TITLE: Fundamental Studies **for the Utilization of Carpet Waste**

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RELEVANCE TO NTC GOALS

One of the NTC goals is to strengthen the nations textile and educational efforts by uniting diverse experts and resources in unique collaborative efforts. The union of a polymer chemist, a mechanical engineer (with a textile engineering undergraduate background), a polymer physicist, and a number of researchers in the textile and processing industries (e.g. Crown America, Reichhold, Shaw Industries, and Haake) to work toward the solution to an important textile waste problem is indeed an example of such a collaborative effort.

Another NTC goal is the development of new materials and improved manufacturing processes. The fabrication of carpet waste fiber reinforced concrete is an excellent example of the development of new materials with potentially important properties. The extrusion of mixtures of compatibilized polypropylene/nylon involves not only the development of new materials but also the development of innovative processing techniques with the cooperation of process engineers from industry.

Our interactions with industrial companies will facilitate utilization of the developed technologies. Also we are preparing publications of our results and a lecture will be presented describing our work at the Dalton Recycling Conference in September.

RESEARCH SIGNIFICANCE

The annual fiber consumption of the U.S. carpet industry is approximately 2.4 billion lbs. The most common fibers used by this industry and their annual consumption amounts are given in Table 1. Current carpet manufacturing practice involves, tufting the face fiber into a primary backing. The primary backing is bound to the secondary backing using styrene-butadiene-rubber latex (Figure 1). The latex binder holds the face fibers in place. Calcium carbonate is used as an economic filler in the latex, and does not contribute to the properties of the carpet. On curing, the SBR latex is crosslinked and hence becomes a thermoset. Nylon and polypropylene are the current common face fiber and the backing materials respectively. Carpet tiles and automotive carpets are generally made using thermoplastic binders; however, the different thermoplastic components even in these latter categories of carpets are not selected for their compatibility with each other. Therefore the recycling of even the current all-thermoplastic carpets is not straightforward. Over 60 million lbs of annual waste is generated in the carpet industry in Dalton, Ga. In the current work, the attempt is being made to utilize this waste. The suggestion that over half the annual carpet production is used to replace used carpet means that as much as one billion lbs of fiber may currently be going into the US landfills by way of used carpets. Used carpet will in general be heavily contaminated, while most industrial carpet may possibly be retrieved in relatively uncontaminated form. However we anticipate that methods can be developed to de-contaminate the used carpets in such a way that the recycling technologies developed for the industrial waste can be used for recycling the used carpet as well.

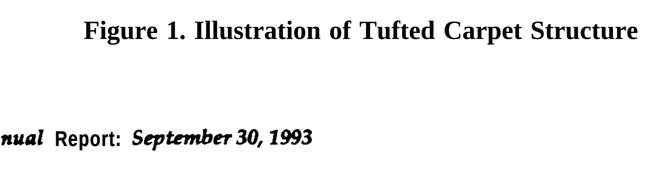

Figure 1. Illustration of Tufted Carpet Structure

TABLE **1:** Principal Fibers Used **in The Manufacture of Carpets**

Fiber Type	Million Pounds (199 1)
Nylon Filament	904
Nylon Staple	776
Olefm Filament	378
Olefm Staple	123
Polyester Staple	229
Total	***2410***

Source : Carpet and Rug Institute

The industrial waste generated before the application of the SBR latex adhesive is termed "soft waste" and the SBR containing waste is termed as "hard waste". The soft waste can be reused for filling purposes. Waste reduction and possible reutilization activities have been initiated over the past few years. Efforts are currently focused to develop cost effective technologies to recycle the carpet waste. A number of options are currently under evaluation, the principal ones being: (a) de-polymerization of the nylon (BASF, ZIMMER AG, Polk) [1,2,3]; (b) manufacture of recyclable single material carpets, based on polyester (Hoechst Celanese) [4]; (c) alloying/ homogenization of the hard waste through reactive extrusion. Shredded carpet waste is also being evaluated for concrete reinforcement [5].

Other interesting alternatives would be the safe and efficient pyrolytic destruction of carpet wastes using plasma arc technology [6] and effective separation of components using super critical solution techniques [7]. In the current work, attempts are made to develop different technologies to convert carpet waste into useful products, including nylon depolymerization, reaction extrusion of the waste, and concrete reinforcement with the waste. Further descriptions of the current status of these technologies will be provided in the next section.

