIS

Polyamide coatings deposited on carbon fibers by means of an interfacial *in situ* polymerization technique have been examined. Two different coating processes were used, depending on the length of the carbon fibers (short or long). The effect of the concentration of the monomer reactants on the quantity of coating deposited on the fibers has been determined using thermogravimetric analysis (TGA). Characterization techniques used to identify the nature of the coating process product included Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), differential scanning calorimetry (DSC), and gel permeation chromatography (GPC). Using the above techniques, IR spectra were collected and identified, and important polymer properties, such as morphology, melting point, and the molecular weight of the polymer coating were determined. © 1995 John Wiley & Sons, Inc.

INTRODUCTION AND BACKGROUND

The mechanical properties of composites depend not only on the properties of each component but also on the nature of the interfacial bonds, and the mechanism of load transfer at the interphase. Stress transfer at the fiber matrix interface requires a strong interfacial bond between the two components. However, improvement of interlaminar shear strength (ILSS), which is considered by many researchers as a measure of adhesive bond strength, usually is combined with a decrease in impact strength and fracture toughness. This is attributed to the fact that too strong adhesion can limit the energy absorption mechanisms of fiber debonding, pullout, and postdebonding friction during the fracture process, leading to a brittle failure of the composite.¹⁻³

Considerable effort has been made to improve the interaction between the resin matrix and the fiber

surface. In particular, for carbon fiber composites, various surface modification techniques have been developed to promote interfacial bonding. Oxidative treatment of carbon fibers is the most commonly used modification method of fiber surface and is a standard treatment for commercially available carbon fibers. Oxidation methods, carried out in either gas or liquid phase, remove the original weak outer layer from the fiber surface and provide an increased fiber surface area as a result of microetching. In addition to that, oxygen-bearing groups are introduced on the fiber surface that interact with the polar matrix.⁴⁻⁷ Significant improvements in the ILSS have been reported with a simultaneous decrease in impact strength. Moreover, weight loss of the fibers and reduction of fiber strength along with possible formation of voids and flaws near fiber surface have been observed.^{5,6,8}

An additional standard treatment of commercial fibers involves the application of sizings on the fiber surface. Sizings can be mixtures of different additives, usually epoxy prepolymer based, that are applied on fibers by solution or emulsion coating. Their main purpose is to enhance handling and processing

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of the fibers. The effect of those sizings is not completely understood as far as composite properties are concerned. It has been reported⁹ that epoxy coatings on carbon fibers before the incorporation in an epoxy matrix lead to the formation of a brittle third phase between fiber and matrix, and to an increase in the interfacial shear strength of the composite.

An alternative approach to the interface modification problem is to coat the fibers with a thermoplastic polymer, which forms a flexible interlayer between fiber and matrix. Such coatings are likely not only to affect adhesive bond strength but also to form a phase of chemical and mechanical properties different from those of the bulk resin. The thermoplastic coatings possess advantages over traditional sizings in that they would form microductile layers at the interface. This type of an interphase would absorb impact energy by deformation and function as a stress relief medium to reduce residual stresses caused by matrix shrinkage occurring during curing. Composite properties that need to be optimized by the coating process are the adhesive bond strength and the fracture toughness. These properties depend on physical and mechanical properties of the interlayer introduced by the coating. The glass transition temperature and the thickness of the flexible interlayer have been identified as the major parameters affecting the end properties of the composite.⁹

Several techniques have been used for this type of interface modification: coating of carbon fibers with a thermoplastic by electropolymerization utilizing the high conductivity of carbon,^{2,3,10-12} solution dip-coating of carbon fibers with a crosslinkable elastomeric adduct¹³ or a silicon rubber,¹⁴ coating with a polyurea⁸ or a polyimide by some form of interfacial polymerization starting from monomer reactants.¹⁵

Interfacial polymerization as a method of coating offers exceptional flexibility and breadth in the range of polymers and their properties as well as an approach that may be used under practical processing conditions. The process involves transformations that are irreversible under the conditions of the reaction and it is, therefore, a typical case of non-equilibrium polycondensation. The essence of interfacial polycondensation, established as a synthesis method by Morgan,^{16,17} lies in the monomers being dissolved in two immiscible phases, an organic and an aqueous one. The two phases are brought in contact and the reaction proceeds either at the interface or in the organic phase, depending on the relation of the chemical reaction rates and the rate of diffusion of the diamine.^{1,16,17,18}

