

Structure and Property Characterization of Spunbonded Filaments and Webs Using Thermal Analysis

DONG ZHANG, QIN SUN, GAJANAN BHAT, LARRY WADSWORTH

Textiles and Nonwovens Development Center, The University of Tennessee, Knoxville, Tennessee 37996

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ABSTRACT: The thermal history and inherent properties of the filaments are of utmost importance in spunbonded webs. A good understanding of the structure and properties development of individual filaments is helpful in comprehending the whole process better. Polypropylene homopolymer and a copolymer were processed using the Reicofil® spunbonding line at the Textiles and Nonwovens Development Center of the University of Tennessee, Knoxville. The properties of the filament samples taken before thermal bonding were determined through a variety of thermal analyses, such as differential scanning calorimetry, thermomechanical analysis, thermal deformation analysis, and dynamic thermal analysis, as well as mechanical properties. The performance properties of the bonded nonwoven fabrics from these filaments were evaluated, and the structure and properties of the fibers were compared with those of the bonded fabrics. © 1998 John Wiley & Sons, Inc. *J Appl Polym Sci* 69: 421–434, 1998

Key words: spunbond; polypropylene; filaments; nonwovens

INTRODUCTION

Thermal analysis is widely used to study the structure and properties development of polymeric materials during processing. Spunbonding is a unique process that links the innovative polymer, technological, and textile research, which involves web formation and bonding of web to impart strength, cohesiveness, and integrity to the structure. Spunbonding is based on the spinning technique and bears many similarities.¹ The spunbonding process involves extrusion, followed by nonisothermal extensional flow of the melt and crystallization, accompanied by molecular orientation. The kinematics and development of morphology in the filaments are governed to a large extent by a combination of elongational deformation and stress-induced crystallization. Understanding the influence of the spinline dynamics

on the structure of the filament can help in manipulating the process conditions to achieve the desired properties in the filaments. In the Reicofil system,² the molten polymer is forced by spinning pumps through special spinnerets having a large number of holes. The primary blow ducts, located immediately below the spinneret block, continuously cool the filaments with conditioned air. Secondary blow ducts, located below the primary blow ducts, continuously supply auxiliary air at room temperature. A blower at the bottom of the enclosed spinline creates a suction for the filaments and mixed air. The continuous filaments are sucked through a venturi (high-velocity low-pressure zone) and a distributing chamber, which ensures entangling of the filaments. The entangled filaments are deposited as a random web on a moving porous belt for thermal calendaring.

The effect of stress-induced crystallization has been studied with PET-based polymers in melt spinning.³ The role of stress on oriented crystallization was demonstrated by deformation of uniaxially loaded fibers exposed to temperatures in the

Correspondence to: D. Zhang.

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Table I Parameters of Processing Variables

Sample Specification	1, 2	3, 4, 5	6, 7
Base weight (g/m ²)	40.0	40.0	40.0
Spin pump speed (rpm)	8.6	12.9	17.2
Gross throughput/h (kg)	44.6	66.9	89.2
Gross throughput/hole (g hole ⁻¹ min ⁻¹)	0.2	0.3	0.4
Primary air speed (rpm)	762.1	963.7	1267.2
Suction blower speed (rpm)	1652.9	2012.0	2252.4
Spin belt speed (mpm)	15.2	22.9	30.5
Upper roll temperature (°C) for PP	147.2	147.2	147.2
Upper roll temperature (°C) for PP/PE	126.7	126.7	126.7
Lower roll temperature (°C) for PP	144.4	144.4	144.4
Lower roll temperature (°C) for PP/PE	123.9	123.9	123.9

processing range. The response of the filaments in subsequent thermomechanical processing (such as calendaring) is greatly influenced by the structure of the filaments. This response can range from severe embrittlement of initially very ductile material to enhancement of ductility. New regimes of post-spinning deformation can be inferred from these studies to produce fabrics with superior properties.

Although the effect of processing variables on the properties of the webs has been studied, there is no information available on the structure and properties development of the filaments.⁴ This research was done to elucidate the filament morphology and property development using thermal analysis. The changes taking place during calendaring were compared with that of the corresponding bonded webs.

EXPERIMENT

Polypropylene (PP) homopolymer and polyethylene (PE)-based polypropylene copolymer were processed using the Reicofil[®] spunbonding machine, based on the established optimum process window for 35 melt flow rate (MFR) polypropylene by earlier researchers,⁵ and a series of filament samples (before bonding) and the corresponding nonwoven samples were collected. Processing conditions used are reported in Table I. Different primary air temperatures were used to study the influence of thermal history on the structure and properties development.

Thermal Characterization

Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) of the filaments and the nonwoven samples was carried

out using the Mettler DSC 25. The samples were scanned at a heating rate of 10°C/min in the nitrogen environment. Enthalpies of melting were determined so that the crystallinity of the samples could be calculated. A ΔH value of 146.5 J/g for 100% crystalline polypropylene was used for estimating the crystallinity of the samples.⁶

Thermomechanical Analysis

Thermomechanical analysis responses were recorded using the Mettler TMA 40 with a heating rate of 10°C/min at tension, compression, and dynamic mode. The samples were scanned in the nitrogen environment with different levels of pretension using tension, compression, and dynamic mode. The gauge length for the samples was 10 mm, and the width of nonwoven samples was approximately 1 mm. The samples were held by copper clips at both ends, and a constant load was applied to the samples before starting the scan. The changes in length taking place with the increase in temperature were recorded. Isothermal scanning of the samples was done to determine the length changes with time at different temperatures and pretensions.

Thermal Deformation Analysis

For thermal deformation analysis (TDA), a tubular oven mounted on rails (Fig. 1) was used. One end of the leader filament was fixed, while the other end was passed over a smooth roll and pretensioned by applying required load. The tension or stress was adjusted by changing the mass hung at the free end. Changes in dimension of the samples exposed to different temperatures were measured.

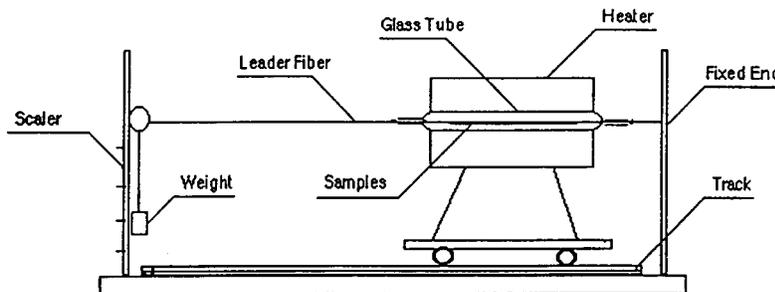


Figure 1 Experimental setup for TDA.

