

Dedicated to Professor Bernhard Wunderlich on the occasion of his 65th birthday

THERMAL ANALYSIS OF POLY(2-METHYLPENTA-METHYLENE TEREPHTHALAMIDE)

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Abstract

Poly(2-methylpentamethylene terephthalamide) (Nylon M5T) is a new high temperature aromatic polyamide developed by Hoechst Celanese. In this paper thermal properties of Nylon M5T chips, as well as as-spun and drawn fibers were studied by DSC, DMA, hot stage microscopy and WAXS. T_g of the fully amorphous Nylon M5T is 143°C when measured by DSC; T_g increases with crystallinity to 151°C. The temperature dependence of the solid and melt specific heat capacities has also been determined. The heat capacity increase at the glass transition of the amorphous polymer is 103.9 J °C⁻¹ mol⁻¹. T_g by DMA for the as-spun fiber is 155°C, for a drawn fiber is 180°C. Three secondary transitions were observed by DMA in addition to the glass transition. These correspond to a local mode relaxation of the methylene groups at -120°C, onset of rotation of the amide-groups at -65°C and the onset of the rotation of the phenylene-groups (at 63°C). The crystallinity of Nylon M5T strongly depends on the rate of cooling from the melt. The isothermal crystallization data are melt temperature dependent: two-dimensional crystallization takes place when the samples are crystallized from higher melt temperatures, and this phase changes into a spherulitic structure during cooling to room temperature. Spherulitic crystallization occurs when lower melt temperatures are used. This polymer has three crystal forms as indicated by DSC, DMA and WAXS data. The crystal to crystal transitions are clearly visible when amorphous samples are heated in the DSC, or the DMA curves of as-spun fibers are recorded. It is experimentally shown that a considerable melting of the lower temperature crystal forms takes place during the crystal to crystal transitions. The equilibrium melting point as measured by the Hoffman-Weeks method, has been determined to be 339°C.

Keywords: crystal-crystal transitions, crystal forms, DMA, DSC, Nylon M5T, X-ray

Introduction

Poly(2-methylpentamethylene terephthalamide) (Nylon M5T) is a new high temperature partially aromatic polyamide developed in Summit by Hoechst Celanese [1]. Originally this polymer was developed for tire-cord applications, as well as for applications for which the hydrolytic stability of PET is inadequate. Nylon M5T may also find application in the preparation of mono-filaments for pulp-filters and drying drums used by the paper industry because

of its good high temperature hydrolytic stability. It has also some advantages when compared to other Nylons (like 6,6) due to its higher glass transition temperature. However, as will be seen from the results reported here, the thermo-oxidative stability of this polymer is poor, and this may limit its industrial applications.

Very limited information is available about the properties of this polymer [1–3], and there is only one paper on its thermal properties [4]. This paper will describe the results obtained on Nylon M5T using numerous characterization techniques, such as DSC, DMA, hot stage microscopy and WAXS.

Experimental

The Nylon M5T polymer (IV=0.92 in 0.1% solution in hexafluoro-isopropanol) has been prepared from terephthalic acid and 2-methylpenta-methylene diamine as will be published later [1].

The DSC measurements were carried out using a TA Instruments 2100/910 DSC and 2100/2920 modulated DSC, and also a Perkin Elmer DSC7. When evaluating the DSC traces, the following parameters were obtained from heating traces: T_g is the glass transition temperature, which was taken as the temperature at half of the heat capacity increase, °C; T_{mp} , the peak temperature of melting, °C; T_m , the melting point, which is the highest temperature point of the melting endotherm, °C; ΔH_f is the heat of fusion, J g⁻¹; ΔC_p is the heat capacity increase at the glass transition, J°C⁻¹mol⁻¹. On cooling runs, the following parameters were obtained: T_{co} , the starting temperature of crystallization, °C; T_{cp} , the peak temperature of crystallization, °C.

The TG measurements were carried out using a Perkin Elmer TGA7. DMA measurements were performed on a Rheometrics RSA2 using as-spun and drawn Nylon M5T fibers. Polarization optical microscopy observations were done at a magnification of 400× on a Nikon microscope fitted with a Leitz 1350 hot stage and a Micricon 823 temperature controller using 5µm thick microtomed samples and freezer-milled fiber samples.

Wide angle X-ray scattering experiments were carried using the INEL 120 curve position sensitive detector mounted on a Philips goniometer utilizing monochromatic copper K_α radiation. A furnace with temperature controller is also mounted on the instrument for in-situ high temperature experiments. Both the controller and data acquisition system are handled by the computer for full automation.

Results and discussion

A. Crystal to crystal transitions (different crystal forms)

Nylon M5T (formula shown in Fig. 1) is a semicrystalline polymer. It is very difficult to crystallize Nylon M5T by cooling from the melt, cooling rates as

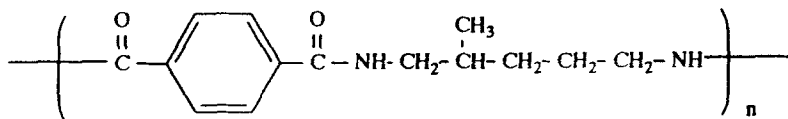


Fig. 1 The formula of the repeating unit of Nylon M5T

low as $10^\circ\text{C min}^{-1}$ quench this polymer to a completely amorphous state. When the amorphous polymer is heated, multiple exothermic-endothermic transitions are observed, as shown in Fig. 2 as a function of heating rate (2, 5, 10, 20 and $40^\circ\text{C min}^{-1}$). It is likely that this behavior is caused by the existence of several crystal forms. When these crystal forms are designated with A, B, and C, then the first exotherm corresponds to the melt \rightarrow crystal A transition (i.e., cold crystallization), and it is strongly dependent on the heating rate. This exotherm

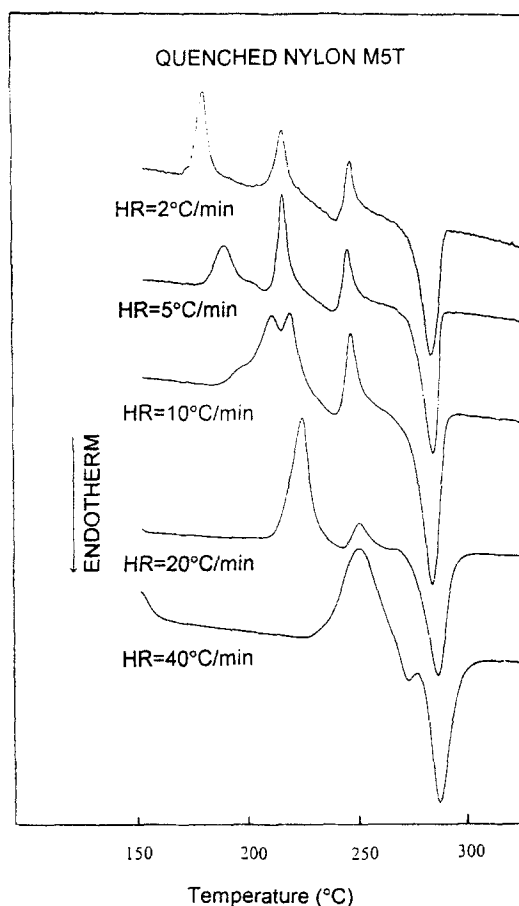


Fig. 2 The heating DSC traces of quenched (amorphous) Nylon M5T recorded at different heating rates (HR). The heating rate is shown at each curve

