

# Crystallization of Nylon 11 Under Compressive High Strain Rates

J. O. FERNANDEZ, G. M. SWALLOWE, S. F. LEE

Department of Physics, Loughborough University, Loughborough, Leicestershire, LE11 3TU, United Kingdom

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**ABSTRACT:** The mechanical behavior of semicrystalline Nylon 11 was studied at strain rates between  $10^{-3}$  and  $8800 \text{ s}^{-1}$ . X-ray diffraction and DSC were employed to examine the crystal structure and the crystallinity content. The as-received material comprised a mixed structure of a predominately triclinic ( $\alpha$ ) form. DSC revealed that the material gave rise to two melting peaks. The compressive flow stress of Nylon 11 experienced a large increase at  $1200 \text{ s}^{-1}$  and decreased at higher strain rates. The maximum level of the flow stress corresponded with a higher level of crystallinity and a structure mainly of a pseudohexagonal form. The subsequent drop in stress at higher rates was associated with a decrease in the crystallinity content and a mixed crystal structure, different from that observed in the as-received material. After compression, the low melting peak disappeared and the material melted over an increased temperature range. © 2001 John Wiley & Sons, Inc. *J Appl Polym Sci* 80: 2031–2038, 2001

**Key words:** Nylon 11; high strain rate; crystallinity

## INTRODUCTION

Nylon 11 is an amide which is widely used for industrial applications. One of the characteristics of amides which has a major influence on their mechanical behavior is their propensity to absorb water. A high moisture content is characteristic of amides. The equilibrium water absorption of Nylon 11 is low for an amide, up to 2%, compared to almost 10% for other polymers of the same family, such as Nylon 66, which has a larger concentration of the amide group in the polymer backbone. The repeat unit of Nylon 11 comprises 10 methylene groups, one carbonyl group, and one amide. Nylon 11 has been extensively investigated because of its piezoelectric and pyroelectric proper-

ties. The crystal structure of Nylon 11 has not been clearly established since X-ray diffraction shows very few reflections. One triclinic  $\alpha$  form, one monoclinic  $\gamma$  form, and two pseudohexagonal forms, namely,  $\delta$  and  $\delta'$ , have been ascribed to Nylon 11. However, some workers believe that the distinction between the  $\gamma$ ,  $\delta$ , and  $\delta'$  forms is merely a matter of semantics rather than fact<sup>1</sup> and the term pseudohexagonal is often used to describe all three forms. Using X-ray diffraction fiber diagrams, Slichter<sup>2</sup> showed that all the reflections of Nylon 11 could be indexed on the basis of a structure similar to the triclinic  $\alpha$  form proposed by Bunn and Garner<sup>3</sup> for Nylon 66. Solution-cast Nylon 11 crystallizes into the triclinic form, according to Chen et al.<sup>4</sup> However, Schmidt and Stuart<sup>5</sup> reported that isothermal crystallization of Nylon 11 from the melt gave the  $\alpha$  modification, while Kim et al.<sup>6</sup> concluded on the basis of their results that the melt-crystallized films did not have the  $\alpha$ -form structure despite the general similarity of the diffraction patterns of melt and

Correspondence to: G. M. Swallowe (G.M.Swallowe@lboro.ac.uk).

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solution-crystallized Nylon 11. Chen et al.<sup>4</sup> reported that Nylon 11 crystallized at atmospheric pressure contained both the  $\alpha$  and one of the pseudohexagonal crystal  $\delta'$  forms, different from the variation  $\delta$  observed when annealed at high pressures. As mentioned above, Kim et al.<sup>6</sup> presented two different patterns: The solution crystallized nylon had the  $\alpha$  form with  $d$  spacings of 11.33, 4.44, and 3.72 Å, corresponding to the (001), (100), and (010) planes, respectively. On the other hand, the melt-crystallized nylon had  $d$ -spacings of 11.95, 4.40, and 3.82 Å, corresponding to the same families of planes. Wu et al.<sup>7</sup> quoted Hasegawa et al.<sup>8</sup> who proposed a model based on a triclinic  $\alpha$  form with antiparallel chain packing for samples quenched from the melt. The peak positions of uniaxially stretched Nylon 11 corresponded in accordance to their model to the (200) and (020) peaks, the second one being the most intense. However, the first set of Miller indexes seem to be more widely accepted. More recently, Moffatt et al.<sup>9</sup> carried out a series of experiments uniaxially deforming Nylon 11 at a low strain rate and at a temperature of 80°C. They found a transition from the  $\alpha$  to the  $\gamma$  form, commencing at a draw ratio of  $\sim 2$  with an increasing fraction of the  $\gamma$  phase as the draw ratio further increased.