The success of the interfacial polycondensation as well as the reaction rate are determined to a great extent by the correct choice of the organic solvent. The organic solvent determines the distribution of the reagents between the phases, the surface tension between the phases, the diffusion rate of reagents, the swelling capacity and permeability of the formed polymer, and the extent of side reactions. Nonpolar solvents favor very rapid reaction, resulting in a diffusion-limited process because the reaction rate is considerably higher than the interpenetration rate of reacting substances. Common features of very rapid polycondensation reaction in a liquid-liquid system are low activation energy values and strong exothermicity. Moreover, they are conducted, as a rule, at room temperature. This allows for the use of thermally unstable monomers, protects unsaturated bonds and other reactive groups in the macromolecules, and prevents the thermal destruction of the polymer. The polymerization reaction can be described as a second-order reaction with a rate constant greater than 10^3 liter/mol s.^{1,16,18,19}

The influence of different factors on the polymer molecular weight is determined by the relationship between the rates of chain propagation and termination, i.e., by the contribution of side reactions. As a rule, the ratio between hydrolysis, which is the main side reaction, and chain propagation is very low. Moreover, solubility of the polymer in the organic phase must be such that polymer chain growth should be completed before precipitation begins. A broad range of molecular structures and polymer properties can be achieved varying the parameters affecting the polycondensation reaction. The most important parameter affecting polymerization is the ratio K between the molar concentrations of the two reactants in their respective solutions. A change in the concentration of the monomers can noticeably affect the molecular weight distribution (MWD) of the polymer. The optimum polymer molecular weight is reached with a nonequimolar ratio of reagents. Another crucial condition of successful interfacial polycondensation is the use of acid acceptors in order to neutralize the hydrochloric acid evolved in the course of the reaction because it may cause termination of the macromolecular chain. Therefore, the presence of an acid acceptor in the aqueous phase is one of the prerequisites for the formation of polymer at the interface. The quantity of acceptor should not be lower than that required to neutralize the evolving acid, and may even exceed this quantity.^{1,16}

Polyamides are among the polymers that can be produced by interfacial polymerization. We have shown that chrysotile (asbestos) fibers coating can

be achieved by a technique based on the principles of interfacial polymerization.²⁰⁻²³ Hexamethylenediamine was dissolved in water to form the aqueous phase, and adipoyl dichloride was dissolved in carbon tetrachloride (a nonpolar solvent) to form the organic one. Serial application of the two solutions on asbestos fibers and the incorporation of those fibers in an epoxy resin resulted in very improved composite tensile properties.

This work was later extended to continuous carbon fibers. Epoxy-based unidirectional composites reinforced with coated fibers were tested with satisfactory results.^{24,25} The impact strength was enhanced, whereas the ILSS remained practically unchanged. Moreover, ionomer-based composites reinforced with polyamide-coated short carbon fibers were prepared, resulting in improved mechanical properties.²⁶ In order to explain the enhancement in composite properties achieved by the polyamide coating, an understanding is needed of the relationship of structure and properties of the coating material to the coating techniques employed. In this work, we report characterization results for polyamide films obtained during coating of short or long carbon fibers.

EXPERIMENTAL

Materials

Continuous carbon fibers used in the present work were PAN-based 6K HTA-6000 Tenax fibers from ENKA AG. Short fibers were chopped to a length of 3 to 5 mm from the continuous tows. Hexamethylenediamine, adipoyl chloride, sodium carbonate, and carbon tetrachloride were all proanalysis grade reagents from Fluka AG.

Coating Techniques

Two methods were used for polyamide coating, based on the principles of interfacial polymerization. In both methods, aqueous solutions were produced by dissolving hexamethylenediamine and sodium carbonate in water and organic solutions by dissolving adipoyl dichloride in carbon tetrachloride. Sodium carbonate was used to neutralize hydrochloric acid, which is a side product of the polymerization reaction and is added at a molar ratio of 2 ($\text{Na}_2\text{CO}_3/\text{diamine}$) to the diamine. The ratio of the molar reactant concentrations (diamine/dichloride) was kept constant at 2.5, which gave good results in terms of mechanical properties of the composite in the case of asbestos fiber.²⁰ The effect of diamine

concentration on the quantity and quality of the polyamide coating was examined by a series of coating runs on both short and continuous carbon fibers. The diamine concentration levels used for different coating runs of short fibers were 10.25%, 17.11%, 23.93%, and 41.06% w/v (grams of hexamethylenediamine per 100 mL of water), whereas for the long fibers were 1%, 2%, and 3% w/v. Concentration levels of different order of magnitude were used in the two cases because of the two different coating processes employed, as established previously.^{24,26} The processing methods are described below:

Short Carbon Fiber Coating

Fibers were treated with the aqueous solution for 30 min in a Werner mixer to ensure complete wetting. Subsequently, the organic solution was added into the Werner mixing chamber. Mixing was continued for 30 min, after which the reaction product was dispersed in 10 liters of water and then washed thoroughly in a centrifugal separator until a neutral pH was reached. Finally, the fibers were dried in a vacuum oven at 80°C for 2 h.