can be found for the heating rates of 2, 5, and $10^{\circ}\text{C min}^{-1}$. Its intensity slightly decreases with increasing heating rate, and it shifts to higher temperatures. At heating rates of 20 and $40^{\circ}\text{C min}^{-1}$ this exotherm, i.e. the melt \rightarrow crystal A transition, cannot be found, it is likely, that a melt \rightarrow crystal B transition takes place at higher heating rates. The second exothermic process at the lower heating rates is the crystal A \rightarrow crystal B transition, and the heating rate dependence of this transition is negligible up to $10^{\circ}\text{C min}^{-1}$. Beyond $10^{\circ}\text{C min}^{-1}$, this transition does not exist, because no crystal form A can be developed at higher heating rates as mentioned above (probably due to kinetic reasons). Finally, the third exotherm corresponds to the crystal B \rightarrow crystal C transition, and its heating rate dependence is also negligible below ca. $20^{\circ}\text{C min}^{-1}$. As expected, the resolution at 20 and $40^{\circ}\text{C min}^{-1}$ heating rates is poor, but the exothermic transitions still can be identified.

Polarization optical microscopy experiments are not too helpful, even at magnifications of $2000\times$ in following the cold- and recrystallization behavior of originally amorphous Nylon M5T samples. This is because of the extremely small crystallite size formed during cold crystallization.

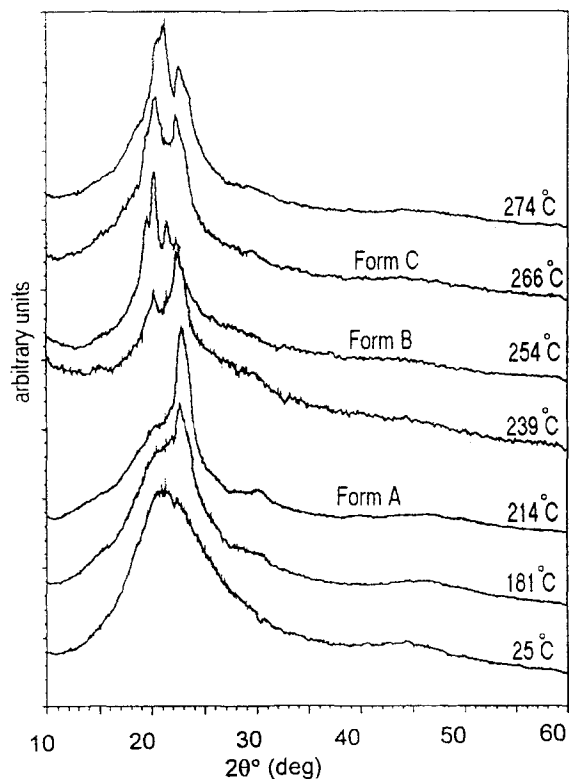


Fig. 3 The wide angle X-ray diffraction patterns of amorphous Nylon M5T chip recorded at different temperatures

WAXS experiments were carried out on powder samples quenched in DSC pans from temperatures corresponding to the formation of crystal forms A, B and C. The results are shown in Fig. 3 along with the results from the as-quenched powder from melt. It can be observed that the melt quenched powder shows only a broad amorphous peak at $\sim 4.4\text{\AA}$ attributed to the average chain to chain correlation distance. The major peaks of forms A and B appear to be very similar ($\cong 4.2, 4.1$ and 3.1\AA), however, there are some small differences manifest in three additional peaks ($7.1, 4.8$ and 2.4\AA) which can be observed in the form B sample. Form C shows a distinct peak at 13.1 and 4.5\AA . A large d -spacing, typically may suggest smectic type structure.

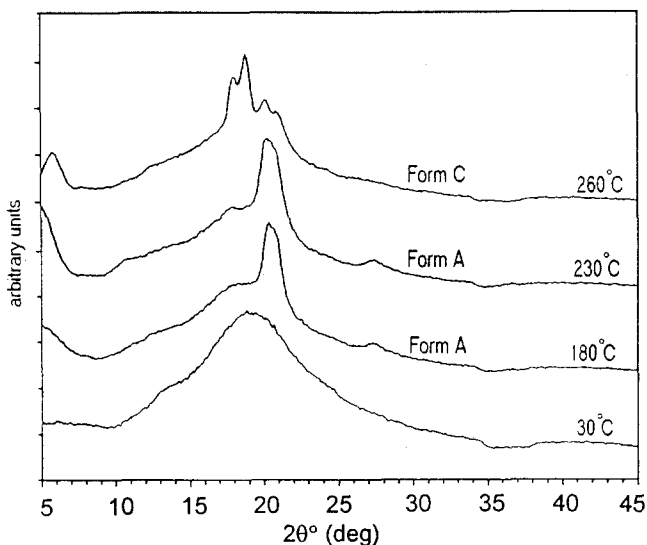


Fig. 4 The wide angle X-ray diffraction patterns of as-spun Nylon M5T fiber recorded at different temperatures

In order to elucidate the differences in these crystal forms, elevated temperature wide angle X-ray diffraction was also carried out on quenched (i.e., amorphous) Nylon M5T chip and as-spun Nylon M5T yarn in order to confirm the crystal to crystal transitions. The temperature was increased at approximately 2°C min^{-1} . Representative X-ray patterns are shown in Figs 3a and 4 for the amorphous chip and fiber, respectively. Again, the as-quenched form melt indicates a broad amorphous peak at 4.4\AA . At ca. 180°C crystallinity (cold crystallization) develops indicated by the emergence of a peak at 4.1\AA . Because of instrumental resolution, this is probably a combination of two peaks observed as 4.1 and 4.2\AA . At 239°C , an additional peak is observed which can be interpreted as a re-ordering of the crystal structure. However, at 254°C , additional peaks at $4.8, 4.65$ and 4.3\AA are observed with the reduction in intensity of the 4.2\AA peak suggesting a transformation from crystal form A to

form B. On increasing temperature, the pattern changes with more emphasis in the 4.6 Å peak, the development of the 4.2 Å peak and the disappearance of the 4.3 Å peak. Again, these changes indicate a transformation of crystal structure from form B to form C.