Polyamides exhibit a complex behavior variation with temperature and Nylon 11 is not an exception. This is due to mainly two phenomena: On the one hand, moisture is lost endothermically above the glass transition. Moisture acts as a plasticizer because of the hydrogen bonding of water with the amide linkage and lowers the glass transition temperature from approximately 318 K for the dry material to 293 K for the moist material. On the other hand, Nylon 11 has a very high rate of crystallization, and cold crystallization starts just above the glass transition for amides of low crystallinity content. This has given rise to what Khanna and Kuhn<sup>10</sup> called silent crystallization. After cold crystallization, the differential scanning calorimetry (DSC) baseline does not return to the value before the transition and the material seems to continuously crystallize until the onset of melting. This problem is aggravated by the large temperature range between cold crystallization and melting for amides. In particular, this range is about 120 K for Nylon 11. Melting of Nylon 11 presents two melting peaks which correspond to two species of crystals according to Chen et al.<sup>4</sup> It is also possible that the two melting peaks correspond to one kind of

crystal differing in size or degree of perfection. Under different conditions of crystallization and temperature, one of the species may transform into the other and the material may only show one peak in a DSC thermogram. A crystal-crystal transition from the  $\alpha$  to the  $\gamma$  form when nylon is heated to 95°C has been reported by several workers.<sup>1</sup>

Surprisingly, there appears to be a lack of information in the area of the effect of a high-rate compressive deformation on crystallographic and thermal properties of Nylon 11 and this article is a report of studies on this topic. It has been widely reported that the yield stress and flow stress of many semicrystalline polymers increase slowly as a function of the strain rate until strain rates of  $\sim 10^2$  are achieved and then increase very rapidly thereafter.<sup>11-14</sup> Occasionally, decreases in yield stress at very high strain rates have also been reported.<sup>14</sup> A comprehensive set of experiments carried out at a range of very high strain rates on Nylatron, a MoS<sub>2</sub>-filled Nylon 66, showed a rapid increase in the flow stress at strain rates in the region of  $10^3$  to  $5 \times 10^3$ , with a rapid decrease in flow stress at higher strain rates. Estimates of crystallinity based on the heats of fusion of samples recovered after testing have mirrored this flow-stress variation with higher crystallinity corresponding to higher flow stresses. This has led to speculation that the crystallinity changes may be the cause of the flow-stress increases.<sup>15</sup> The present work extended this investigation of the relationship between crystallinity and flow stress to another member of the amide family.

## EXPERIMENTAL

The material used was purchased from Goodfellow Cambridge Ltd., Cambridge, UK. The samples were machined from a 10-mm-diameter rod into cylinders of approximately 4-mm diameter by 2-mm height. A selection of samples annealed for 15 min at 443 K and 30 min at 463 K were also tested in the same manner as was the as-received material. An estimate of the water absorption of as-received Nylon 11 is important for discussion of the material behavior. Accurately weighed thin slices of weights  $\sim 20$  mg were left in a vacuum desiccator for 4 weeks. The weights after that period showed mass losses of approximately 0.5%, indicating that the water content of the Nylon 11 used in this work was small.

**Table I** Variation of Nylon 11 Flow Stress (MPa) at 15% Strain Tested at 298 K with Strain Rate and Annealing Temperature

Rate (s <sup>-1</sup> )	As-received	15 min at 443 K	30 min at 463 K
0.002	37 ± 2	37 ± 2	41 ± 2
0.02	42 ± 2		
2.0	58 ± 2		
1200	115 ± 8	115 ± 8	120 ± 8
8800	108 ± 8	107 ± 8	118 ± 8

### Mechanical Testing

Samples were compressed in a direction parallel to the rod axis. Compressive tests were carried out using a conventional Hounsfield tensile/compressive test machine and two high-strain-rate machines, namely, a dropweight and a crossbow system, extensively described elsewhere.<sup>16,17</sup> Tests in the Hounsfield were performed at a strain rates of 0.002, 0.02, 0.2, and 2 s<sup>-1</sup>. The dropweight system produced strain rates in the range 500–3000 s<sup>-1</sup>, and the crossbow system, strain rates of ~9000 s<sup>-1</sup>. Samples were recovered after mechanical testing for X-ray and DSC examination. The flow stress at 15% strain as a function of the strain rate and annealing condition is shown in Table I. A minimum of three experiments were carried out for each test condition and the data in Table I show the average value and the spread of the results obtained.