Long Carbon Fiber Coating

Tows were wound around a Teflon[®] frame producing a regular array of parallel and aligned fibers. The assembly was immersed in a bath containing the aqueous solution. The fibers were removed from the bath and the excess of the diamine solution was allowed to drain. Afterwards, the fibers were immersed for 2 min in the organic solution, where the interfacial polyamidation took place. Finally, the fibers were washed with excess of water until neutral pH, and they were dried in a vacuum oven at 80°C for 2 h.

Characterization Methods

A variety of tools were used to examine the composition, uniformity, and thickness of the coating.

A Nicolet 20DXB-IR FT-IR infrared spectrophotometer was used to verify the composition of the coating. Thin coating films were extracted from coated continuous fiber arrays and were sandwiched between 2 KBr discs. The IR chamber was purged with dry air during the experiments. The spectrometer was equipped with a triglycine sulfate (DTGS) detector. A resolution of 4 cm^{-1} was used and 10 scans were taken.

The morphology and the microlevel homogeneity of the coatings on the fibers was examined by scanning electron microscopy (SEM). SEM micrographs were obtained by a ISI WB-6 and a Philips 501 SEM

instrument at 29 KV. A Denton Vacuum DESK-1 unit was used to deposit a thin layer of metallic gold. This film, while so thin and conforming so that it does not significantly reduce resolution, is electrically conductive enough to prevent the sample from charging. Sputtering was used for this application instead of vacuum evaporation because sputtering coats uneven surfaces much more completely.

The weight of the polyamide coating was determined by thermogravimetric analysis (TGA) on a Du Pont 951 thermogravimetric analyzer under nitrogen flow. Samples of about 30 mg were tested and the temperature was ramped to 500°C with a heating rate of 10°C/min. At least 12 samples were tested for every coating corresponding to a different concentration of diamine.

The melting points of coatings scraped from the surface of short and long carbon fibers was determined by differential scanning calorimetry (DSC) by means of a Du Pont 910 differential scanning calorimeter. The temperature was ramped to 300°C with a heating rate of 10°C/min. Nitrogen purge was used in all the runs, and all the samples had a weight of about 3 to 10 mg.

The molecular weight of the coating was determined by gel permeation chromatography (GPC). A SHODEX GPC HFIP 80M column was employed with a Waters GPC. The solvent used was hexafluoroisopropanol (HFIP) with sodium trifluoroacetate. The weight and number average molecular weight (M_w and M_n , respectively) were measured as well as the polydispersity factor defined as the ratio of M_w over M_n . The polydispersity factor gives an indication of the breadth of the molecular weight distribution.

RESULTS AND DISCUSSION

Infrared Spectroscopy

Extensive studies^{27,28} on the infrared spectra of simple amides have shown that two characteristic peaks, commonly referred to as amide I and amide II, appear in the infrared spectra of both synthetic and natural polymers, such as polypeptides containing the secondary amide group—CONHR. In particular, the amide I peak located at about 1640 cm^{-1} for Nylon 6,6[®] in the solid state, has the C=O stretching mode as its major component, but there is almost certainly some mixing with the in plane N—H deformation vibration and possibly with the C—N stretching vibration. Amide II at 1540 cm^{-1} , is largely the in plane N—H deformation vibration,

but it is very possible that it involves the $\nu(\text{C}=\text{O})$ and $\nu(\text{C}-\text{N})$ vibrations. Amide II also has an overtone at 3070 cm^{-1} .

Despite their complex origin, these two modes give good group frequencies as does amide III at about 1280 cm^{-1} , whose origin is also probably complex. In addition, the secondary amide group shows a characteristic peak for N—H stretching at 3310 cm^{-1} . Together, these peaks provide a sure method for the identification of Nylons as a class.²⁸ Figure 1 shows the infrared spectrum of the polyamide coating deposited on carbon fibers by interfacial polymerization. The characteristic peaks of Nylons amide I, II, and III together with the peak at 3310 cm^{-1} are all present in our spectrum.

Scanning Electron Microscopy

The morphology and the microlevel homogeneity of the coating were examined by SEM. Scanning electron micrographs of control and coated long fibers are shown in Figures 2 and 3, respectively. The fiber bundle before treatment seems to be composed by many discrete and very well-aligned carbon fibers. On the contrary, the coated carbon seems to be composed of fibers entrapped into a surrounding polymer membrane. It is very likely that only the outer fibers are covered by polyamide, but the exact percentage of fully coated fibers is hard to estimate. Moreover, there is some small agglomeration of the coating polymer that occurs to a greater extent between the fibers than on their surface. The fiber alignment does not seem to have been greatly affected by the coating process.