The results from the as-spun fiber along the meridian direction with increasing temperature are shown in Fig. 4. Initially, the as-spun fiber shows a broad amorphous peak. Crystallinity develops at ca. 180°C (cold crystallization). A possible change in the crystal structure occurs at 230°C, and a definite crystal to crystal transition takes place at 260°C. The crystal A → crystal B transition is not as clear in the fiber samples as in the powders. This may be due to differences in heating rates. Form C showed an additional peak at large d -spacing (13.1Å). This result suggests that the diffraction of form C is consistent with smectic liquid crystal structure.

DMA measurements were carried out on the as-spun and drawn M5T yarns. The dependence of the tensile storage modulus on temperature is shown in Fig. 5. As can be seen on this figure, the behavior of the drawn fiber is as usual: there is an order of magnitude decrease in the value of the storage modulus at the glass transition temperature (starting at 150°C), which is characteristic of semicrystalline polymers. The behavior of the as-spun fiber is drastically differ-

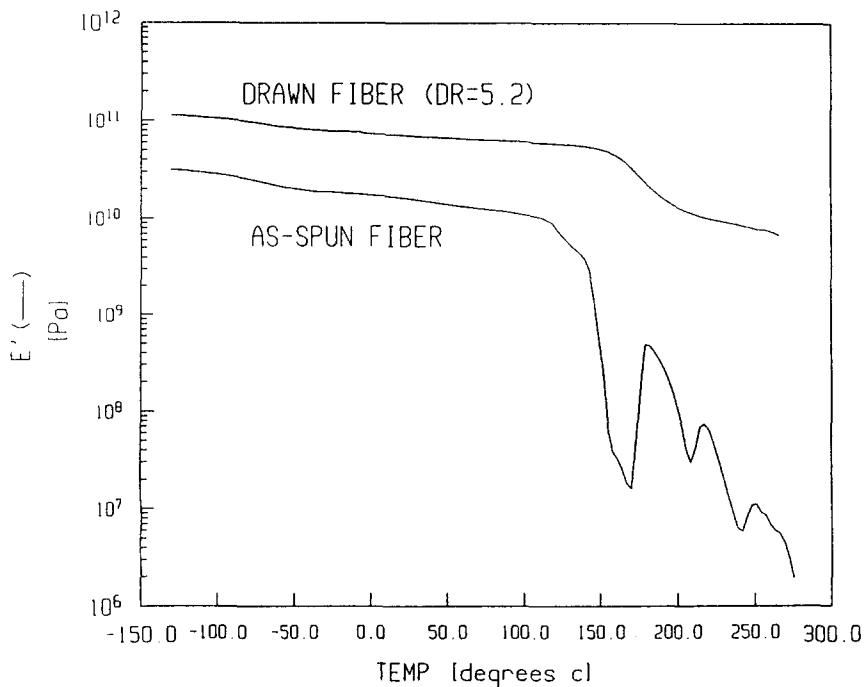


Fig. 5 The temperature dependence of the tensile storage modulus of the drawn and as-spun Nylon M5T fibers

ent. At the glass transition temperature there is a ca. 3 orders of magnitude decrease in the tensile storage modulus, which is characteristic of amorphous polymers. Then, at 170°C, the modulus suddenly increases due to the onset of the cold crystallization (i.e., melt \rightarrow crystal A transition) process. After the completion of the cold crystallization, the modulus decreases again, and increases starting at 210°C, which is an indication of the crystal A \rightarrow crystal B transition. Then, after a further decrease, the modulus increases once again, this time due to the crystal B \rightarrow crystal C transition, and a further decrease is observed at higher temperatures due to melting of crystal form C. This DMA-trace indicates multiple crystal to crystal transitions. The strong decrease of the tensile storage modulus of the M5T yarn is an experimental proof that the crystal to crystal transitions proceed through considerable melting.

Thus, the DMA measurements confirmed that three crystal forms exist for Nylon M5T. When the DSC and DMA results are compared to the WAXS observations, it is likely that crystal forms A and B are similar in structure. These crystal forms can be prepared using the following procedure. Amorphous M5T sample (or as-spun fiber) is heated to 180°C, and cooled to room temperature producing crystal form A. The DSC trace of the amorphous sample is shown in Fig. 6 (curve #1), exhibiting three exothermic peaks (one melt-to-crystal and two crystal-to-crystal transitions). The DSC trace of crystal form A is shown by curve #2 (two exothermic peaks corresponding to two crystal-to-crystal transi-

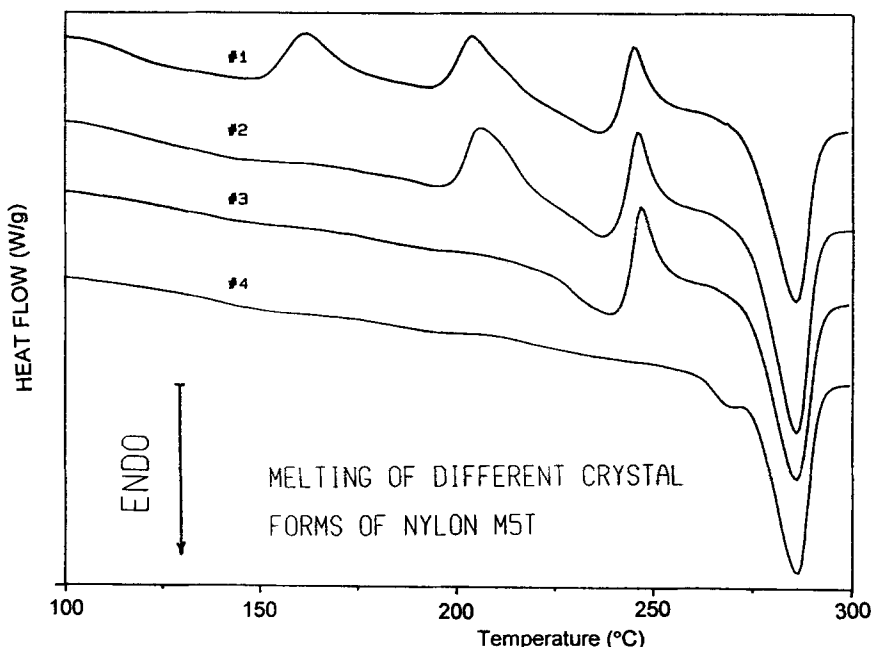


Fig. 6 The heating DSC traces of the different crystal forms of Nylon M5T: curve #1: amorphous M5T, curve #2: crystal form A, curve #3: crystal form B, curve #4: crystal form C. Heating rate = 10°C min⁻¹

tions). When the amorphous sample is heated to 210–220°C and cooled back to room temperature, crystal form B is produced; its DSC trace is shown in curve #3 (one exothermic peak corresponding to the crystal B → crystal C transition). Finally, when the amorphous sample is heated to 250–260°C, and cooled back to room temperature, crystal form C is produced, and its DSC trace is shown by curve #4 in Fig. 6.

