Oriented Crystallization in Electrospun Composite Fibers Revealed by Simultaneous SAXS and WAXS Synchrotron Radiation

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In this report, simultaneous SAXS and WAXS were used to investigate the crystal transformation and lamellar variation of the electrospun Nylon 6 nano-fibers with a diameter of 200 nm during step-wise heating. The results were compared with those obtained from the solution cast Nylon 6 film with a thickness of ~10 μm.

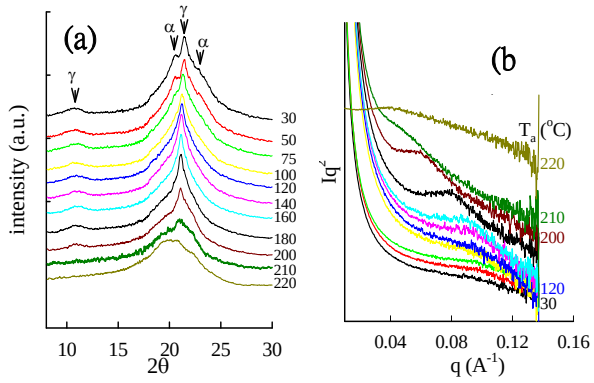

Fig. 1: (a) WAXD intensity profiles of electrospun Nylin 6 fibers at selected annealing temperature T_a from 30 to 220°C, (b) SAXS intensity profiles (Lorentz-correccted plot) of the electrospun fibers at the correnting T_a.

As shown in Fig. 1, the as-spun Nylon 6 fibers possess the mixed crystal modifications of α-form (2θ= 20.61, 23.36°) and γ-form (2θ= 10.73, 21.41°) in the absence of regular lamellar spacing. No siginificant variation of the WAXD and SAXS intensity profiles are seen for an annealing temperature (T_a) of 50°C. At T_a= 75~140°C, the amount of α-form is slightly reduced and a barely-seen SAXS hump is detected (q_{max}= 0.0946Å^{-1}), suggesting the gradual development of ordered lamellae during heating. In the tempearure range of 140~180°C, no crystal melting takes place, but the SAXS peak becomes pronounced and slightly shifts to the low q region (q_{max}= 0.0783Å^{-1}), indicating the increase of the lamellar thickness. For T_a above 200°C, some γ-form crystals start to melt, and the position of SAXS peak shifts pronouncedly towards to a low q region, indicating the rapid increase of the lamellar spacing. The long period is ca. 126Å^{-1} at T_a=210°C. The diffraction peak at 2θ= 10.73°, which is the fingerprint of strong orientation of γ-form, finally disappears at 210°C. At T_a= 220°C, featureless structure is detected from the WAXD and SAXS scattering.

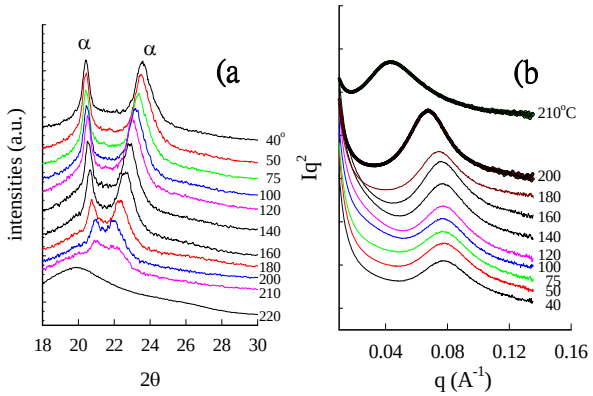

Fig. 2: (a) WAXD intensity profiles of solution-cast Nylon 6 films at selected annealing temperature T_a from 40 to 220°C, (b) SAXS intensity profiles (Lorentz-correccted plot) of the solution-cast film at the correnting T_a.

Solution-cast films possess the α-form crystals with a SAXS peak at 0.0776Å^{-1} at 40°C. On heating, the two WAXS diffraction peaks (2θ= 20.42, 23.60°) move closer to each other with a gradual reduction of the peak intensity. At 210°C, the two WAXD diffraction peaks are located at 20.89 and 21.81°. At 220°C, all the crystals are melted away and only the amorphous halo (2θ= 19.84°) is seen. According to the T_a-depencence of SAXS intensity profiles, it is evident that q_{max} remains intact for T_a<140°C. For T_a>160°C, q_{max} gradually moves to the low q region and simultanesouly the intensity of SAXS peak is also increased until T_a=200°C. At 210°C, a large long period of 164Å is reached but the intensity is reduced due to the occurrence of major crystal melting, as-revealed by WAXD intensity profile. Based on Fig. 2, it is concluded that no $\alpha \rightarrow \gamma$ crystal transformation takes place, but a gradual melting of the α-form crysatls together with the lamelaar thickening occurs during step-wise annealing of the solution-cast Nylon 6 film.