Physical Properties

Tensile properties of the filaments and the nonwoven samples were measured using the United Tensile Tester with test conditions described in the ASTM D3822-91 for filaments and ASTM D1117-80 for nonwoven fabrics.⁷ For filament samples, a gauge length of 5 cm and an extension rate of 5 cm/min were used. The filaments were carefully separated from unbonded webs, avoiding any deformation of the sample during that process. The sample was then mounted in a window with a slight pretension for further testing. For nonwoven samples, a gauge length of 10 cm, a width of 2.54 cm, and an extension rate of 10 cm/min were used in both the machine direction and the cross direction. The tenacity, breaking elongation, and initial modulus were recorded and processed statistically by the connected computer.

RESULTS AND DISCUSSION

Thermal Properties

PP and PP/PE filaments were produced at different throughput and primary air temperatures and collected before thermal bonding. The sample identification, fiber diameter, and birefringence of the filaments are shown in Table II. The lower primary air temperature resulted in higher air draw-down force, which resulted in smaller fiber diameter and higher birefringence. The crystallinity of filaments was determined by DSC. Figure 2 shows the DSC scans of filament samples produced at the same throughput and different primary air temperatures. The melting peaks show some differences, especially in their heat of melting, although melting temperatures show no difference. The melting enthalpy in-

Table II Sample Specification and Physical Properties

Sample	Throughput (g hole ⁻¹ min ⁻¹)	Primary Air Temperature (°C)	Diameter (μm)	Birefringence
H ₁	0.2	10	21.8	0.0244
H ₂	0.2	18	23.6	0.0213
H ₃	0.3	10	24.8	0.0224
H ₄	0.3	18	26.2	0.0209
H ₅	0.3	27	27.1	0.0191
H ₆	0.4	10	27.2	0.0203
H ₇	0.4	18	28.9	0.0195
C ₁	0.2	10	23.78	0.02077
C ₂	0.2	18	24.52	0.01810
C ₃	0.3	10	25.07	0.01852
C ₄	0.3	18	26.10	0.01657
C ₅	0.3	27	27.53	0.01363
C ₆	0.4	10	27.77	0.02031
C ₇	0.4	18	28.15	0.01611

Note: H is the PP homopolymer; C is the PP/PE copolymer.

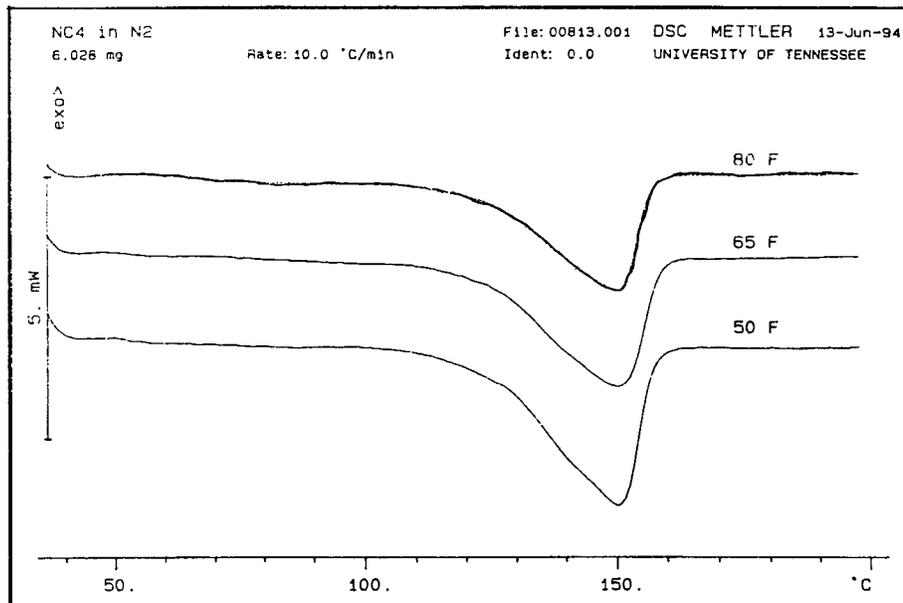


Figure 2 DSC scans for filament samples produced at different primary air temperatures.

creased as primary air temperature decreased, indicating that the low primary air temperature results in structure of the filaments with more crystallinity and, sometimes, more orientation of polymer molecules. The values of crystallinity (Table III) of both the filament and the nonwovens decreased as primary air temperature increased. The crystallinity decreased with increasing throughput. The results indicate that low primary air temperature is helpful for crys-

Table III Crystallinity from Density and DSC for PP Spunbond Filaments

PP Spunbond Filaments	Crystallinity (%) from Density	Crystallinity (%) from DSC
H ₁	52.04	57.54
H ₂	47.55	55.36
H ₃	49.35	55.77
H ₄	43.14	53.17
H ₅	42.01	52.6
H ₆	50.63	54.4
H ₇	48.19	53.24
C ₁	44.44	41.45
C ₂	43.89	40.41
C ₃	44.23	41.32
C ₄	43.62	40.18
C ₅	43.07	39.49
C ₆	43.34	41.19
C ₇	41.71	39.89

tallization of filaments. These results are consistent with that from density as shown in Table III. Figure 3 shows the difference of DSC scan between the PP homopolymer and the copolymer. As shown, the melting temperature and enthalpy of melting for the copolymer are lower than those of the homopolymer. That allows the bonding temperature and processing temperature of the copolymer to lower than that of the homopolymer. The lower enthalpy for the copolymer is an indication of lower crystallinity because the PE segments in PP are not favorable for crystallization of PP filaments during solidification of spinning process.