PROGRESS SUMMARY

Significant progress has been made in all the three areas: depolymerization of nylon, extrusion of waste by compatibilization, and use of waste for concrete reinforcement. Results to date are summarized below.

Depolymerization of Nylon:

Backaround: Our main objective is to develop a low temperature, atmospheric pressure process for the depolymerization of nylon 6 and nylon 6,6 in carpet waste utilizing sodium hydroxide and phase transfer catalysis. In 1948, there was an Italian patent issued for the saponification of nylon 6,6 in an autoclave. Another Italian patent was issued in 1956 for the saponification of nylon 6,6 at temperatures of 150°C and above 25 atmospheres. In 1958, a US patent was issued to Miller at duPont for the hydrolysis of nylon waste with NaOH and isopropanol at 180°C and 305 psi to produce HMDA by distillation of the isopropanol and adipic acid by acidification of the aqueous phase.

More recently, BASF applied for a patent for the recycling of carpet. The BASF carpet recycling process consists of six steps: collection, separation, monomer recovery, depolymerization, spinning, and carpet manufacture. Monomer recovery involves the depolymerization of nylon 6 pellets using phosphoric acid and superheated steam. All of these depolymerization processes involved high temperatures and pressures.

Results and Discussion; A series of experiments were run in order to examine the applicability and efficiency of benzyltrimethylammonium bromide (BTEMB) as a phase transfer catalyst in the depolymerization of Nylon 6,6.

A known amount of polymer fiber was mixed with 200 ml of a 50% aqueous solution of sodium hydroxide and 0.2g of BTEMB in 5 different runs. The mixture was heated in a mineral bath at a bath temperature of 143°C and allowed to reflux for 24 hours with continuous stirring. After 24 hours, on cooling a white powder mixed with short whiskers settled down at the bottom of the flask. The solid was collected by filtration through a sintered filter funnel.

The residue was washed with distilled water at least four times until the washings were neutral to litmus paper. After drying in a vacuum oven overnight, the samples were weighed and stored.

The reduced viscosity of the depolymerized Nylon 6,6 was determined in 88% formic acid at 25°C in a Ubbelhode viscometer. The viscosity-average molecular weight was calculated from the equation:

$$[\eta] = K\, M_v^{a}$$

where Mv is the average molecular weight, K = 35.5 x 10^{-5} dl/g and a = 0.786

Table 2 shows the effect of the feed ratio of the Nylon 6,6 to BTEMB on the viscosity-average molecular weight of the depolymerized nylon.

TABLE 2. **Effect of Feed Ratio on Molecular Weight**

Experimental Run No.	*1*	*2*	*3*	*4*	*5*	*6*
Feed Ratio of Nylon 6,6 to BTEMB	5	10.3	20.6	29.4	59.8	-
Depolymerization Efficiency	40.3	49.5	55.5	42.5	40.8	15.9
Intrinsic Viscosity (dl/g)	0.114		0.134	0.122	0.160	0.119
Viscosity-Average Molecular Weight	1556		1912	1697	2396	1644

It was discovered that the product of the run with no phase transfer agent showed a 16% increase in weight compared to the weight of the original nylon 6,6. The oligomer obtained had a viscosity-average molecular weight of 1644 (the original nylon 6,6 had a molecular weight of 30,944). The other runs with phase transfer agent produced oligomers with decreases in weight of 40 - 50% in each case. These results suggest that in the absence of phase transfer agent only oligomers are formed; however, soluble low molecular weight products are formed in the presence of phase transfer agent.

Reactive Extrusion of Carpet Waste:

Background: It has been shown [8-10] that the maleic anhydride grafted polypropylene could be an effective compatibilizer for alloys of virgin polypropylene and nylon 6. Ide and Hasegawa [8] synthesized maleic anhydride grafted polypropylene and used it as a compatibilizer ("interpolymer") in blends of PP and nylon 6. They reported superior mechanical properties and a marked increase in dispersibility for the compatibilized blends. Park and coworkers [9] noted that the viscosity-composition curve, at low shear rates, indicated a negative deviation from the simple additive rule for PP/nylon 6 blends and a positive deviation for PP/nylon 6/MA-g-PP blends. The change in the nature of the viscosity-composition dependence has been attributed to the increased interaction between the phases in the presence of MA-g-PP. Grof et al. [10] studied the structure and properties of fibers made from PP/nylon 6 as a function of MA-g-PP content. They observed that the MA-g-PP acted as an interfacial agent and improved the processibility and properties of the fibers. Adur and Constable [11] showed that 15% MA-g-PP compatibilizer in 70/30 nylon/polypropylene resulted in 25% increase in the tensile strength over an uncompatibilized blend. Impact strength, % elongation and flexural modulus also increased. On compatibilization, the melt flow rate and the moisture absorption decreased significantly. Modic and Pottick [12] used modified styrenic block copolymers (Shell Kraton G 1901X and Kraton RP6530) as compatibilizers for PP/nylon blends and reported better low temperature impact performance as well as a higher tensile strength for the blends compared to MA-g-PP containing blends. Acrylic acid grafted PP has also been tried as a compatibilizer, however it has relatively lower degradation temperature [13] (235°C) as compared to MA-gPP. Liu et al. [14] have reported the synthesis of poly(2-alkyloxazoline-b-i-caprolactone) di-block copolymers, poly(ethyl vinyl ether)-poly(2-alkyloxazoline) graft copolymers, and poly(butyl vinyl ether)-poly(2-methyloxazoline) graft copolymers for use as compatibilizers in blends of PP and nylon 6. An ionomer with zinc counterion, poly(ethylene-co-methylacrylate-co-butylacrylate) - trade name Surlyn 9020TM, was also reported to result in enhanced mechanical properties of the polypropylene/nylon [15]. Several other workers also reported enhanced mechanical properties in the polypropylene/nylon system with the use of compatibilizers [16-18]. Utracki and Favis [19] contend that the use of polymer modifiers such as maleated polypropylene for the compatibilization of polypropylene/nylon blends probably does not involve the formation of covalent bonds between amide and maleic anhydride. Role of compatibilization in polymer miscibility has been discussed by Gaylord [20] and Noolandi [21], and that of the polymer miscibility in polymer recycling has been discussed by Stein [22].

Experimental: Two types of mechanically shredded industrial carpet wastes were obtained from Crown America Inc. One batch contained calcium carbonate filled SBR, while the calcium carbonate and the SBR were mechanically removed from the second batch. Maleic anbydride grafted polypropylene (PolybondTM 3002) was obtained from BP Chemicals. MA-g-PP had a melt flow rate (at 230°C and 2.16 Kg) of 7 g/10 min., density 0.91 gm/cm^3, tensile strength and ultimate elongation of 32 MPa and 436% respectively [13]. The shredded carpet fibers from the second batch, from which SBR latex and calcium carbonate had been removed, were melt blended and subsequently pelletized with and without 3 wt% compatibilizer using a Haake Rheocord 90, equipped with a TW 100 twin screw extruder. The extrusion conditions are listed in Table 3. In order to circumvent the difficulties arising from hopper feeding of fibers and to ensure compositional uniformity of the alloys, a "double pass" technique was used during the compounding. The second batch, from which SBR and the calcium carbonate had been removed was successfully extruded. Attempts to extrude the first batch, which contained the chunks of calcium carbonate filled thermoset SBR, were predictably unsuccessful.

TABLE 3. Extrusion Parameters

Temperature ("C)				Screw Speed
Zone 1	Zone 2	Zone 3	Zone 4	(RPM)
150	220	225	230	70

The extruded resins were compression molded at two different temperatures. The compression molding parameters are listed in Table 4. Stress-strain plots of the compatibilized blends were obtained on an Instron 1125 universal testing machine, using the compression molded test specimens (ASTM D1708). The tensile specimens were 0.19" wide and 0.04" thick, the gage length was set at 0.875 ", and the crosshead speed was 0.20"/minute. The dog-bone shaped tensile specimens were cut using a hollow die punch. Twenty specimens were tested for each sample. The compression molded specimens of the uncompatibilized blends were brittle and thus unsuitable for specimen cutting using hollow die punch, and therefore the tensile properties of the uncompatibilized blends were not measured. The shredded carpet fibers, the extruded carpet resins, and the compression molded sheets were rigorously dried at 80°C for at least 18 hours, before every processing and testing step.

TABLE 4. Compression Molding Parameters.

