Effect of Crosslinking on Flatspotting of Nylon 6 Tire Yarn

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Synopsis

Tires containing nylon tire cord exhibit a temporary deformation upon standing under load. The delayed recovery from this deformation, due to the fact that the yield point of nylon is exceeded under normal inflation pressures, results in the phenomenon of "flatspotting." Previous attempts to improve the viscoelastic characteristics of nylon by crosslinking have been discouraging on account of the general degradation of properties caused by the treatment. It has now been found that short, vapor-phase treatments with diisocyanates, particularly tolylene diisocyanate, produce nylon 6 of greatly improved flatspotting resistance, without undesirable side effects. The development of this procedure, the evaluation methods employed, and the proposed mechanism are discussed.

INTRODUCTION

Nylon is widely used as the cord material in replacement passenger automobile tires, but it has not found general acceptance in the original equipment market because of its undesirable "flatspotting" behavior.

Flatspotting is the temporary deformation of an inflated tire caused by standing under a load. A flatspot develops in the footprint of the tire, with the result that thumping takes place when the tire begins to rotate. Under the stress of motion, the flat spot is soon "run out," but such behavior is clearly objectionable, especially so in tires on new automobiles being demonstrated after standing in a dealer's show room. The elimination of the flatspotting characteristic of nylon could greatly expand its use in original passenger automobile tires.

The cord in the carcass of an inflated tire is under tension, except in the footprint area, where it is under compression (or, at least, lower tension).¹ The difference in stress between the two segments results in differential deformation. The degree of deformation varies with different fibers in the order nylon > polyester > rayon > glass. The most important fiber property relating to these differences is the temperature dependence of cord modulus.

On this basis, crosslinking of the amorphous regions of a nylon tire fiber, an approach which has been extensively investigated,² is one way of raising the high temperature modulus of nylon and substantially reducing its tendency to flatspot.

The first and most widely investigated nylon crosslinking agent has been formaldehyde. In the simplest method, nylon is treated with anhydrous formaldehyde vapor at 120–180°C.³⁻⁵ The polymer can also be treated in formic acid solution at 60–100°C., or it may be soaked in aqueous formaldehyde in the presence of weak acid^{3,6,7,36,37,39} or alkaline catalyts.^{3,8} The reaction of formaldehyde with nylon in the presence of alcohols has been extensively reported.^{3,6,8–13,38} The results suggest the formation of *N*-alkoxymethyl groups followed by crosslinking upon heating.

Nylon has also been crosslinked by γ -rays, x-rays, and ultraviolet irradiation,¹⁴⁻¹⁸ by treatment with ethylene oxide,¹⁹⁻²² epoxides,^{23,24} acyl polylactams,²⁵⁻²⁹ with diacid chloride and diisocyanates,³⁰ and with diisocyanates alone.³¹⁻³³

Though an increase in the initial modulus can be attained by most of these treatments, it is usually accompanied by drastic modification of the polymer structure. This often brings about other changes in yarn properties, rendering the material unsuitable for tire cord use.

Our investigation of the crosslinking approach began with a search for bifunctional agents which would increase the modulus of the yarn without substantial impairment of other functional properties. Preliminary screening investigations led to the selection of diisocyanates for detailed study; the exploration also indicated that short-term vapor-phase treatments were the most promising. This conclusion was confirmed by the studies of the vapor-phase crosslinkings of nylon 6 with diisocyanates described in this paper.

EXPERIMENTAL

Materials

Yarn. Commercial 840/136 denier Caprolan* nylon tire yarn was used in this investigation.

Crosslinking Agents. Crosslinking agents used were 2,4-tolylene diisocyanate (TDI) (Nacconate 100, Allied Chemical Corp.), 80% 2,4- and 20% 2,6-tolylene diisocyanate (Nacconate 80, Allied Chemical Corp.), 65% 2,4- and 35% 2,6-tolylene diisocyanate (Nacconate 65, Allied Chemical Corp.), *m*-phenylene diisocyanate (DTT Chemical Company), diphenylmethane 4,4'-diisocyanate (Nacconate 300, Allied Chemical Corp.), hexamethylene diisocyanate (K and L Laboratories, Inc.), *trans*-vinylene diisocyanate (Aerojet-General Corp.), and phenyl isocyanate (Eastman Organic Chemicals).