### X-ray Diffraction

X-ray diffraction patterns were obtained in the reflection mode using a Philips PW1050/25 diffractometer running at 40 kV and 20 mA. A nickel filter was used to select the copper K $\alpha$  peak. The accuracy of the detection and analysis method employed to find the diffraction peak angles was tested using a silicon sample. The difference between the experimental results and the International Tables of Powder Diffraction values for silicon was less than 0.002 Å for each *d*-spacing. For the nylon work, three different samples were used to obtain an average value for the *d*-spacing for each test condition. Data were analyzed to determine peak positions using the commercial software packages Traces and PEAKFIT. Results obtained for *d*-spacings from both packages agreed to within 0.1 Å and the values from different samples subjected to the same treatment ana-

lyzed using with the same software package agreed in every case to within 0.05 Å.

### Thermal Analysis

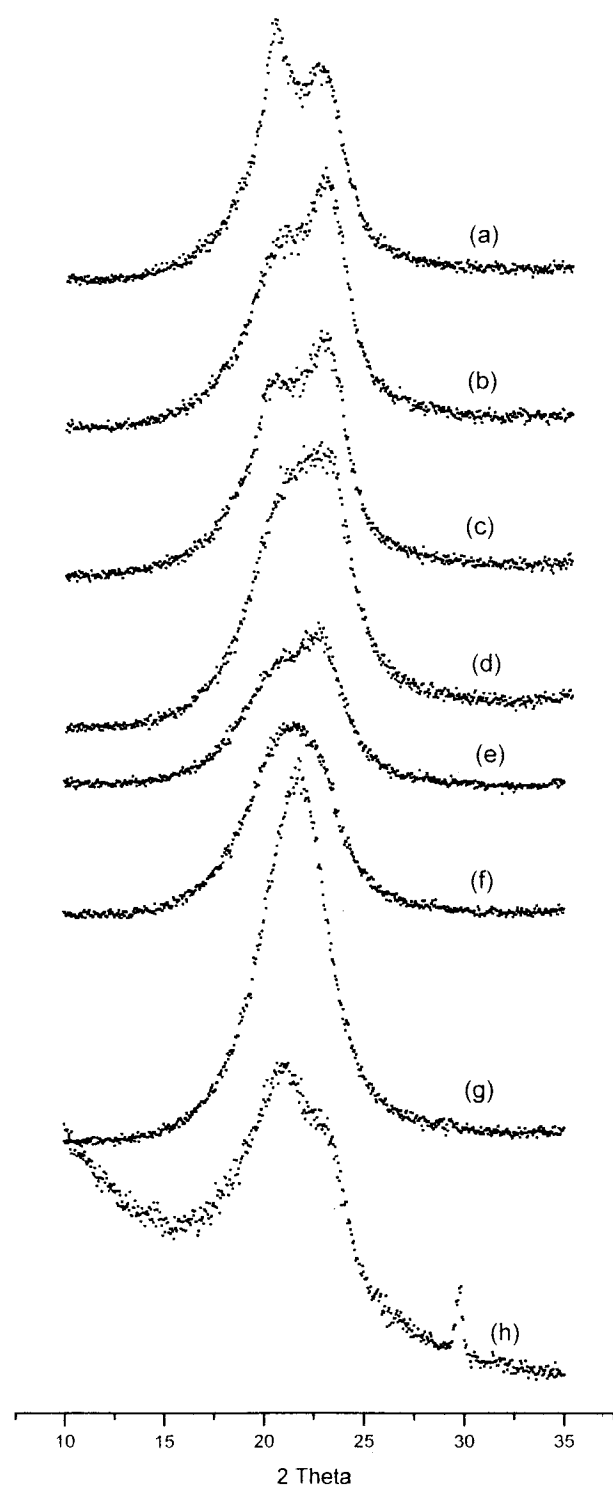
Thermal analysis was carried out in a Mettler DSC30 differential scanning calorimetry system. The system was subjected to heat flow and temperature calibration using standard materials. DSC was performed on approximately 10-mg samples in an argon atmosphere at a heating rate of 10 K min<sup>-1</sup>. The data values reported are the average of six scans. The degree of crystallinity was calculated as

$$\chi_c = \frac{\Delta H}{\Delta H_m^0}$$

where  $\Delta H$  is the heat of fusion and  $\Delta H_m^0$  is the heat of fusion of 100% crystalline Nylon 11 equal to 206 J g<sup>-1</sup>.<sup>18</sup>