Temperature ("C)		Pressure
Upper plate	Lower plate	(psi)
227	227	2500
277	277	2500

The melt flow rate of the uncompatibilized and the compatibilized blends were measured on a Melt Flow TQ (Ceast USA) instrument at 275°C, using a load of 2.16 kg. The mean of twelve readings are reported here. A Perkin Elmer DSC4 was used to study the melting transitions of the as-received shredded fibers and the compatibilized alloys at a heating rate of 200°C/min. The shredded carpet waste fluff (second batch - from which SBR and calcium carbonate was removed) and the compatibilized extruded resins were treated with 88% formic acid (Fisher Scientific) and filtered using a wire-cloth filter. This process dissolves nylon and leaves the other components in the carpet waste undissolved, and were removed in the filtering process. Nylon 6 was subsequently regenerated from solution using water as the non-solvent. Nylon 6 from carpet fluff formed clear solutions in formic acid, while solutions from extruded and compatibilized blends were cloudy.

FTIR spectroscopy of the nylon 6 samples recovered from carpet fluff and from compatibilized and extruded blends by dissolution were performed on a Perkin Elmer FTIR spectrometer. SEM analysis of extruded pellets of the uncompatibilized and compatibilized alloys were carried out on a Hitachi S-800 microscope. For SEM study the polypropylene component was dissolved out using hot xylene (Fisher). The specimens were coated with gold.

Results and Discussion: Tensile and melt flow test results are presented in Table 5. The tensile specimens exhibited fairly high strengths and brittle failure. The tensile strength values of the samples molded at 227°C are higher than the values of samples molded at 277°C. It must be stressed that the data presented in Table 4 are the results of the unoptimized process. Optimization will include: (i)selection of the optimum extrusion and compression molding parameters, and (ii) the selection of the most effective compatibilizer and its optimum weight percentage. Tensile data of the samples molded at 227°C indicates that the industrial carpet waste/compatibilizer blend can be successfully extruded to yield a material with useful mechanical properties. These data indicate that when the process and compatibiiization optimization are completed, then resins with excellent mechanical properties can be obtained from the industrial carpet waste. Our results compare well with those presented by Adur and Constable [11] for blends of the virgin PP and nylon 6 resins containing 15% maleated PP as compatibilizer.

TABLE 5. **Tensile Properties and Melt Flow Rates (MFR) of Extruded Blends**

Compatibilizer *(Weight %)*	Molding Temperature *(°C)*	Tensile Strength *(KPSI)*	Strain at Break *(%)*	MFR *(g/l Omin)*
3	*227*	10.8 ± 1.3	10.3 ± 1.2	29.5 ± 1.3
3	*277*	*7.4 ± 0.4*	*9.2 ± 0.9*	—
0	*2271277*	—	—	*64.9 ± 2.8*

The sharp decrease in MFR of the compatibilized blend (29.5 g/10min) compared to the uncompatibilized one (64.9 g/10min) is in congruence with observations of other researchers [1 1] working with virgin polymers. This radical drop in MFR has been attributed to an increase in molecular weight due to grafting reactions between the maleic anhydride of the compatibilizer and the terminal amino groups of the nylon. The solubility studies also support the above observation. It was observed that while the formic acid extract was water-clear in the case of the carpet fluff, it was very turbid in the case of the compatibilized alloy. The development of the turbidity in the latter instance could be rationalized in terms of decreased solubility of the species due to the presence of polypropylene

The FTIR spectroscopic analysis was carried out on the regenerated nylons from the two formic acid extracts (one from the fiber fluff and the second from the compatibilized blend) to identify possible structural changes arising from the proposed reaction. In the compatibilized blend the presence of absorbance band at 1743 cm^{-1} clearly supports the chemical reaction between the compatibilizer and nylon.

The DSC scans of the carpet fluff showed endothermic transitions corresponding to PP as well as nylon 6. The onset of melting for nylon 6 was 212°C. PP had two unresolved melting endotherms with onset at 152.5°C and 159.5°C. Maleated polypropylene, the compatibilizer, exhibited the onset of melting at 152°C. The compatibilized blend had two melting transitions with onset at 149.2°C and 208.5°C.

In summary, preliminary studies show that chemical compatibilization of co-mingled carpet waste through reactive extrusion could be used to produce reasonably high strength alloys. Presently, work is being carried out to evaluate the effectiveness of the different commercially available compatibilizers and to optimize their dosage and processing conditions.