B. Non-isothermal crystallization

As mentioned above, relatively slow cooling rates (i.e., 10°C min⁻¹) from the melt will quench this polymer to a completely amorphous state. Non-isothermal crystallization experiments can be carried out only at very slow coolings. The effect of the cooling rate on the DSC trace recorded during the subsequent heatings is shown in Fig. 7 for cooling rates of 10, 5 and 2°C min⁻¹ (the heating rate

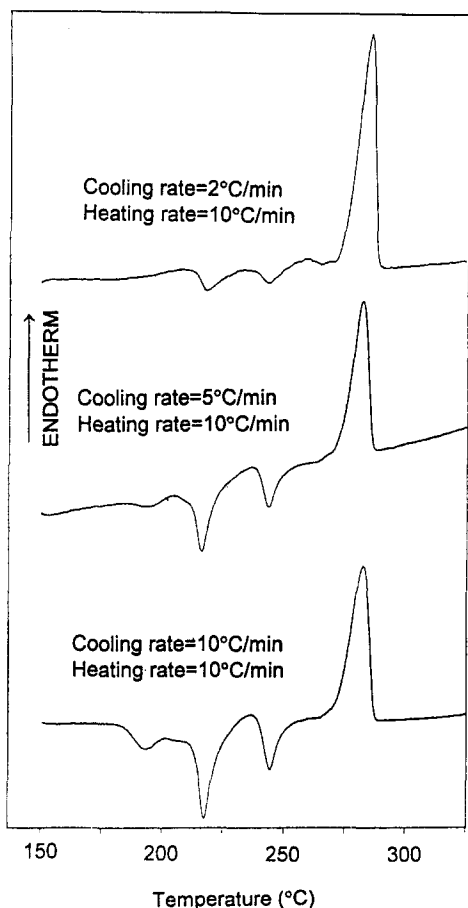


Fig. 7 The effect of the rate of cooling from the melt on the subsequent reheating (CR = cooling rate; HR = heating rate)

Table 1 Non-isothermal crystallization and reheating of Nylon M5T

C. R.	Cooling		Reheating			ΔH_f
	T_{co}	T_{cp}	T_g	T_{mp}	T_m	
2	245	220	148	279	286	40.2
5	239	212	144	281	288	26.8
10	—	—	143	281	288	—

$C. R.$ is the cooling rate, $^{\circ}\text{C min}^{-1}$

T_{co} is the starting temperature of crystallization, $^{\circ}\text{C}$

T_{cp} is the peak temperature of crystallization, $^{\circ}\text{C}$

T_g is the glass transition temperature, $^{\circ}\text{C}$

T_{mp} is the peak temperature of melting, $^{\circ}\text{C}$

T_m is the melting point, the last, highest temperature point of the melting endotherm, $^{\circ}\text{C}$

ΔH_f is the heat of fusion, J g^{-1}

is $10^{\circ}\text{C min}^{-1}$ for all three curves). The crystal \rightarrow crystal transitions can be found on all these DSC traces, although with smaller intensity than on the amorphous sample. The peak temperatures corresponding to the crystal A \rightarrow crystal B and the crystal B \rightarrow crystal C transitions are unchanged. This means that the fraction of crystal form C increases with decreasing cooling rate. This is an expected result, because crystal form C must be formed at the highest tem-

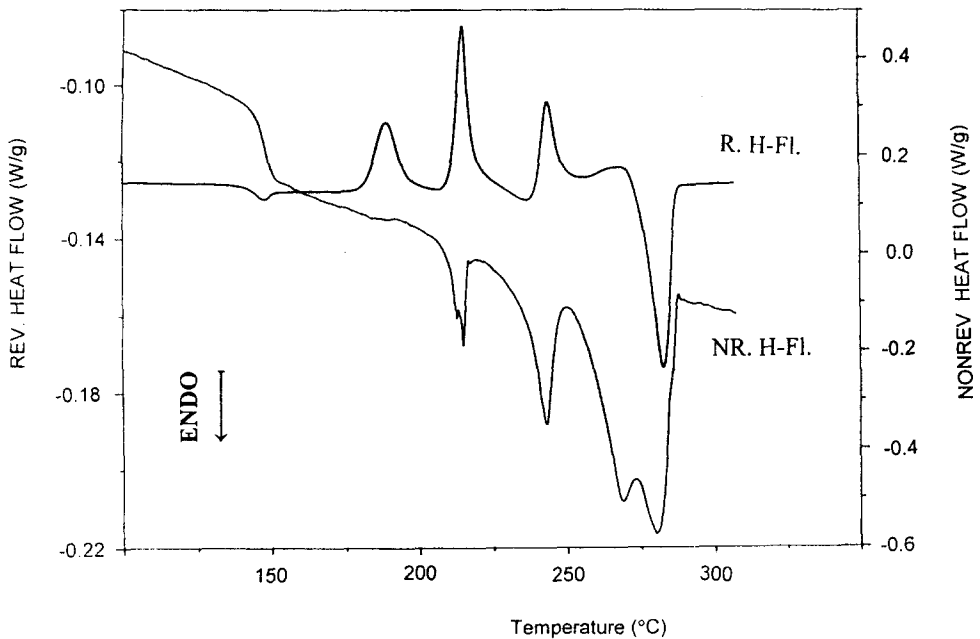


Fig. 8 The reversible and non-reversible heat flow during heating an amorphous M5T sample. R. H-FI is the reversible heat flow DSC curve; NR. H-FI is the non-reversible heat flow DSC curve

peratures, and thus, the decrease in the cooling rate will favor the conditions for development of crystal form C. Therefore, it may be suggested that the development of the different crystal forms is purely temperature dependent: during heating and cooling of the amorphous Nylon M5T, crystal form C develops at the highest temperatures (above 220°C), crystal form B develops between 180 and 220°C, and crystal form A develops below 180°C. The basic non-isothermal thermal data for the crystallization and melting experiments of Nylon M5T are shown in Table 1. Since the crystallinity of the samples increases with decreasing cooling rate, it is obvious that the glass transition is somewhat crystallinity dependent. T_g is 143°C for the purely amorphous sample, and goes up to 148°C for the more crystalline sample produced at a cooling rate of 2°C min⁻¹. The highest glass transition temperature was obtained for the sample crystallized isothermally (see below) at 267.5°C: $T_g = 151^\circ\text{C}$ (Fig. 15).

The DSC-traces recorded by a modulated DSC are very helpful when multiple peak processes are studied. The reversible and non-reversible heat-flow recorded during a 5°C min⁻¹ heating for the M5T samples cooled fast (amorphous M5T), and at 5, 2, and 1°C min⁻¹ coolings are shown in Figs 8–11. The curves on these DSC-traces indicate that

1. The crystal-to-crystal transitions really do proceed through considerable melting supporting the conclusions of the DMA measurements (see the non-reversible heat flow in Fig. 8).