### RESULTS

The X-ray diffraction pattern of Nylon 11 tested at room temperature at different strain rates can be seen in Figure 1. The as-received material was semicrystalline and of predominately the triclinic ( $\alpha$ ) form with two clear peaks given by planes of spacing 4.34 and 3.92 Å. The planes can be indexed as (100) and (010). On compressing the as-received material at all strain rates up to ~1000 s<sup>-1</sup>, the (100) reflection decreased in intensity but the two  $\alpha$  peaks can still be distinguished. At rates of 2 s<sup>-1</sup> and above, the  $\gamma$ -form peak became evident, and for rates between 1000 and 3000 s<sup>-1</sup>, there was only one reflection in the diffraction pattern and this corresponded to the  $\gamma$  form. At 8800 s<sup>-1</sup>, the intensity of the reflection decreased and two peaks similar to those of the low-rate tested samples were found. The annealed samples tested at room temperature showed a similar type of behavior. The *d*-spacing values obtained are tabulated in Table II. Figure 2 represents the data of Table II. The data for the annealed samples are shifted one and two units vertically, respectively, in order to present the data together. A feature of particular note is that for strain rates of greater than 2 s<sup>-1</sup> the triclinic peaks moved toward each other and disappeared, leaving a single peak characteristic of a pseudo-hexagonal structure at strain rates greater than



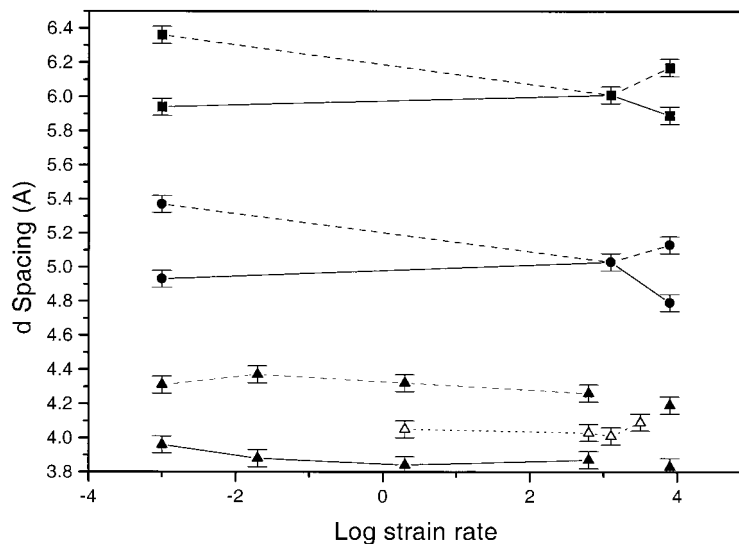
**Figure 1** X-ray diffraction patterns of Nylon 11 tested at 298 K: (a) as-received; (b)  $4 \times 10^{-3} \text{ s}^{-1}$ ; (c)  $0.2 \text{ s}^{-1}$ ; (d)  $2 \text{ s}^{-1}$ ; (e)  $600 \text{ s}^{-1}$ ; (f)  $1500 \text{ s}^{-1}$ ; (g)  $3000 \text{ s}^{-1}$ ; (h)  $8800 \text{ s}^{-1}$ . The small peak at  $\sim 29.7^\circ$  is due to the sample holder.

**Table II** Variation of Lattice Spacing ( $\text{\AA}$ ) with Strain Rate and Annealing Temperature

Rate ( $\text{s}^{-1}$ )	Treatment	(100) ( $\pm 0.05$ )	(010) ( $\pm 0.05$ )	(Ps) ( $\pm 0.05$ )
0	As-received	4.34	3.92	
0.001	As-received	4.31	3.96	
0.02	As-received	4.37	3.88	
2	As-received	4.32	3.84	4.05
600	As-received	4.26	3.87	4.03
1200	As-received			4.01
3000	As-received			4.09
8800	As-received	4.19	3.83	
0	15 min at 443 K	4.39	3.86	
0.001	15 min at 443 K	4.37	3.93	
1200	15 min at 443 K			4.03
8800	15 min at 443 K	4.13	3.79	
0	30 min at 463 K	4.43	3.88	
0.001	30 min at 463 K	4.36	3.94	
1200	30 min at 463 K			4.01
8800	30 min at 463 K	4.17	3.89	

$600 \text{ s}^{-1}$ , but reappear again at higher rates. Table II lists only the  $d$ -spacings of well-defined peaks. Indications of a pseudohexagonal peak were observed in many of the samples tested at all strain rates, but it did not emerge as a well-defined peak in the PEAKFIT analysis until testing was carried out at strain rates of  $2 \text{ s}^{-1}$  or higher. The (100) family underwent a more drastic change in the  $d$ -spacing and seems to be more sensitive to the strain rate than is the (010) family. The data of Table II show that on annealing the untested material experienced an overall increase in the (100) spacing while the (010) decreased, indicating that the volume remains almost constant, within errors. The data also show that the family of planes (100) after compression at  $8800 \text{ s}^{-1}$  did not recover to the value of the untested or low-rate tested sample. The average  $d$ -spacing was approximately  $4.19 \text{ \AA}$  compared to a pretesting average of  $4.34 \text{ \AA}$ .