The thermomechanical behavior of the filaments and nonwoven samples are shown in Figures 4–7. Figure 4 shows the thermomechanical responses of the filaments produced at different primary air temperatures. The filaments produced at lower primary air temperatures were not easier deformed than those spun at higher temperatures. For same primary air temperature, the thermomechanical stability of filaments improved with decreasing throughput, as shown in Figure 5. That indicates that the morphology of the filaments spun at lower throughput was better developed than those produced with higher throughput. Figures 6 and 7 show the thermomechanical analysis (TMA) scans of nonwoven samples produced from different primary air temperature and

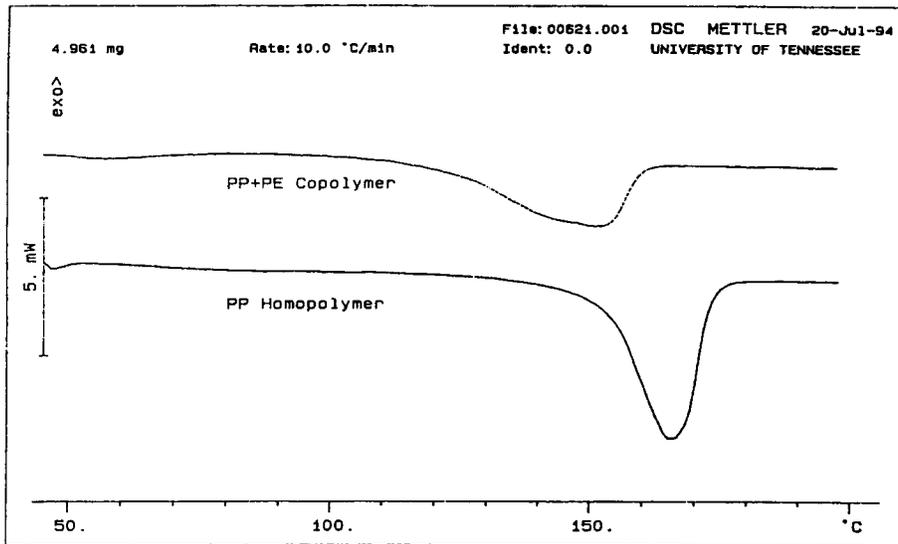


Figure 3 DSC scan for PP homopolymer and PP/PE copolymer.

throughput, respectively. The responses are consistent with those of filament samples. TMA studies indicate there is a large difference in the observed response for the filaments, especially in the temperature range of 90 to 120°C.

Figure 8 shows the dynamic TMA scan for filament samples. As shown, there is a transition temperature around 100°C. That means the mechanical properties of these samples can change

dramatically after this temperature. Primary air temperature shifts the transition temperature by about 10°C. Lower primary air temperature helps in crystallization and molecular orientation and, therefore, results in high transition temperature of the filaments. Figure 9 shows the difference of TMA scan of filament samples between the PP homopolymer and PP/PE copolymer. PP homopolymer filament shows better thermomechanical

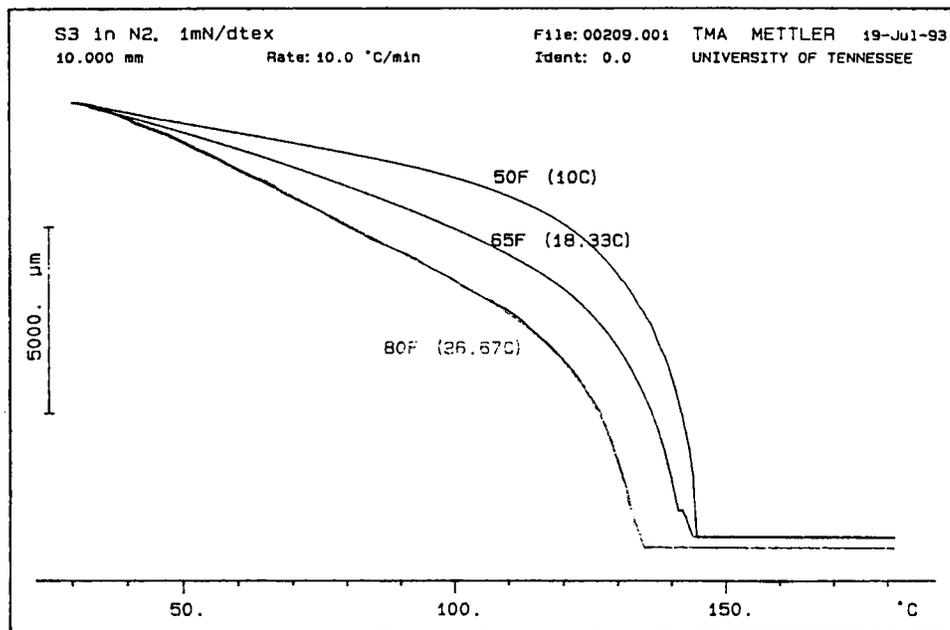


Figure 4 TMA scans for filament samples produced at different primary air temperatures.

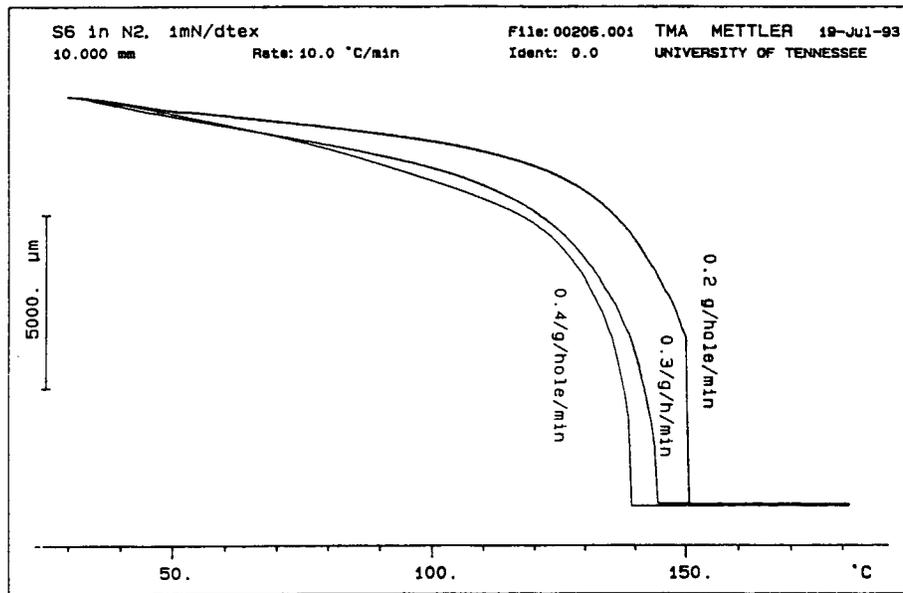


Figure 5 TMA scans for filament samples produced from different throughput at 10°C primary air temperature.

properties than those of PP/PE copolymer filaments. That is due to the fact that the PP homopolymer filaments are of higher crystallinity and molecular orientation than those of PP/PE copolymer filaments. This also results in the thermomechanical properties of PP homopolymer nonwovens being better than those of PP/PE copolymer nonwovens, as shown in Figure 10. More impor-

tantly, response of the filaments during the calendaring process will be different in the two cases, which, in turn, determine the properties of the bonded nonwovens.