* Caprolan is a registered trademark of Allied Chemical Corporation for its continuous-filament synthetic yarns.

Apparatus and Methods of Treatment

Initial screening experiments were performed by means of a batch treatment. The most promising approach, using TDI, was then extensively studied in a laboratory-scale continuous process.

Batch Treatment. A diagram of the apparatus employed in the batch vapor-phase treatment is shown in Figure 1. The apparatus consists of a reaction vessel and a carburetor. The yarn, wound on a stainless steel frame, 5 in. long and ${}^{3}/_{4}$ in. wide, is suspended in the reaction vessel, which in turn is immersed in an oil heating bath. Nitrogen gas, used for sweeping the apparatus, is introduced through the heated carburetor.



Fig. 1. Schematic diagram of the batch-treating apparatus.

The yarn is wound on a frame under a tension of 0.5 g./den. and inserted into a reaction vessel. The apparatus is flushed with nitrogen gas and the diisocyanate added through the carburetor.

Continuous Treatment. The single-end continuous unit is shown schematically in Figure 2. The apparatus consists of three stainless steel reaction vessels and a carburetor vessel which supplies the diisocyanate vapors to the second reaction vessel. Each reaction vessel contains an assembly comprising two idler yarn-accumulating rollers and two tubes for the entrance and exit of the nitrogen gas. The three vessels are immersed in a constant temperature oil bath, controlled to $\pm 1^{\circ}$ C. A feed roll is used to pull the yarn from the supply package through a tension compensator (festoon). A variable speed pull roll serves to pull the cord through the treating unit. The yarn is finally wound on a constant tension take-up device.

In the first vessel, the yarn is predried by heating in nitrogen sweep gas $(-50^{\circ}C. \text{ dew point})$. It then passes over external guide rollers into the



second reaction vessel into which the crosslinking agent has been introduced. In the third vessel, unreacted isocyanate is swept off by the nitrogen stream. The reaction time (time in the second and third vessels) can be changed by varying the speed of the take-up unit or by varying the number of wraps around the rollers.

Evaluation Tests

Insolubility in Formic Acid. One of the characteristics of crosslinked nylon is its insolubility in the usual nylon solvents. To determine the extent to which a yarn is crosslinked, a microscopic technique was developed based on the insolubility and swelling of the crosslinked fiber in 90% formic acid.

A 90% formic acid solution was prepared containing 0.15% Lanamid Red 3 BS (Colour Index Acid Red 182) dye (Allied Chemical Corp.) to distinguish the undissolved nylon yarn by coloration. Samples to be tested were cross-sectioned into small lengths with scissors, wetted in the formic acid-dye solution, and observed after several minutes through a microscope under low magnification.

Insolubilization invariably proceeded from the periphery to the core of the fiber; the proportion of the fiber crosslinked was estimated by a visual determination of the portion of the fiber which was undissolved. This is shown schematically in Figure 3. Sections A show a fiber having about a quarter of its area crosslinked. Sections B were crosslinked through half of its total area. In sections C, about three-quarters of the fiber was crosslinked, only a small center portion being soluble. Sections D represent an interesting phenomenon which was observed occasionally: it shows a fiber crosslinked throughout its area, but the extent of crosslinking is less in the core than in the periphery, as judged both by the lower color



Fig. 3. Appearance of nylon 6 sections at various degrees of insolubilization in 90% formic acid.

intensity and the greater swelling (seen in the longitudinal view) of the central portion. Sections E show swelling uniformly across the fiber diameter. No protrusions are seen in longitudinal segments.

It was considered of interest to determine whether the swelling evident in sections of type D was accompanied by any significant dissolution of the polymer. To establish this, 0.5 g. samples of fibers of type D and E were shaken in 30 ml. of 90% formic acid at room temperature, the residue being recovered on tared, sintered filtering funnel, dried, and weighed. The weight loss was practically the same in the two cases: 7.9% in type Dand 7.3% in type E.

Tensile Properties. The tire yarn was conditioned in a relaxed state, at 21° C. and 65% R.H., for at least 16 hr. before being tested on an Instron tensile tester.

In the single-cycle stress-strain test, 10-in. specimens were extended at a strain rate of 60%/min.; the initial modulus, the breaking stress and the elongation at break were measured.