Figure 3 depicts selected DSC scans of Nylon 11 samples tested at various strain rates at room temperature. All the samples underwent a glass transition at approximately  $316 \text{ K}$ . Thermograms of the annealed samples show that the shape of the melting peak varies depending upon the thermal treatment. As-received samples showed two melting peaks, the high-temperature peak being predominant. Under compression at any strain rate, the low-temperature melting peak disappeared. Likewise, samples annealed at  $443 \text{ K}$  for



**Figure 2**  $d$ -spacing (Å) versus strain rate for samples tested at 298 K. (Dashed lines) triclinic (100) family; (continuous lines) triclinic (010); (dotted line with open symbols) pseudo-hexagonal. (Top) Annealed 30 min at 463 K; (middle) annealed 15 min at 443 K; (bottom) as-received. Middle and top figures are offset one and two units, respectively.

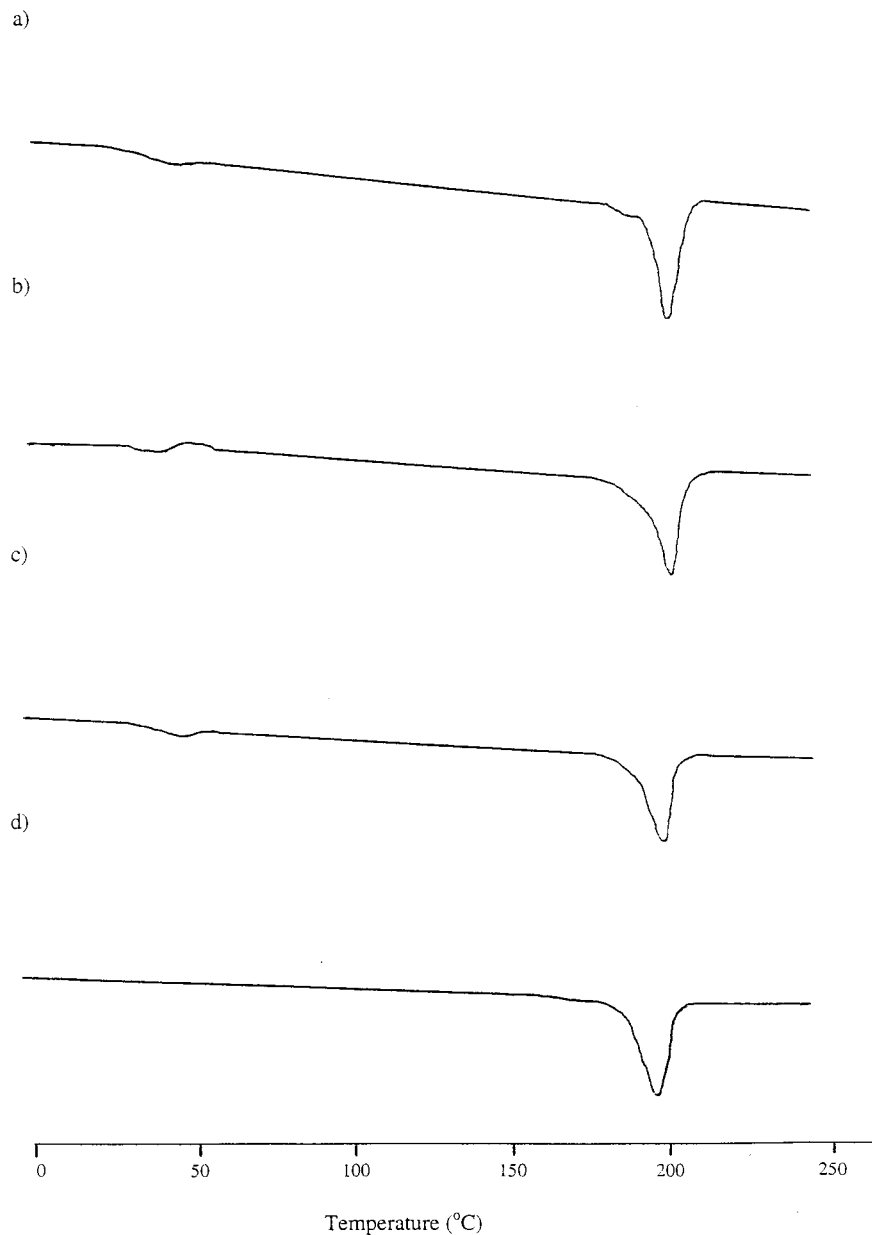
15 min showed two melting peaks, but on compressing, the material showed only one broad melting point. Samples annealed at 463 K for 30 min showed only one melting peak. The heat-of-fusion values were used to obtain the crystallinity values, by dividing by  $206 \text{ J g}^{-1}$ , and these are tabulated in Table III. Although the changes were very small, the crystallinity content consistently appears to increase with the strain rate up to  $1200 \text{ s}^{-1}$  and then decrease at the higher strain rate. This is similar to the behavior of Nylon 66 reported earlier.<sup>15</sup> The flow stress and crystallinity variation with the strain rate are illustrated in Figure 4. Crystallinity was also calculated for the as-received and 463 K annealed samples from the areas of the X-ray peak fits. This is an intrinsically less accurate method than is DSC since the amorphous background peak is not accurately known but the same pattern of crystallinity change as that observed in the DSC method is observed. A consistent increase of  $\sim 10\%$  between the unstrained material and that tested at a high strain rate is seen for all samples followed by a decrease at the highest testing rate. The crystallinity estimated from X-ray peaks is indicated in parentheses in Table III.