Fiber Reinforced Concrete Using Carpet Waste Fibers:

Background: Concrete is durable, inexpensive, readily molded into complicated shapes, and has adequate compressive strength and stiffness, and it is the most heavily used man-made material in the world. However, concrete has low tensile strength, low ductility, and low energy absorption. Fiber reinforced concrete (FRC) is often made by adding a small fraction (usually below 2% by volume) of short fibers to the concrete mix during mixing. After extensive studies in the last three decades, it is now beyond doubt that such fiber reinforcement can significantly improve the tensile properties of concrete. Orders of magnitude increases in toughness (energy absorption) over plain concrete is commonly observed. Fiber reinforced concrete is currently being used in many applications including buildings, highway overlays, bridges, and airport runways [23-25]. Fibers used in FRC include steel, alkali-resistant glass, and various synthetic fibers, most notably polypropylene. Recycled synthetic fibers such as those from textile industry waste offer low cost alternatives for concrete reinforcement. This work has focused on concrete reinforced with recycled fibers from hard carpet waste. It suggests that using recycled carpet fiber in construction could be a very cost-effective way to improve the durability and performance of concrete structures and at the same time to reduce the needs for landfill spaces.

Sample Preparation and Test Procedure; The concrete matrix consisted of Type I Portland cement, river sand, crushed granite aggregate (maximum size=10 mm), and water. The weight ratios were 1.0 (cement)/0.35 (water)/0.85 (sand)/0.61 (aggregate). Recycled carpet waste fiber used was from hard carpet waste, disassembled mechanically by the Crown America, Inc. of Dalton, Ga. After disassembling, surface yarns (nylon) and some backing fibers (polypropylene) were collected. This collection is referred to as Type I waste fiber and had a typical length range between 12 to 25 mm. The disassembled waste after removal of Type I fiber is referred to as Type II, which contained backing fibers, SBR coated face yarns, and SBR particles. An analysis of Type II fiber indicated that the fiber length was about 3 to 25 mm and that about one third by weight was actually fiber, the other two thirds being CaC03 filled SBR. Fiber volume fractions for the waste fibers were kept at 2%. Only the actual fiber portion was included for calculating fiber volume fractions for Type II waste fiber reinforced concrete. FiberMesh® virgin polypropylene fiber at 0.5% volume fraction was also included in this study for comparison purposes. The fiber length was 19 mm.

A drum mixer was used for mixing. The freshly mixed concrete was filled into plastic molds, and then stored in a saturated curing room (100% RH). Four point flexural test and cylinder compressive test were illustrated schematically in Figure 2. All the tests were performed on a hydraulic testing machine. The age of specimens at testing was 28 days except the 1 day compressive test.

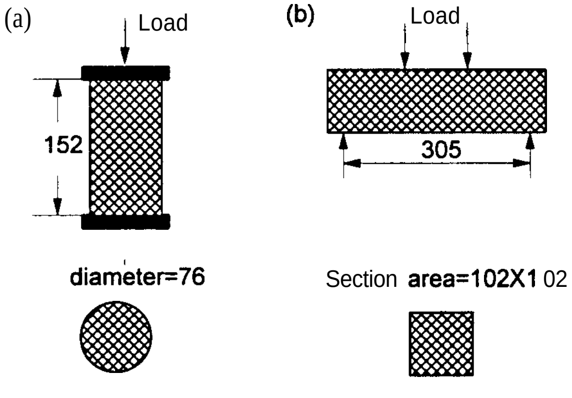

Figure 2. Test Setup for (a) Compression Test, and (b) Flexural Test (Unit: mm)

Results and Discussion; The results for compression and flexural tests of various mixes are given in Table 1. Six or seven specimens were tested for each setup. In the one day compressive test, similar strength values were observed for plain concrete and various FRCs. It appeared that the 28 day compressive strengths of carpet waste FRCs were lower than that of plain concrete. The plain concrete specimens failed in a brittle manner and shattered into pieces. In contrast, all the FRC samples after reaching the peak load could still remain as an integral piece, with fibers holding the concrete matrices tightly together.