Cycling tests were carried out to 3% extension. The specimens were extended at a strain rate of 60%/min., kept extended for 30 sec., retracted, and allowed to rest for 1 min. before commencement of the next cycle. The work of recovery was recorded as a percentage of the work of extension on the same cycle.

All the reported values are averages of three tests which were always in excellent agreement.

Flatspotting Index. A laboratory method which simulates end-use conditions was used. The test used in this study was developed a number of years ago in our laboratory and does not differ from any other used in the industry in any fundamental manner.³⁴

Reaction Mechanism

The following three reactions may be visualized as occurring between an isocyanate and nylon 6.

Formation of an acyl urea derivative by reaction with the amide group:



Formation of a disubstituted urea by reaction with the terminal amino group:



Possible, but unlikely, is the formation of an amide by reaction with the terminal carboxyl groups, carbon dioxide being eliminated:



In the case of a diisocyanate, the second group may then react, in the same manner as the first or differently, with another polyamide chain to form a crosslinkage.

A paper on the details of the crosslinking mechanism has been presented elsewhere.³⁴

RESULTS AND DISCUSSION

Batch System

A wide variety of reagents and treatment conditions were explored by this means. The optimum conditions for treatment with isocyanates were found to be a reaction temperature of 180° C., with treatment times of 5–30 min. Typical results obtained with a range of isocyanates are listed in Table I.

	Rose-	Tensile properties				
Reagent	tion time, min.	Initial modulus, g./den.	Breaking stress, g./den.	Breaking elonga- tion, %	Portion insoluble in HCOOH	
Untreated		33	8.4	18.2	None	
Control (No reagent)	20	35	7.8	14.7	None	
Tolylene diisocyanate						
100% 2,4	7	44	8.0	15.2	Quarter	
	10	48	7.0	12.6	Half	
	25	50	6.2	12.8	All	
80% 2,4-20% 2,6	7	39	8.1	15.0	Quarter	
65% 2,4-35% 2,6	7	36	8.1	15.0	Quarter	
	15	39	5.8	11.2	Third	
	30	44	3.0	8.6	All, core swelling	
<i>m</i> -Phenylene diisocyanate	7	39	7.7	13.9	Half	
	20	48	6.2	13.5	All	
Diphenylmethane 4,4'- diisocyanate	7	36	8.1	15.8	None	
Hexamethylene diisocyanate	7	34	8.0	15.6	None	
	30	36	8.2	16.0	None	
trans-Vinylene diisocyanate	10	34	7.9	15.9	Half	
Phenyl isocyanate	5	38	6.7	12.6	None	
	15	47	2.5	8.0	None	

 TABLE I

 Properties of Nylon 6 Yarn Treated at 180°C. with Various Isocyanates (Batch System)

All the diisocyanates caused some crosslinking under the conditions employed, as judged by modulus increase and loss in formic acid solubility, except for hexamethylene diisocyanate and diphenylmethane 4,4'-diisocyanate (Nacconate 300), the latter reagent probably being sterically too large and involatile to react under these conditions. In all cases, the improvement in modulus is accompanied by some deterioration in ultimate tensile properties. In the case of tolylene 2,4-diisocyanate and of *m*-phenylene diisocyanate, however, a significant benefit is obtained with only minor property changes. Tolylene 2,4-diisocyanate (Nacconate 100) was chosen for detailed study in the continuous system.

It is interesting that a significant modulus increase was obtained after the monofunctional treatment with phenyl isocyanate. As would be expected, no crosslinking occurred, the solubility in formic acid being unaffected. Properties other than modulus underwent only moderate changes.

Continuous System

In this system (Fig. 2) it is possible to attain complete crosslinking in a very short time at elevated temperatures. Figure 4 shows the results obtained with various treatment times at the two temperatures, 205 and 217 °C.; the yarn-residence time was varied by changing the number of yarn wraps around the rollers. The highest temperature which could be employed without encountering filament coalescence was 217 °C. At this temperature, complete crosslinking without evidence of a skin-core structure was realized with TDI in 40 sec., as illustrated in Figure 3*E*.