## DISCUSSION

Nylon samples seemed to undergo an enthalpic relaxation, that is, equilibrium was not reestab-

lished after the glass transition. After the glass transition, the thermogram baseline turned upward again, reaching similar values to pre-glass transition values, and then it continued normally until melting occurred. The relaxation did not show any consistent tendency with the temperature or strain rate, although it was less evident at high strain rates or with untested samples. On the other hand, samples tested at  $10^{-3} \text{ s}^{-1}$  underwent large relaxation phenomena and samples tested at  $1200 \text{ s}^{-1}$  showed the relaxation in as-received samples but not in annealed samples. Thus, a pattern of occurrence cannot be extracted from these data. It could be argued that the samples did not undergo cold or silent crystallization above the glass transition, but the upturn in DSC baseline is due to water which was lost when the temperature exceeded 323 K. Water should lower the glass transition temperature, but the glass transition temperature did not vary by more than  $\pm 3 \text{ K}$  around the value of 316 K. This is very close to the ultradry glass transition temperature and the water content is therefore unlikely to be a factor. This is supported by the low water content found in the as-received material. The origin of the enthalpic relaxation after the glass transition temperature seen in some samples is therefore not clear and it cannot be attributed unambiguously to silent crystallization or moisture.

Khanna and Kuhn<sup>10</sup> argued that for amorphous polymers (based on their findings on Nylon 6) a baseline should be drawn between the state of



**Figure 3** DSC thermograms of unannealed samples of Nylon 11 tested at 298 K: (a) as-received; (b)  $10^{-3} \text{ s}^{-1}$ ; (c)  $1200 \text{ s}^{-1}$ ; (d)  $8800 \text{ s}^{-1}$ .

equilibrium which occurs just above the glass transition temperature and the postmelting equilibrium temperature. They claim that for a crystallinity content larger than 25% the integration between a premelting and postmelting point temperature is correct as long as the moisture content of the sample is low. Owing to its high initial degree of crystallinity, the absence of cold crystallization and the low moisture content of the Nylon 11 used in this work, the thermograms were integrated between the premelting and postmelting points as suggested in ref. 10.

The material changed to an almost purely pseudo-hexagonal structure at strain rates in the range  $1200$  to  $3000 \text{ s}^{-1}$ . DSC showed an increase in the crystallinity level of the samples tested at  $1200 \text{ s}^{-1}$  with respect to low strain rate tests. The transition triclinic to pseudo-hexagonal is considered to be due to the effect of conformational motion and is associated with a packing change within the crystal allowing a greater amount of rotational freedom about the chain axis. Ramesh et al.<sup>19</sup> reported that the transition must be a first-order transition and this was supported by