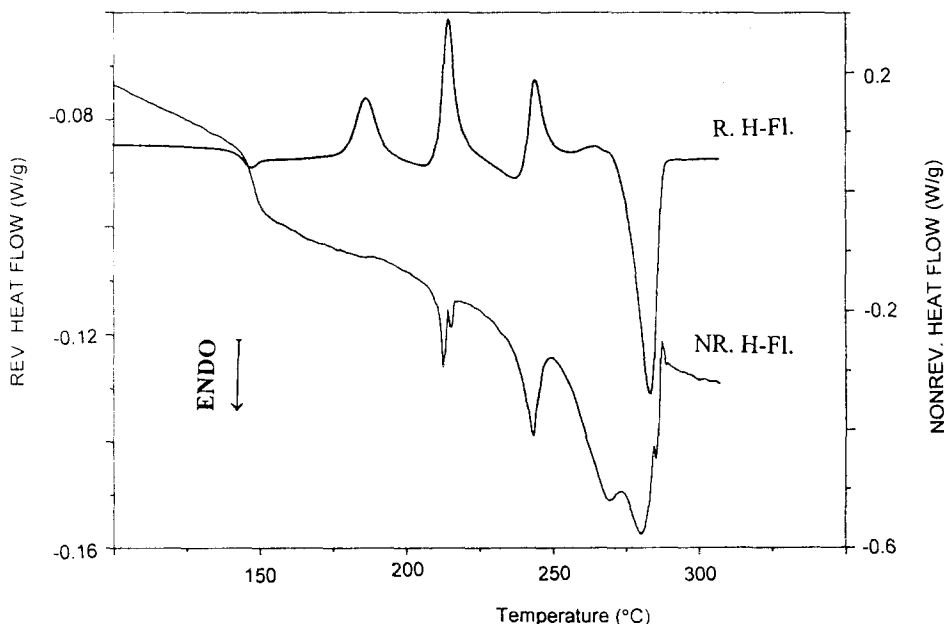


Fig. 9 The reversible and non-reversible heat flow during heating an M5T sample crystallized at a cooling rate of 5°C min⁻¹. R. H-FI is the reversible heat flow DSC curve; NR. H-FI is the non-reversible heat flow DSC curve

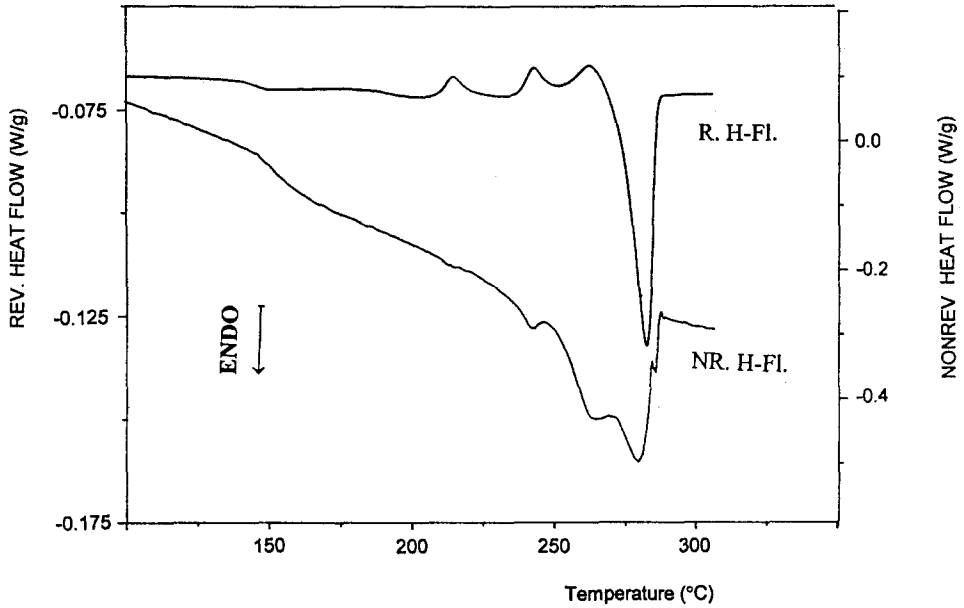


Fig. 10 The reversible and non-reversible heat flow during heating an M5T sample crystallized at a cooling rate of $2^{\circ}\text{C min}^{-1}$. R. H-FI is the reversible heat flow DSC curve; NR. H-FI is the non-reversible heat flow DSC curve

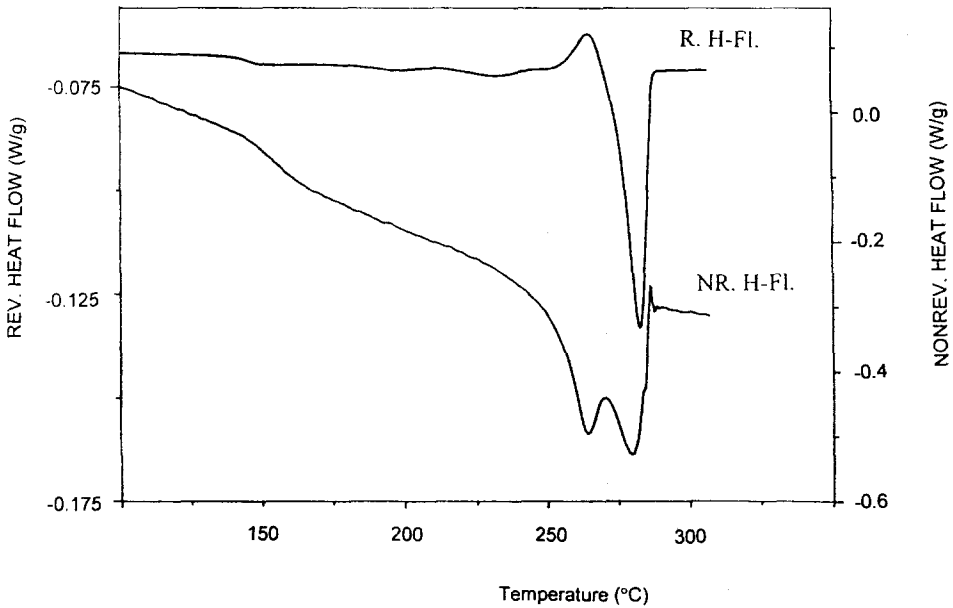


Fig. 11 The reversible and non-reversible heat flow during heating an M5T sample crystallized at a cooling rate of $1^{\circ}\text{C min}^{-1}$. R. H-FI is the reversible heat flow DSC curve; NR. H-FI is the non-reversible heat flow DSC curve

2. The intensity of both the melting and crystallization processes decrease as the crystallinity of the M5T sample increases for the slower cooling rate samples. This is a consequence of a smaller amount of the amorphous portion of M5T available for crystallization. For a sample cooled at $1^{\circ}\text{C min}^{-1}$ from the melt, no crystal-to-crystal transitions take place. This confirms that crystal C (that is the highest temperature crystal form) develops at slow coolings from the melt.