Figure 4 shows details of the surface of a longitudinal section of the "as received" single fiber. The surface is very smooth, and some striations along

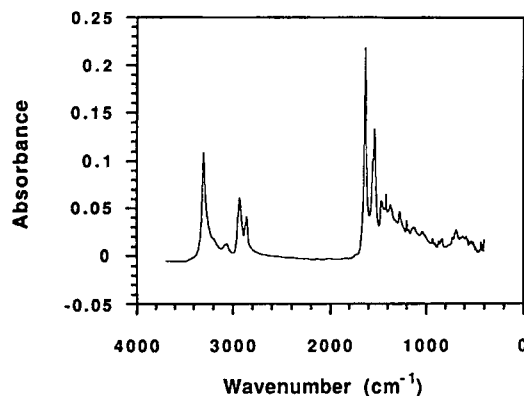


Figure 1 FT-IR spectrum of polyamide coating scraped from the carbon fibers surface.



Figure 2 SEM micrographs of as-received Tenax carbon fibers. Magnification $\times 320$.

the fiber direction can be easily seen. The diameter of the fiber was measured to be $6.9 \mu\text{m}$, which is in good agreement with the nominal fiber diameter of $7 \mu\text{m}$ quoted by the manufacturer. In Figure 5, we see details of the surface of a coated single fiber. The coating perfectly covers the entire fiber and appears to be quite homogeneous. No cracks on the coating surface have been observed, suggesting that the polymer is rather flexible. The texture of the coated surface seems to be fairly rough and wavy. The thickness of the layer adjacent to the fiber was estimated from the electron micrograph to be approximately 400 nm . So, the total diameter of the coated fiber is $7.7 \mu\text{m}$.

Figure 6 suggests that the polymer coating on the short fibers is inhomogeneous. It appears that not all of the fibers are evenly and completely coated, and there are some areas with comparatively large agglomerated material, which occasionally surrounds and entraps several fibers together. This behavior can be attributed to the fact that the technique employed in the short carbon fiber case resembles the bulk interfacial polymerization under stirring. Insufficient stirring can lead to the formation of agglomerated material and incomplete coating of the fibers. However, it must be emphasized that the mechanical performance of the short fiber-

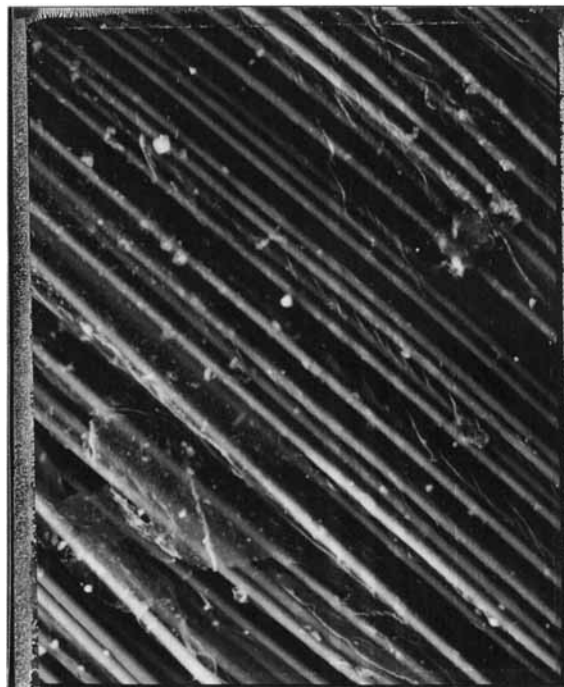


Figure 3 SEM micrographs of polyamide-coated long carbon fibers (1% w/v Diamine). Magnification $\times 320$.

based polymer composites shows significant improvement, despite the apparently insufficient coating.²³

Thermogravimetric Analysis

Figures 7 and 8 show the effect of changing the concentration of reactants on the amount of polyamide

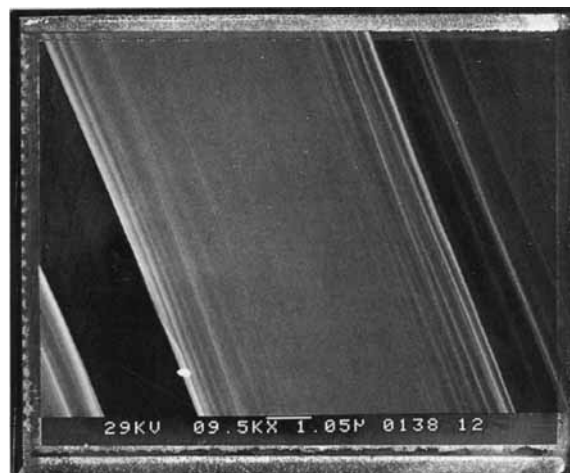


Figure 4 SEM micrographs of an as-received single carbon fiber surface. Magnification $\times 9500$.