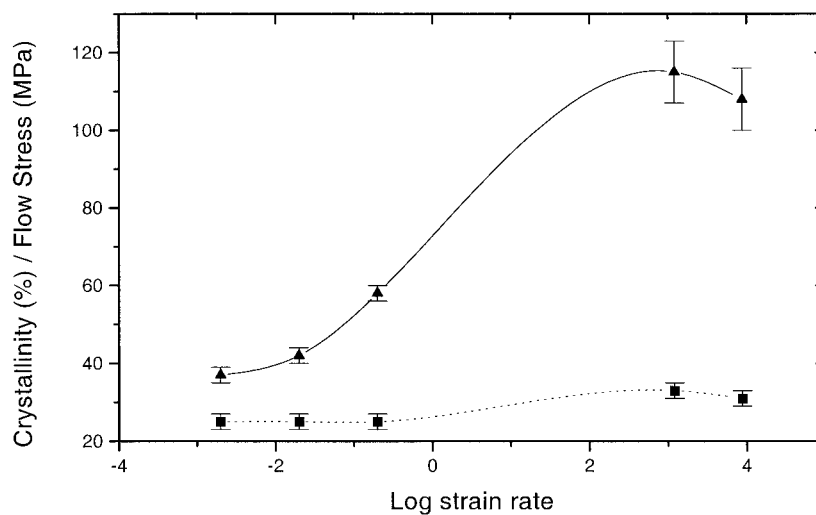
**Table III** Variation of Crystallinity Content (%) with Strain Rate and Annealing Temperature Determined by DSC and X-rays

Rate (s <sup>-1</sup> )	Crystallinity Content (% ±2)		
	As-received	15 min at 443 K	30 min at 463 K
0	25 (24)	30	32 (25)
0.002	25 (27)	33	33 (27)
0.2	25 (22)		
2	25 (25)		
1200	33 (37)	34	34 (34)
3000	31 (32)		
8800	32 (31)	31	32 (33)

The x-ray data are in parentheses and have an estimated error of 4%.

the hysteresis seen on annealing and cooling of the samples. Previous authors reported that the transition occurred on annealing. They found that for Nylon 66 samples crystallized between 469 and 533 K the structure was pseudohexagonal and partially transformed to the triclinic phase only on cooling to room temperature. On annealing, the reverse transition happens at temperatures above the crystallization temperature. However, samples tested in the dropweight machine showed a complete destruction of the  $\alpha$  form, retaining the pseudohexagonal structure after the test was completed. The sample strains at the end of the tests carried out in this work were in the

region of 20–100%. This observation is similar to the strain-induced  $\alpha$ - $\gamma$  transition observed by Moffatt et al.<sup>9</sup> However, unlike in Moffatt's work which was carried out at low strain rates and at a temperature of 80°C, the transformation in this work was observed in tests carried out at room temperature and only at a strain rate of  $\sim 10^3$  s<sup>-1</sup> and not at low rates. The explanation for this is probably due to the adiabatic nature of the high-rate tests. The  $\alpha$ - $\gamma$  transition temperature (95°C) can be achieved if the temperature increases by  $\sim 70^\circ\text{C}$  during deformation. An estimate of the temperature achieved during a high-rate test can be made by integrating the high-rate stress-strain curves and equating the plastic work to an increase in thermal energy in the material. Low-rate tests will be isothermal, whereas high-rate tests will be adiabatic, resulting in increased sample temperature. This procedure leads to estimated temperature increases of  $\sim 70^\circ\text{C}$  in the samples for tests conducted at 1000 s<sup>-1</sup>. It is therefore likely that for deformation at rates  $\sim 10^3$  s<sup>-1</sup> the adiabatic nature of high-rate deformation, rather than the compressive strain or the strain rate, causes the transformation. The low-rate tests will be essentially isothermal with only a modest temperature increase. Based on experiments where Nylon 11 samples were annealed at temperatures in the range 80–170°C followed by quench cooling but no  $\gamma$  phase was observed, Moffatt et al.<sup>9</sup> suggested that strain alone is responsible for the  $\alpha$ - $\gamma$  transition. However, this work suggests that both strain and an elevated temper-



**Figure 4** (▲) 15% strain-flow stress (MPa) and (■) crystallinity (%) versus log strain rate.

ature are required; the discrepancy between the results can be resolved when the fact that Moffatt et al.'s experiments were carried out at 80°C is considered. This is already close to the 95°C transition temperature, and the plastic work during drawing will have probably increased the sample temperatures by several more degrees.