The separation of the reversible and non-reversible signals by the temperature modulation helps to determine the (non-equilibrium) melting temperatures of the different crystal forms at a heating rate of $10^{\circ}\text{C min}^{-1}$. This is summarized in Table 2.

Table 2 The melting point data of different crystal forms of Nylon M5T

Crystal form	T_{mp}	T_m
A	215.0	219.5
B	243.0	250.0
C	280.0	288.5

T_{mp} is the peak temperature of melting, $^{\circ}\text{C}$

T_m is the melting point, the last, highest temperature point of the melting endotherm, $^{\circ}\text{C}$

C. Isothermal crystallization and the equilibrium melting point

Nylon M5T is a unique polymer. As described above, it is extremely difficult to crystallize it by cooling from the melt, but it can be easily isothermally crystallized from the melt over a wide temperature range (from 235°C to 275°C). These crystallization experiments were evaluated using the Avrami equation. When the samples are heated to 320°C (T_F), and their temperature is dropped to the temperature of isothermal crystallization (T_c), a value of 2 is obtained for the Avrami exponent. In polarization optical microscopy experiments the birefringence of the sample increased at the crystallization temperature, but no spherulite formation was observed. Unfortunately, 320°C is fairly close to the thermal degradation temperature of Nylon M5T, therefore, the isothermal crystallization was also performed from $T_F=300^{\circ}\text{C}$ as melt temperature. In these experiments the value of the Avrami exponent was close to 3 and distinct spherulite formation was observed under the microscope in these cases, but only in part of the optical field. A large area of the field remained dark, and formation of much larger spherulites was observed in these areas during cooling to room temperature. The development of these larger spherulites could not be verified by DSC (no exothermic peak was recorded when the isothermally crystallized samples were cooled to room temperature). In summary, it is likely that spherulitic crystallization takes place with heterogeneous nucleation from $T_F=300^{\circ}\text{C}$, and probably two-dimensional crystallization from $T_F=320^{\circ}\text{C}$. It is unknown presently whether this corresponds to the development of a smectic phase.

The isothermally crystallized samples (from $T_F=300^\circ\text{C}$) were reheated at a rate of $10^\circ\text{C min}^{-1}$ in order to determine the equilibrium melting point of this polymer by the Hoffman-Weeks method. As expected, only the highest crystallization experiments (T_c from 260°C to 275°C) could be used to determine the equilibrium melting point: a value of $T_m^\circ = 339^\circ\text{C}$ was obtained (Fig. 12). Double melting peaks were recorded in most cases when the isothermally crystallized samples were remelted (Fig. 13). However, as experiments with different heating rates revealed, these double melting peaks have nothing to do with the presence of several crystal forms. The melting curves of samples crystallized at 247.5°C , and heated at 2, 10, and $40^\circ\text{C min}^{-1}$ are shown in Fig. 14. As can be seen, the ratio of the higher to lower temperature peak amplitudes decreases with increasing heating rate. Therefore, it is likely that crystal form C develops in all these cases, and the double character of the melting curves is simply a consequence of recrystallization (crystal perfection) during melting within the same crystal form (C). These observations are in good agreement with the suggestion stated above that only the temperature determines which crystal form is developed.

D. Glass transition, specific heat capacity and secondary transitions

As mentioned above, the glass transition temperature of amorphous Nylon M5T (therefore, also of the as-spun fiber) when measured by DSC at a heating

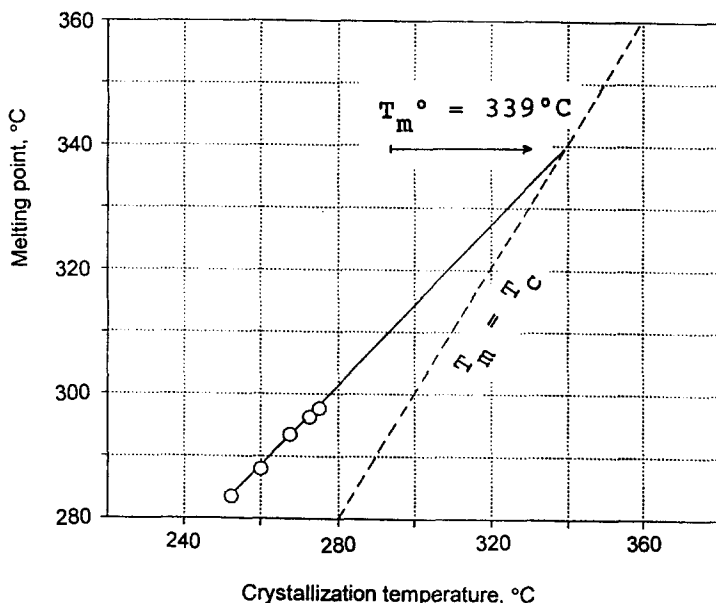


Fig. 12 Determination of the equilibrium melting point of Nylon M5T by the Hoffman-Weeks method (dependence of the recorded melting point on the isothermal crystallization temperature). The measurements were carried out at a heating rate of $10^\circ\text{C min}^{-1}$.

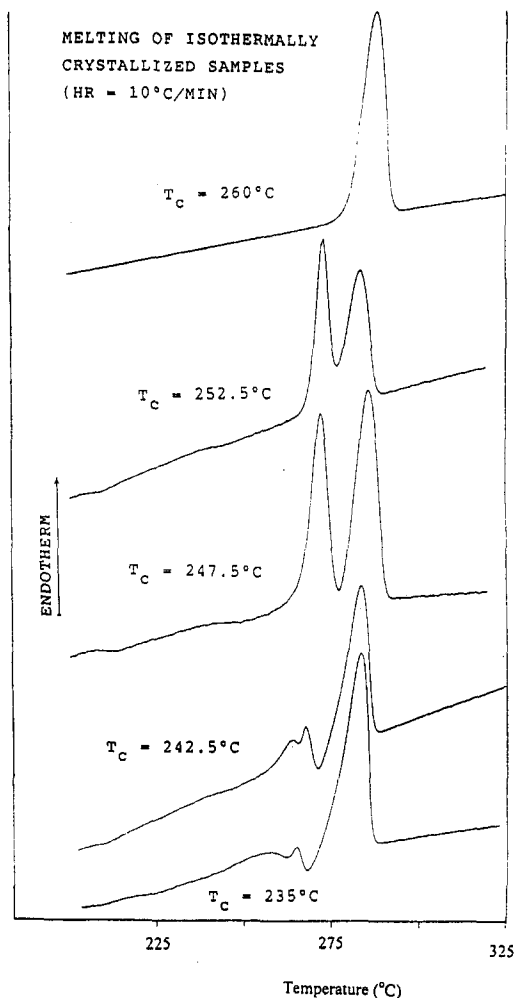


Fig. 13 The melting curves of Nylon M5T after isothermal crystallization at different temperatures (heating rate = $10^\circ\text{C min}^{-1}$)

rate of $10^\circ\text{C min}^{-1}$ is 143°C . The glass transition temperature (as usual) cannot be measured on a drawn fiber, probably due to the high content of the oriented or rigid amorphous phase in the fiber [7, 8]. As for some polymers, T_g increases with crystallinity. The maximum T_g recorded by DSC for this polymer (sample crystallized isothermally at 267.5°C) is 151°C (Fig. 15).