TABLE **1. Compressive and Flexural Test Results**

Fiber in FRC Mix	V_f	Compressive strength		Flexure Test at 28 days		
		1 day	28 days	Strength	Toughness	
	%	*MPa (CV%)*	*MPa (CV%)*	*MPa (CV%)*	I_5	I_{20}
Concrete control	0	20.9 (4.9)	52.6 (3.1)	4.6 (4.0)	1.0	1.0
Fiber Mesh PP	0.5	24.2 (3.7)	52.2 (1.4)	4.6 (3.1)	2.6	6.9
Type I waste fiber	2.0	20.9 (5.7)	39.7 (7.4)	4.7 (2.3)	3.3	7.5
Type II waste fiber	2.0	18.6 (7.6)	40.7 (7.4)	4.4 (4.3)	3.5	9.8

The flexural strengths of all mixes tested were essentially the same and the standard deviations were low. Figure 3 shows the typical load versus displacement curves for the flexural test. The plain concrete samples broke into two pieces once the peak load was reached, with very little energy absorption. The FRC specimens, on the other hand, exhibited a pseudo ductile behavior and fibers bridging the beam crack can be seen. Because of the fiber bridging mechanism, the energy absorption during flexural failure was significantly higher than that for plain concrete. One method of characterizing the energy absorbing ability, or toughness, of FRC is to calculate the toughness indices, as proposed in ASTM C1018. These indices indicate the energy absorption of FRC compared with a brittle material such as concrete (toughness index=1.0) for the deformation range specified.

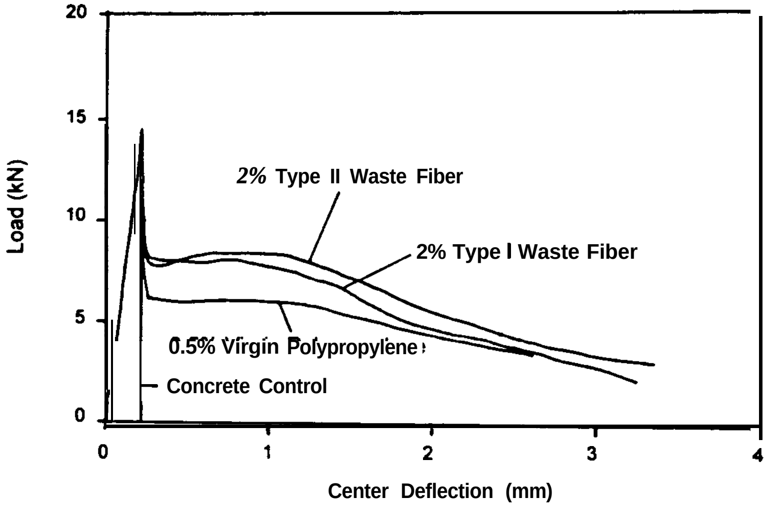

Figure 3. Typical Flexural Test Curves

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THESIS AND PUBLICATIONS

Thesis:

Raj Datta, "Utilization of Polymer Compatibilization for Polymer Blend Recycling", Ph.D. thesis, Georgia Tech, in progress.

D. Scott, "Characterization of Fibrilated Polypropylene and Recycled Waste Fiber Reinforced Concrete", M.S. Thesis, Georgia Tech, August, 1992.

Publications:

R. K. Datta, M. B. Polk, and S. Kumar, "Reactive Extrusion of Industrial Carpet Waste containing Polypropylene and Nylon", in preparation for publication.

Y. Wang, A. Zureick, B.S. Cho, D. Scott, "Properties of Fiber Reinforced Concrete Using Recycled Fibers from Carpet Industrial Waste", in preparation for publication.

Patent:

S. Hansen, L. LeBoeuf, N. Venkatasubramanian, and M. Polk, "Depolymerization of Polyesters and Polyamides", U.S. Patent Application: 5364-K-CIP.

RESOURCE MANAGEMENT

One important resource which is unavailable at present for the depolymerization studies is a capillary gas chromatograph for determining the structures of the soluble materials obtained during the depolymerization studies. A coupled capillary gas chromatograph-mass spectrometer would be even more useful in these studies. At present, we are attempting to borrow a capillary gas chromatograph from another university in the area. Our other needs have been satisfied by the acquisition of a fairly comprehensive extrusion unit and a compression molding unit. As an example of the sharing of resources implicit in the NTC Program, we have agreed to allow Dr. Dick Gregory of Clemson University to use our extrusion equipment for his NTC project. We have successfully utilized resources of such industrial companies as Haake and Crown America in completing our work, and collaboration has been established with Reichhold Chemicals and Shaw Industries.