Samples tested at higher strain rates, 8800 s<sup>-1</sup>, showed the triclinic form. It is not known whether the triclinic structure present in untested samples disappeared during the test and then crystallized again on cooling or if it was continuously present. Adiabatic heating during testing would have increased the sample temperatures to >95°C, so the  $\alpha$ - $\gamma$  transition observed at 1200 s<sup>-1</sup> would be expected to occur. The structure after these tests is slightly different from the initial form. A reduction in the crystallinity content compared to the 1200 s<sup>-1</sup> tests was detected both by DSC and X-ray. The difference in the X-ray diffraction peaks and the decrease in crystallinity could be associated with a disruption of the  $\alpha$ - $\gamma$  transformation process at such a high compressive strain rate.

The flow-stress values followed the same trend as that of the crystallinity content with the strain rate, that is, within errors, they increased up to strain rates of 1200 s<sup>-1</sup> and decreased afterward (see Table I and Fig. 4). However, there is no straightforward link between the two since the 15% flow stress occurs at an early stage in the deformation and the measured morphological changes are associated with the whole experiment. However, the maximum flow stress was observed when the sample had a higher degree of crystallinity and the structure was purely pseudohexagonal, whereas a decrease in the flow stress at higher rates was detected on samples with a mixed crystalline structure and a lower crystallinity content.

## CONCLUSIONS

The morphology of Nylon 11 was investigated after mechanical testing in the range 10<sup>-3</sup>-8800 s<sup>-1</sup>. The link between the postexperiment crystallinity and the flow stress previously observed with Nylon 66 was also observed for Nylon 11 although a causal link has not been established. The original mixed structure comprising crystals of triclinic and pseudohexagonal form was not detected after tests at strain rates  $\sim 10^3$  s<sup>-1</sup>, the strain rate of maximum flow stress, when only the

pseudohexagonal form was present. At higher strain rates, 8800 s<sup>-1</sup>, the flow stress was seen to decrease and the structure detected by X-ray diffraction contained crystals of a triclinic structure. The evidence suggests that the production of the  $\gamma$  form at high strain rates is due to the adiabatic nature of the process which increases the temperature close to the previously reported  $\alpha$ - $\gamma$  transition at 95°C. Low-rate (isothermal) tests showed that strain alone does not produce the transformation.

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## REFERENCES

- Aharoni, S. M. *n*-Nylons: Their Synthesis, Structure and Properties; Wiley: New York, 1997; see Chapter 2.11.
- Slichter, W. P. *J Polym Sci* 1959, 36, 259.
- Bunn, C. W.; Garner, E. V. *Proc R Soc Lond A* 1947, 189, 39.
- Chen, P. K.; Newman, B. A.; Scheinbem, J. I.; Pae, K. D. *J Mater Sci* 1985, 20, 1753.
- Schmidt, G. F.; Stuart, H. Z. *Z Naturforsch A* 1958, 13, 222.
- Kim, K. G.; Newman, B. A.; Scheinbeim, J. I. *J Polym Sci B* 1985, 23, 2477.
- Wu, S. L.; Scheinbeim, J. I.; Newman, B. A. *J Polym Sci B* 1996, 34, 3035.
- Hasegawa, R. K.; Kimoto, K.; Chatani, Y.; Tadoroko, H.; Sekiguchi, H. In *Discussion Meeting of the Society of Polymer Science, Tokyo, Japan, 1974*; Preprint 713.
- Moffatt, S.; Aji, A.; Lotz, B.; Brisson, J. *Can J Chem* 1998, 76, 1491.
- Khanna, Y. P.; Kuhn, W. P. *J Polym Sci B* 1997, 35, 2219.
- Chou, S. C.; Robertson, K. D.; Rainey, J. H. *Exp Mech* 1973, 13, 422.
- Briscoe, B. J.; Hutchings, I. M. *Polymer* 1976, 17, 1099.
- Rao, S.; Shim, V. P. W.; Quah, S. E. *J Appl Polym Sci* 1997, 66, 619.
- Walley, S. M.; Field, J. E. *Dymat J* 1994, 1, 211.
- Al-Maliky, N.; Fernandez, J. O.; Parry, D. J.; Swallowe, G. M. *J Mater Sci Lett* 1998, 17, 1141.
- Hamdan, S.; Swallowe, G. M. *J Mater Sci* 1996, 21, 1415.
- Hamdan, S.; Swallowe, G. M. *Meas Sci Technol* 1996, 7, 1068.
- Mark, H. F.; Gaylord, N. G.; Bikales, N. M. (Eds.), *Encyclopaedia of Polymer Science and Technology*; Wiley: New York, 1966; Vol. 4, p 488.
- Ramesh, C.; Keller, A.; Eltink, S. J. E. A. *Polymer* 1994, 35, 2483.