

Crystal Transformation of Nylon 11 Using *in situ* WAXD

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Abstract: α Form Nylon 11 films were found to exist a non-linear transformation at 70°C during the heating process using *in situ* WAXD. The α Form disappeared but δ form appeared when the temperature was higher 70°C.

Keywords: Nylon 11, crystal transition, WAXD.

Nylon 11 is a high-performance semicrystalline polymer which was used widely in many fields. In recent years, the curious ferroelectric and piezoelectric properties of nylon 11 were found^{1,2}. It is well known that piezoelectricity is related to the crystal form of Nylon 11.

The sample was mounted in a capillary (diameter was 2mm) within a small oven with mica windows. The scattered intensity was recorded from $Q = 0.2$ to 6.5 \AA^{-1} ($Q = 2\pi\sin\theta/\lambda$) using steps of $\Delta Q=0.05\text{\AA}^{-1}$. The values of the peak width and peak position were shown in **Figure 1** and **Figure 2** respectively.

At 30°C, the diffraction patterns of the α form Nylon 11(triclinic system) showed three diffraction peaks, peak 1 (001), peak 2 (100) and peak 3 (010,110). The δ form Nylon 11(pseudohexagonal) would appear when the temperature was above 70°C. The position of peak 1 was stable at $Q = 0.52 \text{ \AA}^{-1}$ even when the temperature was up to 175°C, but its peak position suddenly rose to 0.7 \AA^{-1} . The position of peak 2 increased from 1.43 \AA^{-1} to 1.46 \AA^{-1} , but the peak position of peak 3 decreased from 1.62 \AA^{-1} to 1.57\AA^{-1} simultaneously, and they disappeared when the temperature was up to 70°C. Peak 4 appeared when the temperature was above 70°C and its peak position decreased slowly from 1.52 \AA^{-1} to 1.50 \AA^{-1} . Peak 4 appeared at 1.5\AA^{-1} when temperature was above 70°C, which narrowed significantly as the temperature increase. The positions of crystal peaks were all plotted and shown in **Figure 2**.

We observed that there was a non-linear transformation at 70°C for the peak position and the peak width of the crystal peaks also showed the similar interesting non-linear transformation behavior. The peak width (\AA^{-1}) of Peak 1 reduced from 0.068 \AA^{-1} ($t=30^\circ\text{C}$) to 0.056 \AA^{-1} ($t=170^\circ\text{C}$). The peak 2 and peak 3 only exist below 70°C. The peak width of peak 2 increased rapidly from 0.58 \AA^{-1} to 0.72 \AA^{-1} but the peak width of

peak 3 decreased slowly from 0.78 \AA^{-1} to 0.75 \AA^{-1} . The peak 4 appeared when temperature was above 70°C , and its peak width decreased rapidly from 0.12 \AA^{-1} to 0.05 \AA^{-1} .

Nylon 11 was a typical hydrogen-bonded polymer. Hydrogen bond is the very important function during the crystal transition. When the α form was heated, the C=O and N-H units on the molecular chains would move intensely and the α form unit cell was broken up. At high temperature, the metastable pseudohexagonal δ form was produced by hydrogen bonds.

Figure 1 The peak position of crystal peaks

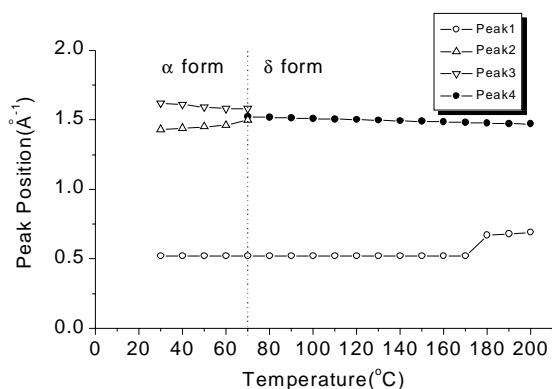
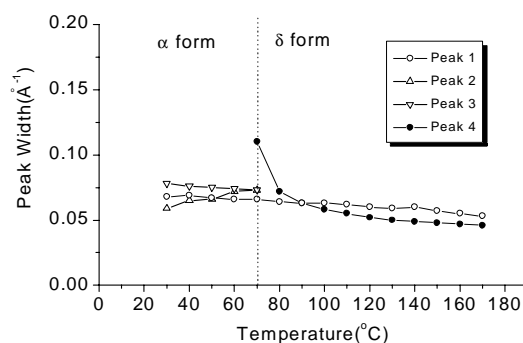


Figure 2 The peak width of crystal peaks



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References

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