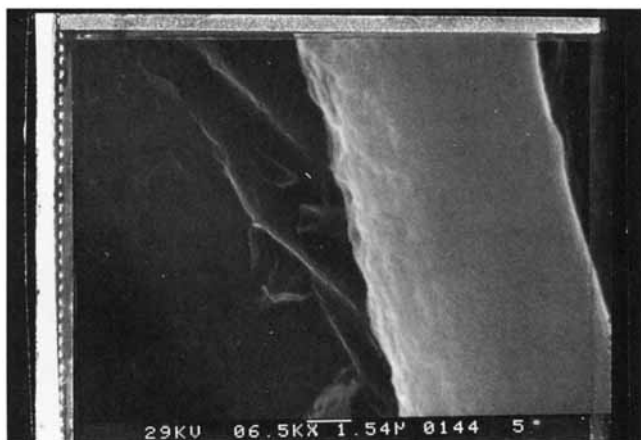


Figure 5 SEM micrographs of the surface of a polyamide-coated single long carbon fiber (1% w/v Diamine). Magnification $\times 6500$.

deposited (expressed in grams of polymer per 100 grams of uncoated carbon) on the short and the long carbon fibers, respectively. In both cases, the weight gain increases linearly with the increase of the reactants concentration. However, in the case of the long fibers, the weight of the coating is more sensitive to a change in the diamine concentration because the

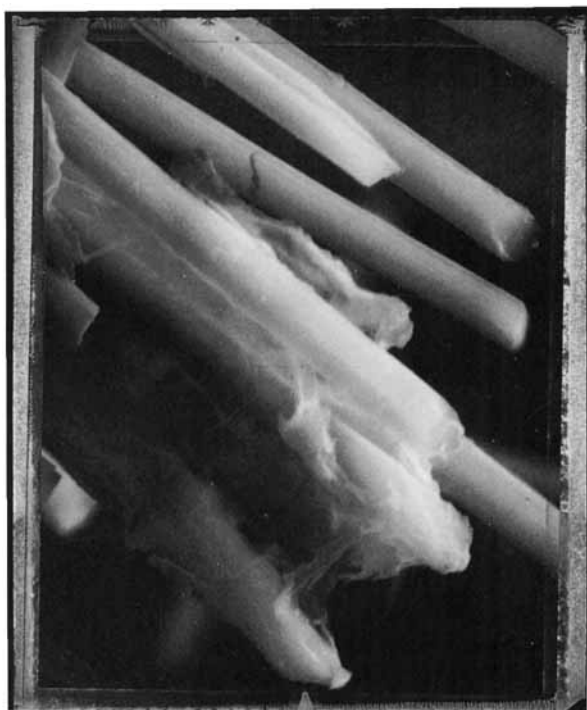


Figure 6 SEM micrograph of polyamide-coated short carbon fibers (41.06% w/v Diamine). Magnification $\times 1250$.

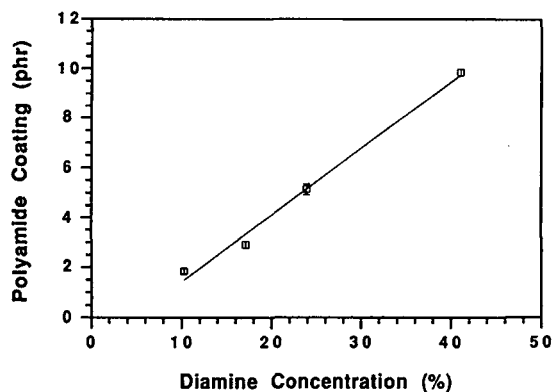


Figure 7 Polyamide content deposited on short carbon fibers vs. diamine concentration.

slope of the line is about 6.5 times higher than that of the short fibers.

The macrolevel homogeneity of the coating is very important when utilization of the carbon fiber as reinforcing material for composites is considered. The homogeneity of the coating along the fibers was examined by taking TGA samples at random locations in at least three arrays of parallel and aligned coated fibers, for every different concentration level. For the short fibers, samples from different batches of coated carbon (same concentration level) were taken. As we can see from the standard deviation of these measurements (around 9% and 8% for the long and the short fibers, respectively) the coating is quite homogeneous on the macrolevel.