Thermal elongation of samples at on-set melting temperature for two min measured at two different tensions is shown in Table VI. At both 0.1 and 0.033 g/den pretension, the thermal elonga-

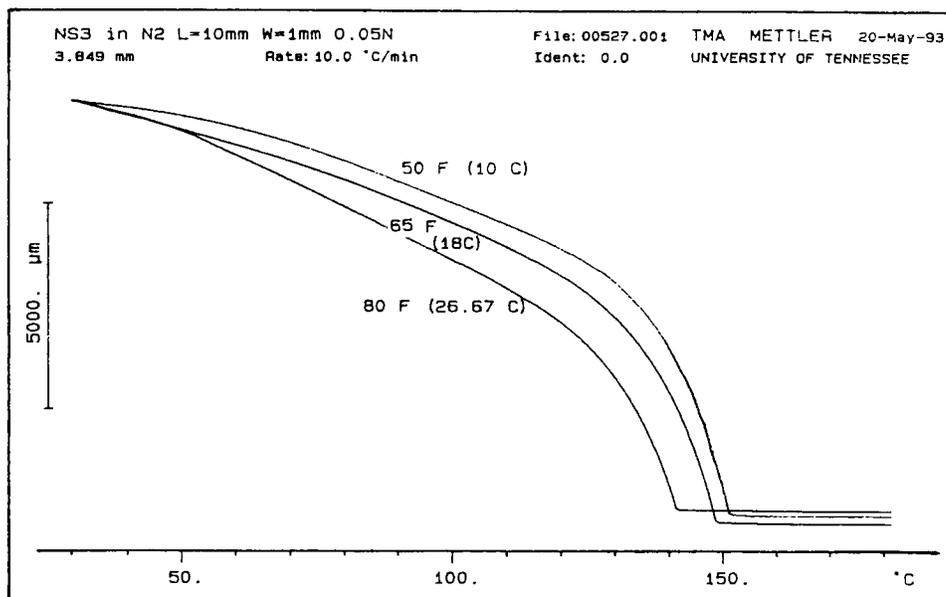


Figure 6 TMA scans for nonwoven samples produced at different primary air temperature ($0.2 \text{ g hole}^{-1} \text{ min}^{-1}$).

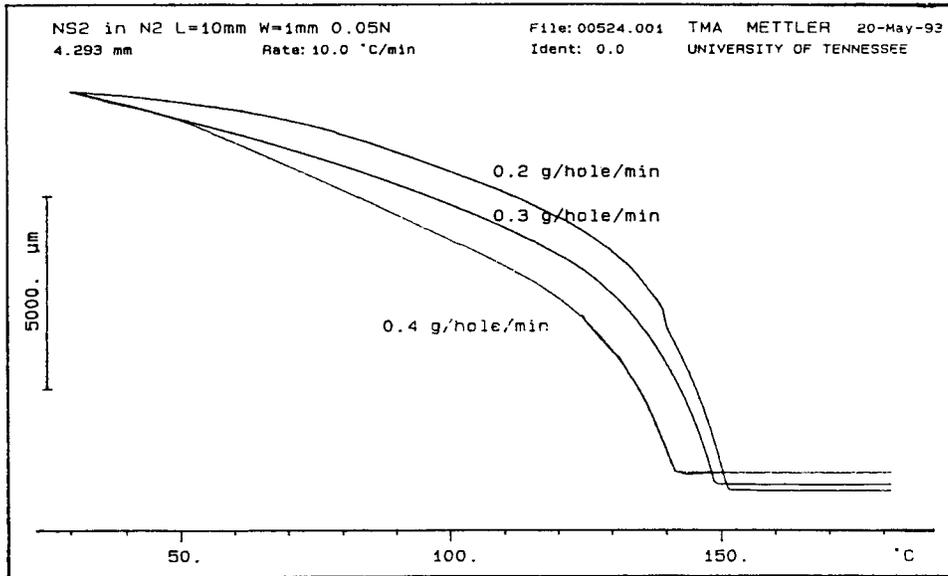


Figure 7 TMA scans for nonwoven samples produced from different throughput.

tion increased as the primary air temperature and throughput increased. The relatively large thermal elongation showed that the filaments have a lot of potential for drawing by decreasing the primary air temperature or increasing the draw-down air speed. With lower crystallinity and orientation of the samples, thermal elongation was higher. The trend observed is the same as seen with the homopolymer filaments.

Figure 11 shows thermomechanical responses of unbonded webs with different compression loads. An effort was made to understand how deformation of the filaments took place during the thermal calendaring under compression. For a constant compression load, the thickness of the web decreased as the temperature increased between 30 and 60°C, the thickness changed notably as the temperature increased. From 60 to 140°C,

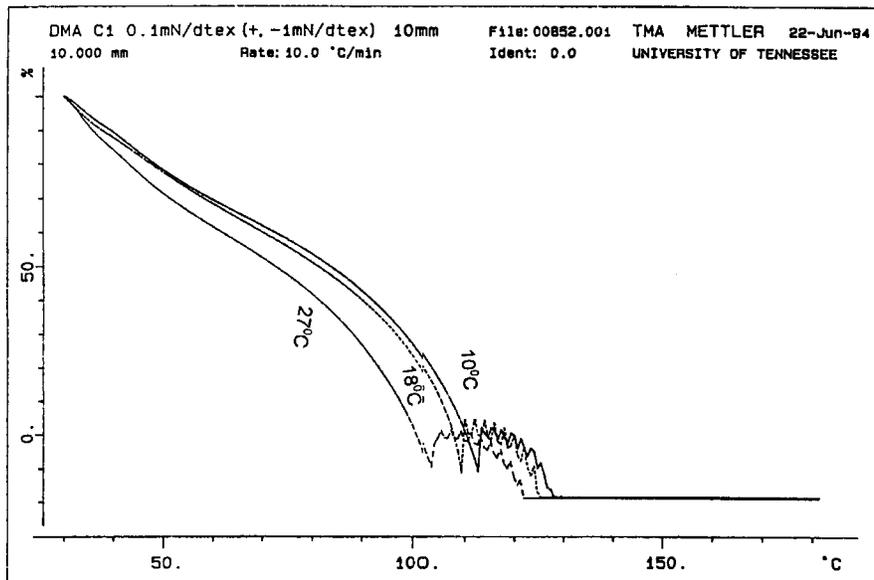


Figure 8 Dynamic TMA scan for copolymer filament samples at different primary air temperature.