Specific heat capacity measurements on Nylon M5T revealed that, as expected, both the solid and the melt heat capacities are linear with temperature, and can be fitted by the following equations:

$$C_{\text{psolid}} [\text{J } ^\circ\text{C}^{-1} \text{g}^{-1}] = 0.00461 T [^\circ\text{C}] + 1.14 \quad (1)$$

$$C_{\text{pmelt}} [\text{J } ^\circ\text{C}^{-1} \text{g}^{-1}] = 0.00222 T [^\circ\text{C}] + 1.904 \quad (2)$$

This leads to a heat capacity increase at the glass transition of $103.9 \text{ J } ^\circ\text{C}^{-1} \text{ mol}^{-1}$, which is close to the value obtained by Wunderlich's rule [5]. The temperature dependence of the tensile storage and loss modulus, as well as the $\tan \delta$ for the as-spun and drawn ($\text{DR}=5.2\times$) Nylon M5T fibers recorded at a frequency of 1Hz is shown in Figs 5 and 16. As expected, the modulus of the drawn fiber is

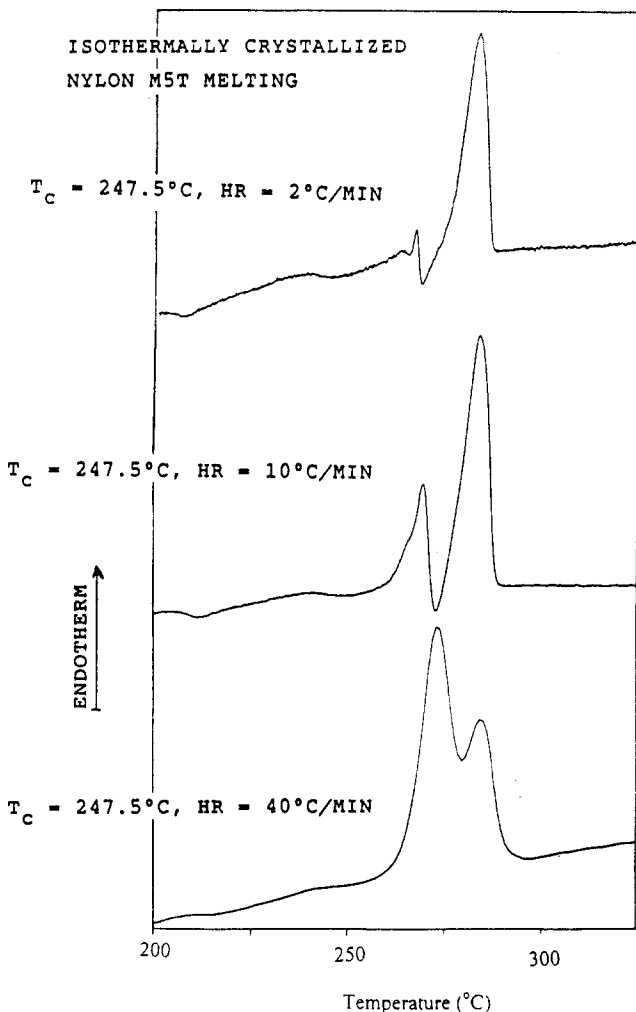


Fig. 14 The melting curves of Nylon M5T crystallized isothermally at 247.5°C , and reheated at heating rates of 2, 10 and $40^\circ\text{C min}^{-1}$

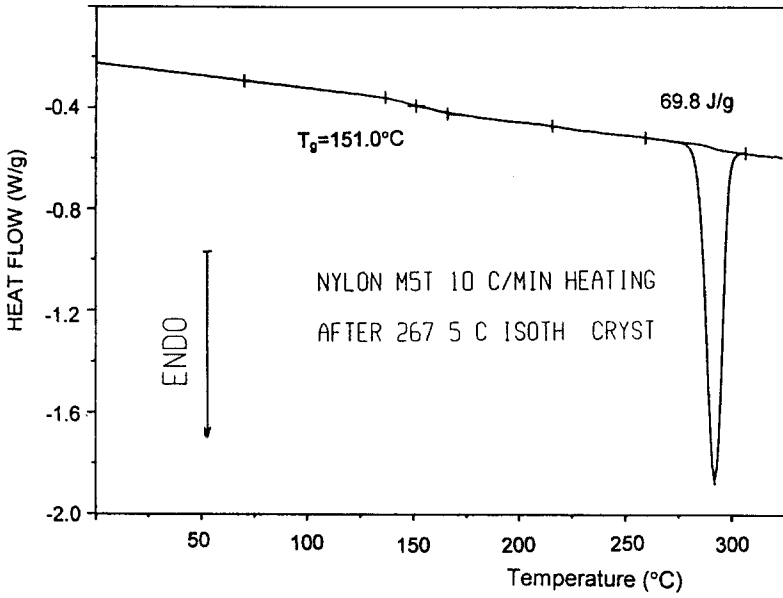


Fig. 15 The heating DSC-trace of Nylon M5T after isothermal crystallization at 267.5°C showing the highest achieved glass transition temperature

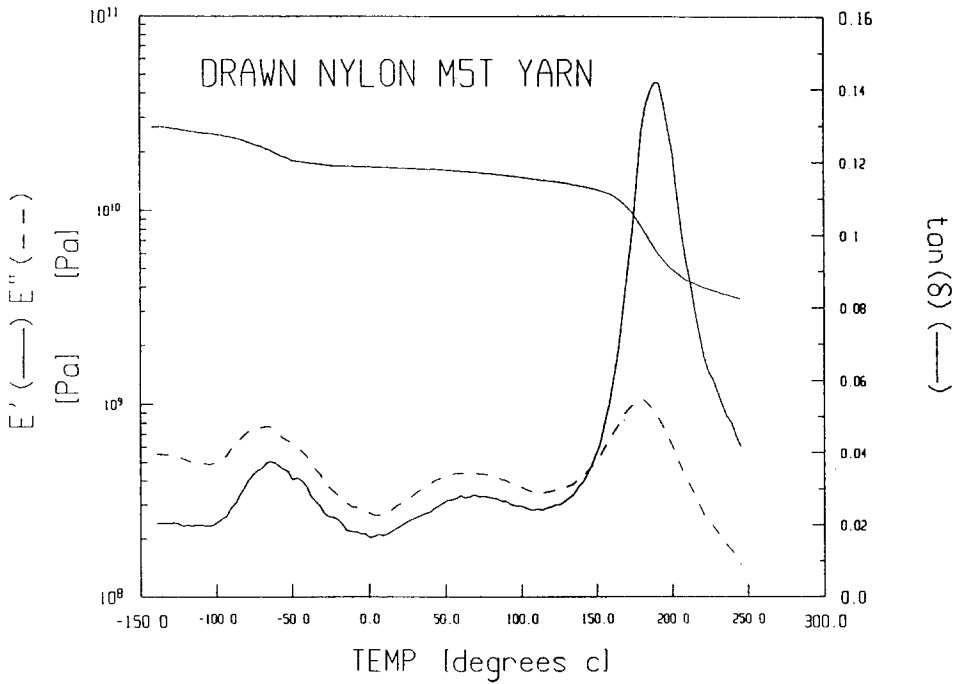


Fig. 16 The DMA trace of drawn Nylon M5T yarn

superior to that of the as-spun fiber, and the as-spun fiber is amorphous as shown by DSC measurements. The temperature dependence of the storage modulus for the as-spun and drawn fibers is consistent with the interpretation of T_g behavior given above.