Differential Scanning Calorimetry

The melting temperatures of polymer coating scraped from the surface of long and short carbon

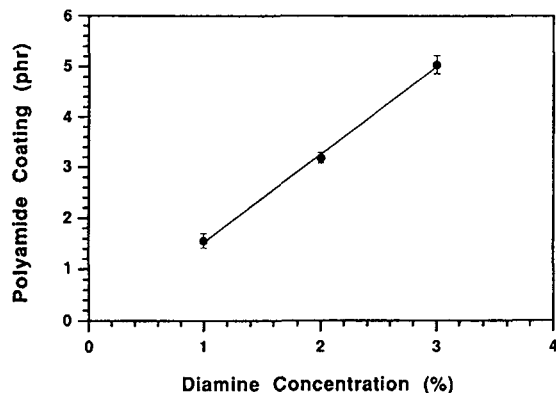


Figure 8 Polyamide content deposited on long carbon fibers vs. diamine concentration.

fibers were approximately 252°C and 251°C, respectively, and they were both independent of the diamine concentration level. The melting points reported here are taken as the minimum of the melting peaks on the DSC thermogram. These values are within the range of melting points of commercial polyamides.¹⁸ However, the commercial poly(hexamethylene adipamide) known as Nylon 6,6[®] melts at about 10°C higher temperature. The lower melting point of the polyamide coating is indicative of a low molecular weight polymer.

Molecular Weight Distribution

The features of the MWD are expected to be dependent upon the diamine concentration and the stirring conditions.

Long Fibers

According to the mechanism of interfacial polycondensation without stirring, the formation of the polymer initially occurs at the phase interface and starts to penetrate into the phase which, of the two, is the better solvent for the starting substances; in our case, it is the carbon tetrachloride phase.^{16,29} The reaction rate exceeds the diffusion rate of diamine. Hence, the mass transfer of the diamine is the rate-controlling step of the polymerization process.¹⁶ As the diamine diffuses into the organic phase, it encounters a layer consisting of the oligomers and the adipoyl dichloride itself. As the concentration and the size of polymer species increases, interchain contacts increase until a network is formed and precipitation results. The absence of major diffusion limitations, at the first stage of the reaction, leads to the formation of a dense polymer film.¹⁹ By the time a coherent film precipitates, polymerization does not stop entirely, but the rate is greatly decreased because of the lower mobility of the polymer chains, the increased distance from the interface, and the decreased diffusion rate of diamine. Although the initial polymer reaches its maximum molecular weight, there is a secondary growth of low molecular weight polymer in the free space of the precipitate and upon the face of the film in the organic phase. This leads to a lowering of the average molecular weight and a broadening of MWD. At the final polymer coating, the layer adjacent to the fiber surface presumably contains the initially formed high molecular weight fraction, whereas a lower weight fraction has formed within the organic phase away from the interface.^{16,19,30}

Figure 9 shows a typical GPC chromatograph for

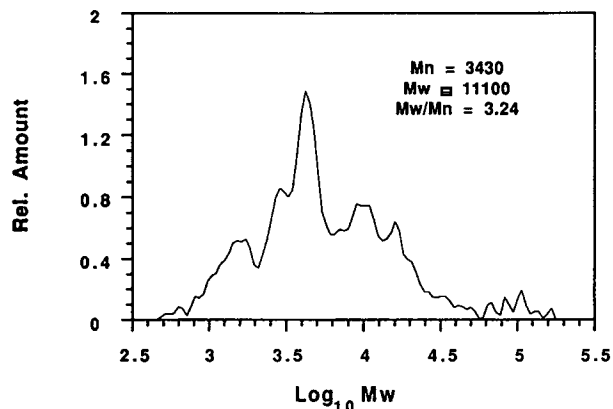


Figure 9 Typical GPC chromatograph for polyamide coating on the surface of long carbon fibers (1% w/v diamine).

the polyamide coating. The MWD appears to be nonuniform and exhibits large low molecular weight fractions (peaks at M_w equal to approximately 1500 and 2900) leading to nonstatistical distributions and low average molecular weights. This is not a surprising result for polymerizations carried out under nonequilibrium conditions in two-phase liquid systems from which polymer precipitates instantaneously, if we consider the mechanism discussed previously along with findings of other researchers.^{16,30}

Figure 10 indicates that higher diamine concentrations lead to lower average molecular weight. Figure 11 shows that the polydispersity factor increases as the diamine concentration increases, i.e., the MWD becomes broader.