The time required for complete fiber crosslinking over the range of 175-217 °C. is shown in Figure 5. The relationship between the time and the



Fig. 4. Insolubilization as a function of time at two temperatures.



Fig. 5. Time for complete insolubilization as a function of temperature.

reciprocal of the absolute temperature is linear over the range studied, so that the data can provide an estimate of the activation energy of the process, provided the assumption is accepted that each point on the line represents the same extent, and type, of crosslinking.* This assumption seems reasonable in light of the fact that the yarns representing the points in Figure 5 had similar initial modulus values (Table II).

Reaction conditions		Initial	
Temperature, °C.	Time, min.	modulus, g./den.	
		45	
175	9	54	
185	6	58	
195	4.5	58	
205	3	54	
217	0.66	59	

TABLE II	
Insolubility and Initial	Modulus

The energy of activation E derived from the relationship $\ln 1/t_{ins} = E/RT + \text{const.}$ (where $t_{ins} = \text{time}$ for insolubilization) is about 30 kcal./ mole, a rather high value for a process which appears to be diffusion-controlled (cf. Fig. 3).[†]

* It is also assumed, of course, that this extent of crosslinking does not represent complete reaction.

 \dagger Underwood³⁵ has shown this type of relationship is valid independently of the order of the reaction, provided the order is constant.

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Work Recovery at 3% Strain			
Fraction crosslinked, %	Work recovery, %		
	First cycle	Tenth cycle	
	55.1	65.9	
25	• 64.6	66.9	
50	65.7	66.7	
100	68.9	69.2	

TABLE III Work Recovery at 3% Stra

Room temperature, single-cycle stress-strain curves of filaments treated at 217 °C. for various times so as to crosslink the fiber section 25, 50, and 100% are shown in Figure 6. Crosslinking of a quarter of the cross-sectional area produces a substantial increase in modulus, accompanied by only a slight loss in ultimate tenacity. Treatment to give 100% cross-linking has little further effect.

Similar behavior is seen in the work of recovery from 3% elongation (Table III). The crosslinked materials have superior recovery in the first



Fig. 6. Effect of crosslinking on stress-strain properties of nylon 6.



Fig. 7. Effect of temperature on crosslinked yarn strength.



Fig. 8. Effect of temperature on crosslinked yarn tensile modulus.

cycle; the difference, however, is largely eliminated by the mechanical conditioning of further cycling.

The effect of exposure to elevated temperature on the strength retention and tensile modulus of the crosslinked nylon is shown in Figures 7–11. The tests of Figures 7 and 8 were performed at the temperatures indicated; those of Figures 9–11 were performed on yarn conditioned at 21°C. and 65% R.H. after the exposure to elevated temperature.



Fig. 9. Effect of treatment in dry heat on strength of crosslinked yarn (treated at 0.02 g./den.).



Fig. 10. Effect of treatment in dry heat on tensile modulus of crosslinked yarn (treated at 0.02 g./den.).

It is apparent from Figure 7 that the loss in strength due to exposure to elevated temperature is lowered by crosslinking. The crosslinked yarn also maintains its initial tensile modulus advantage throughout the temperature range studied, as shown in Figure 8. In order to estimate the effect that commercial tire curing cycles may have on the tensile properties of the crosslinked yarn, laboratory tests involving various thermal treatments under very light load (0.02 g./den.) were performed. Figures 9 and 10 show the effect on the strength retention and the modulus of treating the yarn for 5 min. at the temperature indicated. Figure 11 shows the



Fig. 11. Effect of saturated steam on crosslinked yarn strength (treated free to relax).

effect of exposing the yarn to saturated steam for 15 min. at various temperatures on the strength retention.

These data suggest that crosslinked Caprolan nylon tire cord would suffer little or no loss in strength when the tire is cured at normal industry temperatures.

This is probably due to the fact that crosslinked nylon is more dimensionally stable than uncrosslinked nylon. The reasons for this are indicated by x-ray studies on yarn heated to 195° C. The crosslinking itself has no effect on the extent of crystallinity, but, after exposure to 195° C., the crosslinked yarn retains a greater degree of crystallinity than the uncrosslinked material. Nylon 6 filaments normally begin to fuse at 215–220°C., but the presence of crosslinks in the amorphous regions stabilizes the yarn dimensionally and also prevents the filaments from losing their cylindrical shape and fusing up to 300°C.