When one examines the temperature dependence of $\tan \delta$ for the as-spun and drawn fibers, one obtains glass transition temperatures 155 and 190°C, respectively. In addition to the primary dispersion peak (T_α , glass transition), three other relaxation processes (secondary transitions) are recorded for both the drawn and the as-spun fibers (Fig. 17). The lowest temperature process (δ) takes place at -120°C (local mode relaxation of the $-\text{CH}_2-$ groups); the γ -transition takes place between -76°C and -46°C and the third (β) relaxation process occurs between 50 and 73°C depending on the applied frequency. Comparison with the DMA results on other polyamides [6] indicates that the γ -relaxation probably corresponds to onset of motion of the amide-groups, while the β -relaxation corresponds to onset of rotation of the phenylene-groups.

E. Thermal and thermo-oxidative stability

Bright field optical microscopy observations indicated that extremely fast discoloration of Nylon MST can be observed in air at 300°C: the sample exhib-

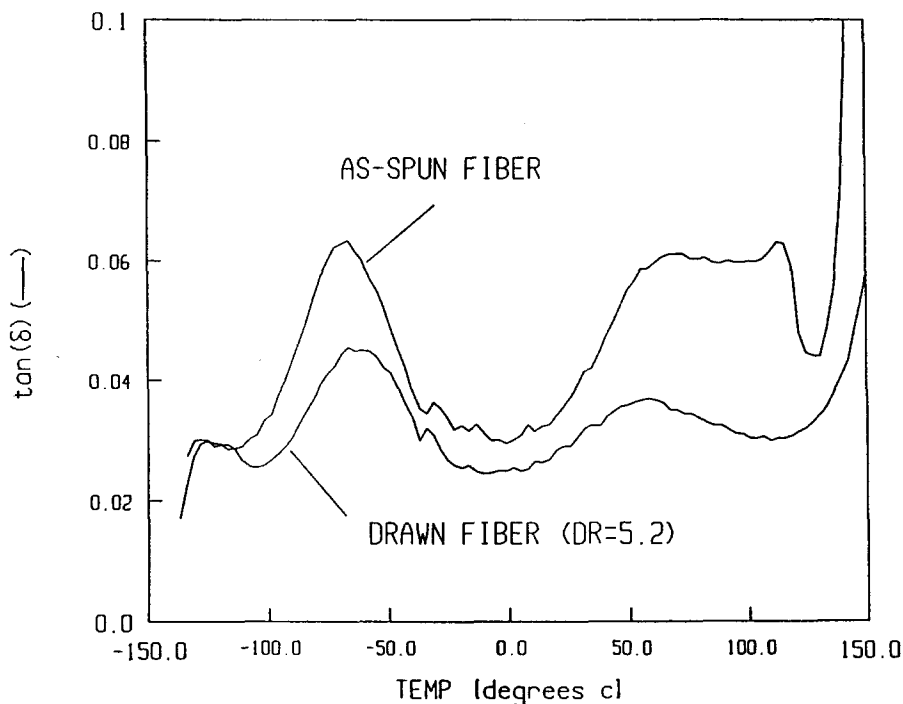


Fig. 17 The secondary transitions in as-spun and drawn Nylon MST yarns

its discoloration even at 0 min at 300°C, and within 15 min it turns completely brown, probably because of the ongoing thermo-oxidative degradation due to the oxygen sensitivity of the tertiary carbon atom in the main chain. No discoloration was observed in nitrogen. This observation indicates that this polymer degrades in air at its melting point, but is stable in an inert atmosphere to higher temperatures. This should be taken into account during the processing.

Table 3 The basic thermal parameters of Nylon M5T

1.	Temperature dependence of specific heat capacities: $C_{p\text{solid}} [\text{J } ^\circ\text{C}^{-1} \text{g}^{-1}] = 0.00461 T [^\circ\text{C}] + 1.14$ $C_{p\text{melt}} [\text{J } ^\circ\text{C}^{-1} \text{g}^{-1}] = 0.00222 T [^\circ\text{C}] + 1.904$
2.	Glass transition temperature of amorphous Nylon M5T: $T_g = 143^\circ\text{C}$ Heat capacity increase at the glass transition: $\Delta C_p = 103.9 \text{ J } ^\circ\text{C}^{-1} \text{mol}^{-1}$
3.	Secondary transitions: $T_\beta = 63^\circ\text{C}$, onset of rotation of the motion of the amide-groups $T_\gamma = -65^\circ\text{C}$, onset of rotation of the methylene groups $T_\delta = -120^\circ\text{C}$, local mode relaxation of the methylene groups
4.	Equilibrium melting point: $T_m^0 = 339^\circ\text{C}$

$C_{p\text{solid}}$ is the specific heat capacity of the crystalline and glassy polymer, $\text{J } ^\circ\text{C}^{-1} \text{mol}^{-1}$
 $C_{p\text{melt}}$ is the specific heat capacity of the polymer melt, $\text{J } ^\circ\text{C}^{-1} \text{mol}^{-1}$
 ΔC_p is the heat capacity increase at the glass transition, $\text{J } ^\circ\text{C}^{-1} \text{mol}^{-1}$
 T_m is the melting point, the last, highest temperature point of the melting endotherm, $^\circ\text{C}$

Conclusions

- The glass transition temperature of amorphous Nylon M5T is 143°C, increasing with crystallinity to 151°C
- In addition to the primary relaxation at 150–185°C (T_α), three secondary transitions were identified by DMA:
 $T_\beta = 63^\circ\text{C}$ (onset of motion of the aromatic groups)
 $T_\gamma = -61^\circ\text{C}$ (onset of motion of the amide-groups)
 $T_\delta = -120^\circ\text{C}$ (local mode relaxation of the methylene groups)
- Three crystal forms were identified by DSC, DMA and WAXS measurements
- The crystallinity and the obtained crystal forms strongly depend on the rate of cooling from the melt or isothermal or non-isothermal character of the crystallization

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