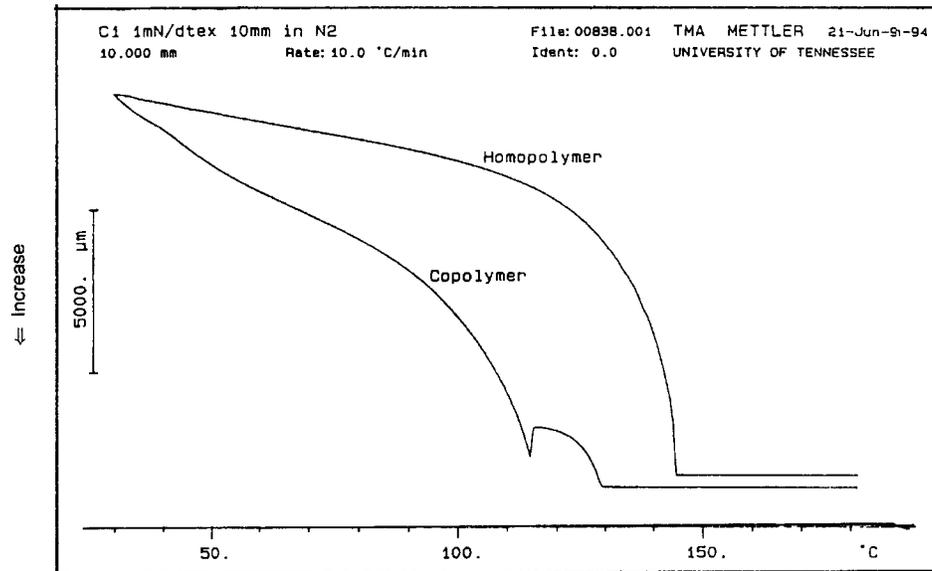


Figure 9 TMA scans for comparison of copolymer and homopolymer of filaments.

the change in the thickness was minimal. After 140 $^{\circ}\text{C}$, the thickness of the web changed rapidly until it melted around 165 $^{\circ}\text{C}$. The thickness of the web decreased as the applied compression load during the scan increased. However, because of the limitation of the instrument, the load used was much lower than actual pressures used in calendering.

Figure 12 shows the TMA scans at the same

compression load of the unbonded webs produced at different primary air temperatures. For the same temperature, the change in thickness of the web from low primary air temperature was smaller than those with high primary air temperatures. That means the filaments spun from low primary air temperature were not easily deformed compared to those from high primary air temperature. That is further confirmed from SEM photo-

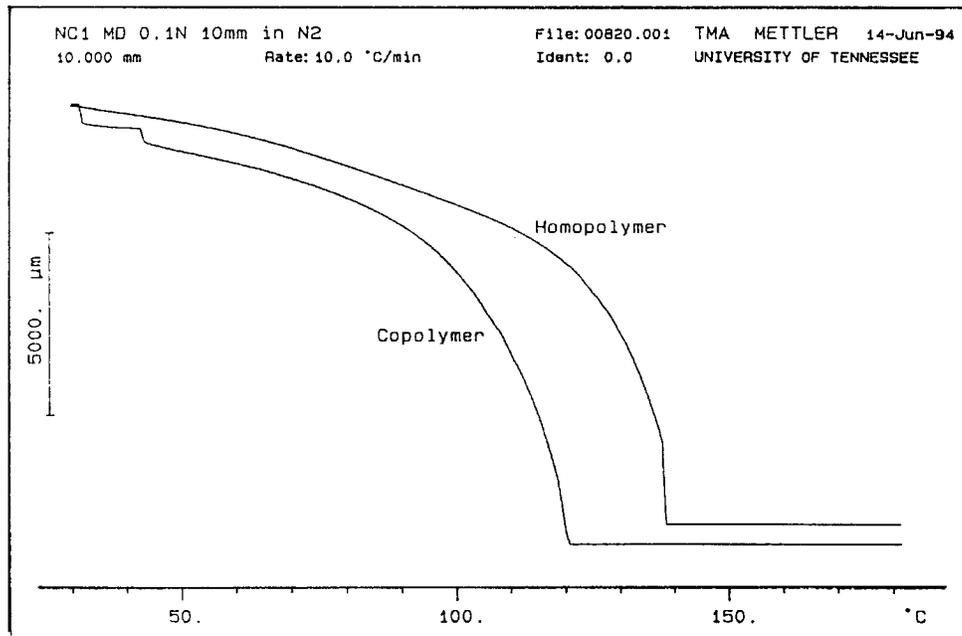


Figure 10 TMA scans for comparison of copolymer and homopolymer of nonwovens.