These results can also be explained by the aforementioned principles of the unstirred interfacial polycondensation. Initially, when there is no polymer formed and, therefore, there are no diffusion limitations, an increase in the concentration of monomers increases the molecular weight of the polymer. However, an increase in the concentrations of the reactants in the bulk does not correspond necessarily to higher local concentrations at the boundaries of the reaction zone. On the contrary, the more dense polymer film formed at the first stage of the polymerization will retard diffusion. Consequently, this results in lower concentrations at the boundaries of the zone, i.e., lower average molecular weight and broader MWD.^{16,19,31}

Short Fibers

Stirring accelerates the process of polymer formation, and increases the molecular weight of the poly-

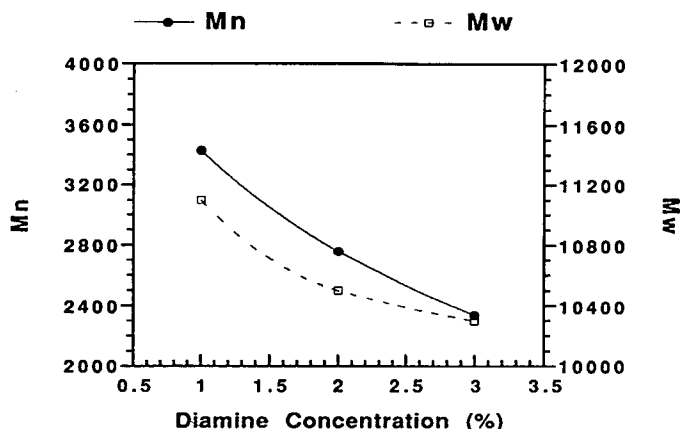


Figure 10 Relation of weight-average molecular weight (M_w) and number-average molecular weight (M_n) to diamine concentration for polyamide 6,6 made by unstirred interphase polycondensation on the surface of long carbon fibers.

amide. This is due to the increase in the availability of the diamine for polymerization and the increase in the reaction surface.¹⁶

The MWD of the polyamide is now quite smooth but significantly broader than random (Fig. 12). This result is consistent with the findings of other researchers¹⁶ and may be attributed to the fact that stirring is not vigorous enough to produce a very fine dispersion of droplets of the organic phase or to tear continually the forming films from larger droplets and provide fresh interface. As a result, low polymer is formed inside the film-coated solvent drops according to the mechanism that was previously mentioned for the unstirred interfacial polycondensation. In addition, the polymerization conditions may vary widely from start to finish of a batch polymerization both in terms of stirring and

concentration ratios of the reactants. Therefore, as the reactant present in lesser quantity is consumed, the proportion in excess becomes relatively greater so that the last polymer to form may be made under unfavorable conditions. Eventually, the stirred system is run to the point of consumption of one of the reactants. On the contrary, in the case of the unstirred system, the reactants are always near to the starting concentrations because the coated fibers are removed from the reaction mixture quickly. That explains why the molecular weight distribution is much broader for the stirred system. However, a direct comparison between the two methods is difficult because the concentration level of the monomer reactants is different.

Figure 14 indicates that higher diamine concentrations lead to a decrease in the polydispersity fac-

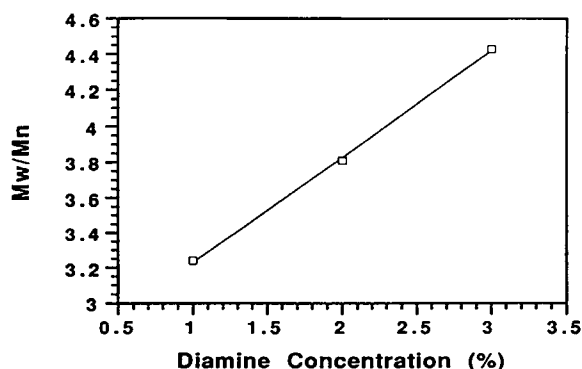


Figure 11 Relation of polydispersity factor (M_w/M_n) to diamine concentration for polyamide 6,6 made by unstirred interphase polycondensation on the surface of long carbon fibers.