		Fat	igue
Material	Static adhesion in rubber, cord breaking strength, lb.ª	Goodyear tube, min. ^b	Goodrich Disc % breaking strength retained
Untreated 100% Crosslinked	12.1 12.8	577 687	84 84

TABLE IV n-Rubber Properties of Crosslinked Nylon 6 Cord

* ASTM Method D-2138-62T.

^b ASTM Method D-885.

The data in Table IV show that neither the rubber adhesion nor the fatigue properties of Caprolan nylon cord are impaired by crosslinking and may, indeed, be improved.

Effect of Crosslinking on the Flat Spot Index

The effect of crosslinking on the flat spot index (FS1) of the Caprolan nylon yarn³⁴ is summarized in Figure 12. A low degree of crosslinking is



Fig. 12. Effect of crosslinking on yarn flat spot index.

sufficient to produce a large improvement in the FSI. This behavior is consistent with the fact that the greatest effect on the stress-strain properties of the yarn takes place at 0-25% crosslinking.

Relationship Between Laboratory Tests and Road Tests on the Severity of Flatspotting

The flatspotting characteristics of nylon 6 tire yarn crosslinked with tolylene diisocyanate, examined by the laboratory test previously described, were evaluated in actual road tests.

Experimental single-end tires were constructed from crosslinked and control sample yarn. These tires were cured under commercial conditions and road-tested using standard tire flatspot evaluation procedures.

Comparative results of the road tests and the laboratory FSI values are given in Table V. It is evident (Fig. 13) that the two test methods yield comparable results. On the arbitrary severity scale used in Figure 13, a level of 40–45 is below the objectionable limit of flatspotting.

`	Flatspot index (laboratory), mm.	Dynamic wheel		
Material		Flatspot severity ^a	Run-out timeª	
Nylon	33	100	100	
TDI-Crosslinked nylon	22	41	40	
Polyester	13	14	13	
Rayon	10	3	5	

TABLE V Comparison of Laboratory and Dynamic Wheel Tests

^a Relative values.



Fig. 13. Relationship between laboratory flat spot index and tire road test.

CONCLUSION

On the basis of all tests, we conclude that vapor-phase crosslinking with TDI can reduce the flatspotting of nylon to an unobjectionable level without adversely affecting the other desirable characteristics of Caprolan tire yarn.

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Résumé

Les pneus contenant des fils nylons montrent une déformation temporaire au repos sous l'ffect d'une charge. Le recouvrement retardé au départ de cette déformation, due au fait que le point de rendement du nylon est dépassé sous pression de gonfiement normal résulte d'un phénomène dénommé "flatspotting". Les essais antérieurs en vue d'améliorer les caractéristiques viscoélastiques du nylon par pontage sont décourageants par suite de la dégradation générale des propriétés causées par suite de ce traitement. On a maintenant trouvé qu'un traitement de courte durée en phase vapeur avec des diisocyanates, particulièrement le diisocyanate de toluène, produisent des nylons-6 de résistance fortement améliorée en celui concerne le flatspotting sans effet secondaire indésirable. Le développement de ce procédé, les méthodes d'évaluation employées et un mécanisme proposé sont soumis à discussion.

Zusammenfassung

Reifen mit Nylonreifencord zeigen beim Stehen unter Belastung eine zeitweise Deformation. Die verzögerte Erholung von dieser Deformation, welche daraus folgt, dass die Elstizitätsgrenze von Nylon unter normalen Aufpumpdrucken überschritten wird, führt zum Phänomen des "Flatspotting". Frühere Versuche, die viskoelastische Charakteristik von Nylon durch Vernetzung zu verbessern, waren wegen der dadurch bedingten allgemeinen Verschlechterung der Eigenschaften nicht sehr erfolgreich. Es wurde jetzt gefunden, dass eine kurze Behandlung mit Diisocyanaten, besonders Toluylensiisocyanat in der Dampfphase, Nylon 6 von stark verbesserter "Flatspotting"- beständigkeit, ohne unerwünschte Nebeneffekte erzeugt. Die Entwicklung dieses Verfahresn, die verwendeten Auswertmethoden, sowie der vorgeschlagene Mechanismus werden diskutiert.

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