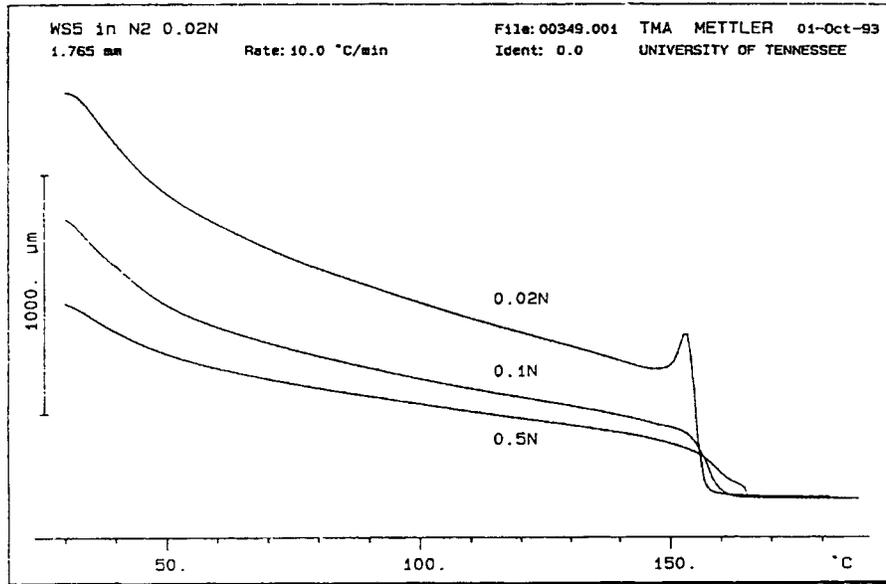


Figure 11 TMA scans for unbonded webs with different compression loads.

graphs of bonded fabrics, which will be discussed later. In the case of low primary air temperature, the bonded area is smaller than that of high primary air temperature. That is due to the fact that the filaments from low primary air temperature had high crystallinity and orientation.

Time-Dependent Thermal Properties

Figure 13 shows the isothermal TMA scans of filament samples at different temperatures for 60

min with a constant tension of 1 mN/dtex. As shown, the creep increased with time and temperature. The maximum extension took place in the first 10 min. After that, time-dependent elongation increased as the time increased slowly and evenly. Apparently, the creep increased proportionately as the temperature of scan increased from 30 to 90°C. However, there is a larger difference in the observed response between 90 and 120°C. This suggests that there is a significant

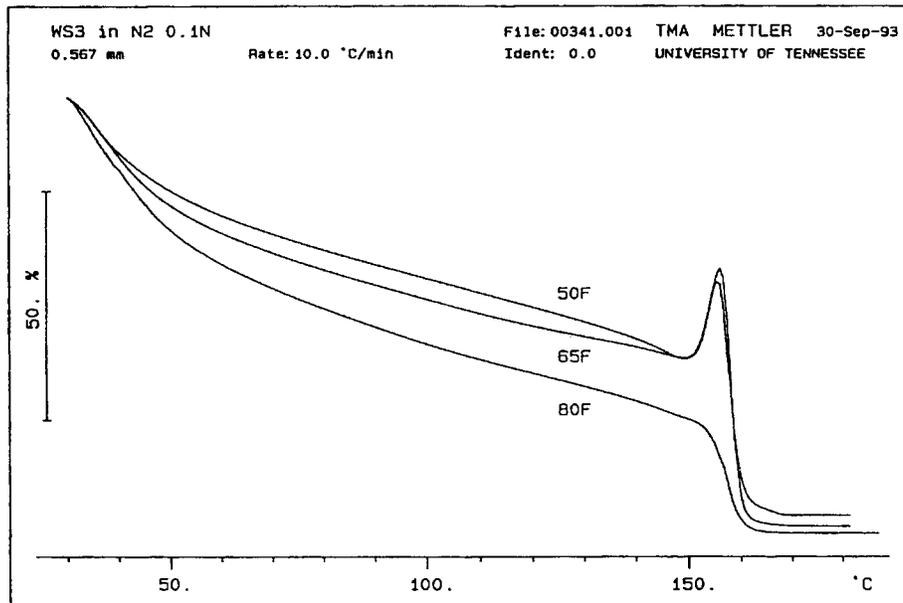


Figure 12 TMA scans for unbonded webs produced at different primary air temperatures.

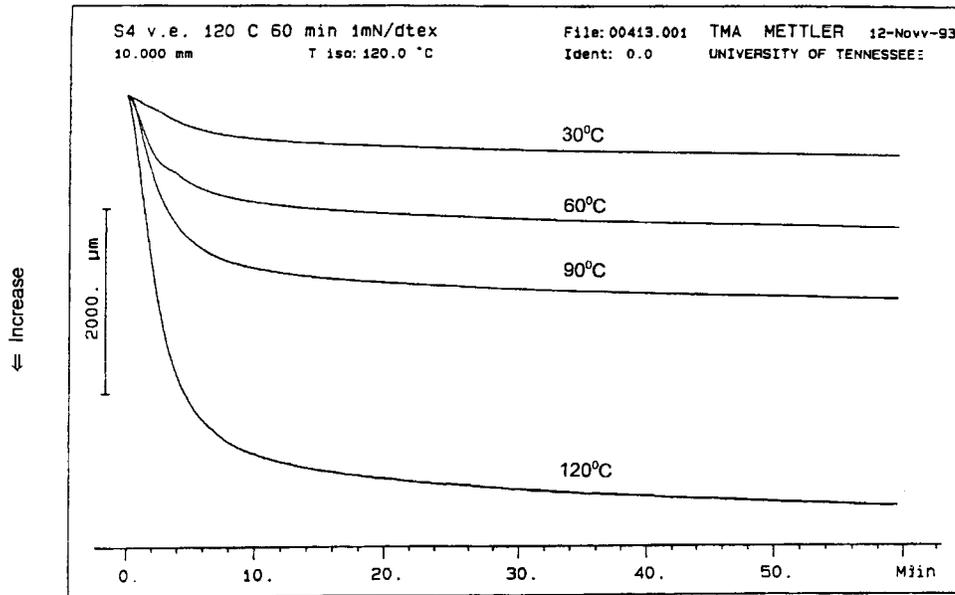


Figure 13 TMA scans of different constant temperatures for filament samples.

effect on mechanical properties at temperatures between 90 to 120°C for the PP filaments. Figure 14 shows the flow behavior of filament samples at a constant temperature of 30°C for 60 min, but with tensions of 1, 2, 4, and 5 mN/dtex. At 1 mN/dtex tension, the time-dependent elongation changed smoothly and slowly with time. The response was similar for 2 mN/dtex tension. How-

ever, when the tensions were 4 and 5 mN/dtex, the time-dependent elongation increased much faster. In about 10 min, the time-dependent elongation reached 100% of its original length.