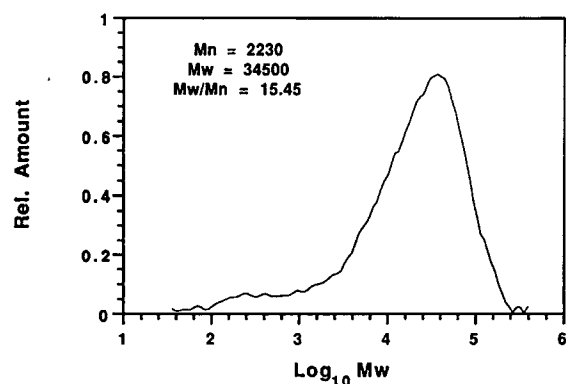


Figure 12 Typical GPC chromatograph for polyamide coating on the surface of short carbon fibers (10,25% w/v diamine).

tor, i.e., narrower MWD. An increase in diamine concentration results in a decrease in the M_w , whereas the M_n increases (Fig. 13). Because the weight average is particularly sensitive to the presence of larger species, whereas the number average is sensitive to the proportion by weight of smaller molecules, these results mean that the number of both very small and the very large molecules decreases with more concentrated solutions.³²

This behavior may be attributed to the fact that higher concentration levels lead to poorer stirrability of the system. Consequently, the availability of diamine in the reaction zone is decreased, resulting in smaller macromolecules, i.e., lower M_w . On the other hand, more dilute solutions presumably favor the formation of larger amounts of cyclic oligomers,¹⁶ resulting in lower M_n .

CONCLUSIONS

The *in situ* interphase polyamidation of poly(hexamethylene adipamide) on carbon fibers was investigated, as an example of interfacial polycondensation coating. The main advantage of coating fibers by means of interfacial polycondensation is that it makes it possible to obtain a wide variety of polymer coatings (such as polyamide coatings), which would be difficult to obtain by an alternative high temperature coating technique. These polymer coatings involve thermoplastics of a high melting point, which is often higher than their decomposition temperature, or polymers that are based on thermally unstable monomer reactants.

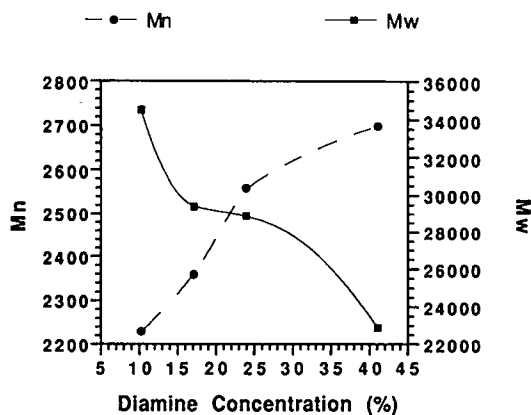


Figure 13 Relation of weight-average molecular weight (M_w) and number-average molecular weight (M_n) to diamine concentration for polyamide 6,6 made by interphase polycondensation under stirring on the surface of short carbon fibers.

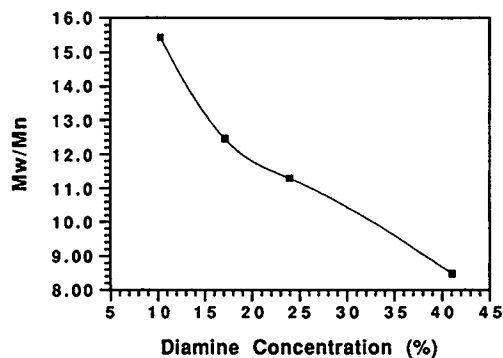


Figure 14 Relation of polydispersity factor (M_w/M_n) to diamine concentration for polyamide 6,6 made by interphase polycondensation under stirring on the surface of shong carbon fibers.

Moreover, the application of polyamide coatings from solutions involves difficulties due to the low solubility of homogeneous polyamides in common solvents. On the contrary, in the case of interphase polycondensation, the solutions employed have very low viscosity and the whole process can be carried out very rapidly, using a very simple apparatus. The simplicity of the coating procedure allows the continuous treatment of long tows of fibers.

In view of utilizing coated short and long carbon fibers as reinforcements for composite materials, the polyamide nature of the coating was identified by differential scanning calorimetry and infrared spectra analysis. Thermogravimetric analysis was used to measure the weight of polyamide deposited on the fibers. Scanning electron micrographs revealed a quite homogeneous coating of the long fibers, whereas short fibers seemed to be not evenly and fully coated. The molecular weight of the polyamide deposited on the surface of long carbon fibers (case of unstirred interfacial polyamidation) was around 10000 (M_w), while stirring in the short fibers system allows the formation of higher molecular weight polyamide (around 30000). Number average molecular weights were consistently low, leading to broader than random molecular weight distributions.

Although this coating seems to enhance the mechanical properties of the composite material, optimization and better control of the molecular weight of the polyamide deposited on the fibers is expected to improve even further its mechanical performance. Therefore, the effect of some crucial parameters, such as the organic solvent used and the molar concentration ratio of the two reactants in their respective solutions, on the molecular weight needs to be investigated.

The development of a laboratory scale unit for the continuous *in situ* coating of carbon fibers with polyamide 6,6 using a winding mechanism is in progress.

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