Figure 15 shows the TMA scans of filament samples produced at the same throughput and different primary air temperature at a constant heating temperature of 30°C and a constant ten-

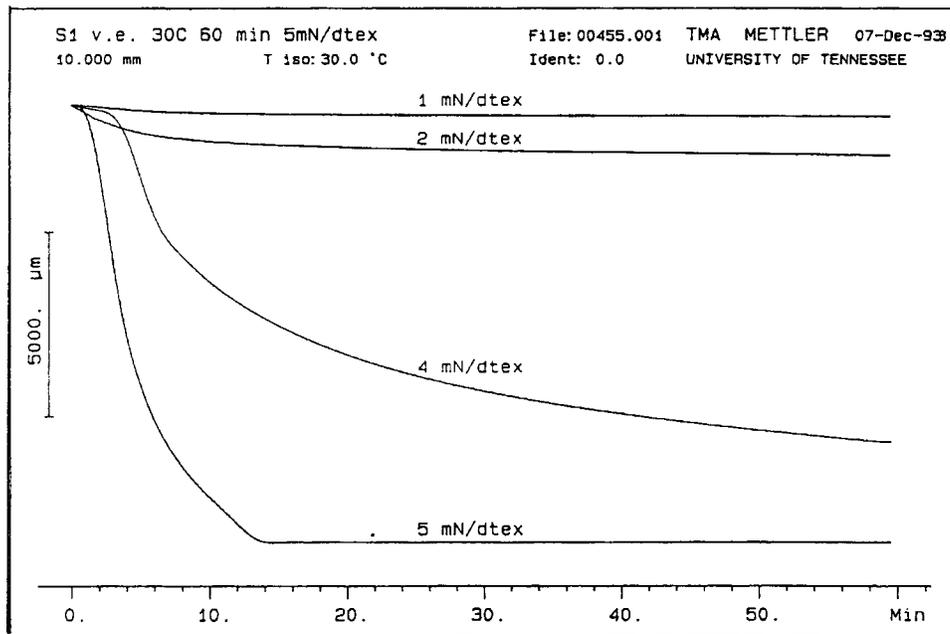


Figure 14 TMA scans of different constant tension for filament samples.

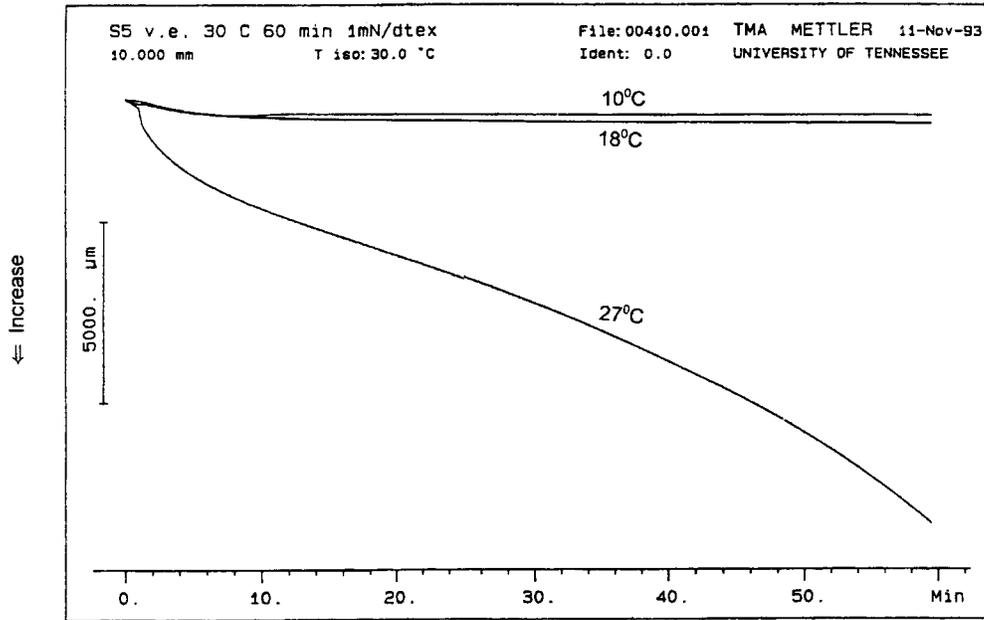


Figure 15 TMA scans for filament samples produced at different primary air temperature.

sion of 1 mN/dtex for 60 min. Creep of the filament of 50°F (10°C) quench air is slightly lower. TMA scans of filament samples with the same primary air temperature but different throughput, at a constant heating temperature of 30°C, and a constant tension of 1 mN/dtex for 60 min, as shown in Figure 16. The creep of the filaments increased as the throughput increased. The ob-

served difference in flow behavior of filaments is due to the difference in structure of these filaments. The smallest creep was observed for the filaments of high birefringence.

The relationship of breaking elongation between the filament and nonwoven samples shows that the higher the filament breaking elongation, the higher the elongation of the nonwoven sam-

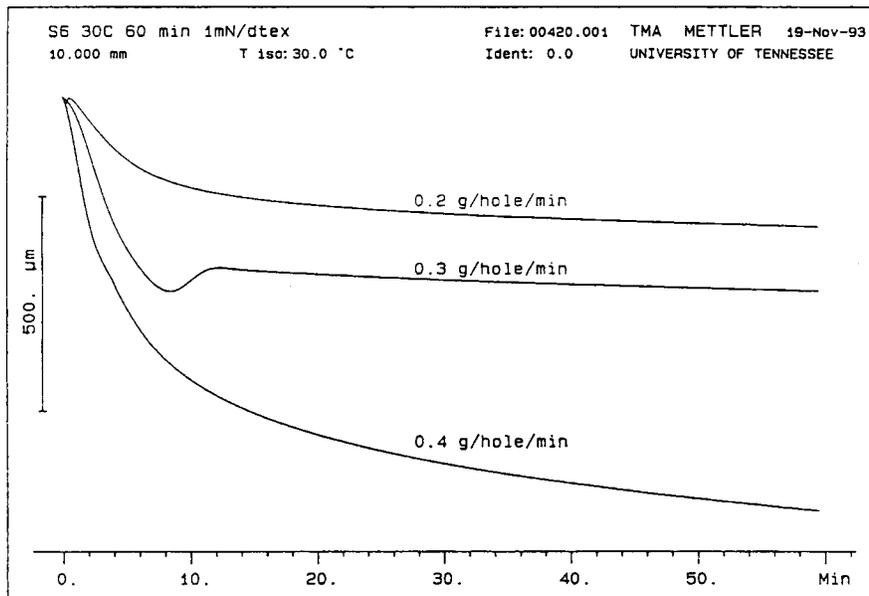


Figure 16 TMA scans for filament samples produced at different throughput.

Table IV Tensile Properties of PP Spunbonded Filaments

PP Spunbond Filament	Tenacity (mN/tex)	Elongation at Break (%)	Initial Modulus (mN/tex)
H ₁	424.3	180	72.0
H ₂	217.5	280	70.4
H ₃	153.1	269	69.9
H ₄	100.8	279	66.9
H ₅	87.5	294	64.5
H ₆	202.3	285	69.6
H ₇	101.2	289	62.7
C ₁	200.1	422	51.8
C ₂	182.7	540	40.5
C ₃	151.0	385	32.5
C ₄	100.9	495	18.5
C ₅	83.7	595	15.0
C ₆	90.9	446	31.9
C ₇	55.4	534	26.1

ples. The trends were similar to those observed with tensile strength and TMA analysis for both filament and nonwoven samples. As shown in Figures 5 and 6, at elevated temperatures, the filaments from higher quench air temperatures deformed easily; so did the nonwovens from higher quench air temperature. Also, scanning electron microscopy (SEM) photographs⁸ show a good relationship with TMA results as easily deformable filaments show high level of deformation in the nonwovens. The bonding area of nonwovens from higher quench air temperature clearly shows this effect.

Physical Properties

The birefringence of the fibers decreased as the throughput and primary air temperature increased. The higher the birefringence, the higher the orientation of the filaments. Therefore, as the primary air temperature increased, the orientation of the fiber decreased. This trend is related to changes in diameter of the fiber as, at the same throughput, lower primary air temperature results in smaller diameter of the fiber. This means the fiber is drawn to a greater extent by cooler air than that of higher temperature air. The low primary air temperature also helps to increase the orientation of the fibers. This point is proved by other characterizations too.

Tensile properties of the formed filaments are shown in Table IV, which show the same trends as seen with the homopolymer. The strength-at-break decreased with increase in the primary air

temperature. The elongation-at-break increased as the primary air temperature and the throughput increased. That further proved that the orientation of the molecules in filaments was higher for low primary air temperature than that for high primary air temperature.

Tensile properties of nonwoven samples are shown in Table V. Since the thermal bonding conditions were same for all the samples, the differences between the tensile properties of the samples reflect the differences in spinning conditions. The tensile strength decreased as the primary air temperature and the throughput increased. For the same reason, the elongation-at-break increased as the two process parameters increased. The strength-at-break of samples in the machine direction is apparently higher than that in the cross direction. The elongation-at-break for MD is smaller than that for CD for the same samples. This suggests that the orientation of filaments in the webs is not random and is preferential along the machine direction.

The tensile strength and initial modulus of copolymer filaments are lower than those of the homopolymer filaments. The elongation of copolymer filaments was higher than that of homopolymer filaments, which is due to the fact that the PE blocks in polypropylene reduce the crystallinity and molecular orientation of the filaments.

CONCLUSIONS

Thermal analysis of the spunbonded filaments and webs was conducted to study the relation be-

Table V Tensile Properties of Related Spunbond Nonwovens

PP Spunbond Nonwovens	Strength MD (mN/tex)	Strength CD (mN/tex)	Elongation at Break MD (%)	Elongation at Break CD (%)
NH ₁	46	21	168	171
NH ₂	45	23	175	212
NH ₃	41	13	168	148
NH ₄	38	21	179	217
NH ₅	34	21	182	241
NH ₆	37	16	171	163
NH ₇	35	22	178	201
NC ₁	32	21	102	137
NC ₂	31	20	110	152
NC ₃	27	20	106	151
NC ₄	26	15	130	162
NC ₅	26	14	147	166
NC ₆	26	18	106	151
NC ₇	22	17	119	174

tween the structure and properties of the filaments and the nonwoven samples of a polypropylene homopolymer and a copolymer of PP. The study showed that the primary air temperature, throughput, and thermal history have a strong influence on the structure and properties of both the filaments (before bonding) and the nonwovens (after bonding). DSC scans showed the crystallinity of filaments changed with thermal history. As the primary air temperature and throughput decreased, there was a tendency for decrease in filament diameter. The decrease in filament di-

ameter was accompanied by a simultaneous increase in their crystallinity, birefringence, tensile strength, initial modulus, thermal stability, and density.

Thermomechanical analysis of the filaments and nonwoven samples showed that filaments from low primary air temperature had better mechanical properties, as well as thermal stability. With well-developed morphology, the deformability of the filaments decreased during subsequent thermomechanical operation. This was clearly evident in the bond patterns of the calendared fabrics. DSC scans did not show any difference in melting point for the filaments or nonwoven samples, but the melting enthalpy increased as the primary air temperature decreased. TMA studies indicated that there was a large difference in the observed response for the filaments, especially in the temperature range of 90 to 120°C.

Dynamic thermomechanical analysis showed there was a transition temperature at about 110°C for copolymer filaments. Time-dependent thermal analysis also showed that there was a significant change between 90 and 120°C. TDA showed that the filaments have a lot of potential for drawing by decreasing the primary air temperature or increasing the drawdown air speed.

The larger diameter fibers also had higher breaking elongation and thermal elongation, which indicated the drawability of the fibers. Lower primary air temperature and throughput produced the filaments with well-developed struc-

Table VI Thermal Elongation at Onset Melting Temperature for 2 Min with Given Tension

PP Spunbond Filaments	Elongation at 0.1 (g/den) Pretension (%)	Elongation at 0.033 (g/den) Pretension (%)
H ₁	17	8
H ₂	23	15
H ₃	31	17
H ₄	36	18
H ₅	42	26
H ₆	33	10
H ₇	54	13
C ₁	31	11
C ₂	38	19
C ₃	40	22
C ₄	45	24
C ₅	49	30
C ₆	39	13
C ₇	47	17

ture and properties. Diameter increase with the increasing primary air temperature may be due to the lower level of spinline stress under those conditions. Higher stress in the threadline leads to a greater reduction in fiber diameter and increase in birefringence. X-ray diffraction studies showed that crystal orientation and crystallinity of homopolymer samples are higher than those of copolymer samples. A mathematical model is being developed for quality control of spunbonding process and objective evaluation of nonwovens quality with regards to their perception of mechanical hand, drape, and so on. Data obtained from the Kawabata Evaluation System is being used to calculate the total hand quality of the fabrics.

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