ESRF	**Experiment title:** **Melt electrospinning of isotactic polypropylene: Influence of applied voltage on structure formation**	**Experiment number:** SC 2310
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Report:

Nanofibers are promising materials for many technological applications such as textiles, filtration and bioengineering, just to mention a few. Electrospinning is considered one of the simplest and most effective methods for fabrication of fibers with diameters ranging from 3 nm to 500 µm [1-5]. The industrial interest in solution electrospinning are relatively scarce because of the fact that the most significant limiting factors to industrial production nanofibers are the solvent recycling issues and low productivity. Melt electrospinning eliminates both of these problems. It also allows to make fibers that are free of potentially harmful solvents, which is important for most biomedical applications. Furthermore, melt electrospinning is the only choice for polymers that do not have proper solvents at room temperature like polypropylene, or where toxic and expensive solvents are needed. The main difference to the solution electrospinning is the way of fiber forming. In solution electrospinning the fibers are formed by evaporation of the solvent, while in the case of melt electrospinning, polymer melt is cooled down to form fibers as described in [6].

Figure 1 shows 1D WAXD curves corresponding to the iPP fibers electrospun at three voltages (15, 20, and 25 kV). The black curves correspond to the alpha and smectic phases of iPP. The fiber crystallinity depends on the applied voltage during fiber production. Thus, an increase of voltage during electrospinning results in a decrease of crystallinity and increase of the smectic

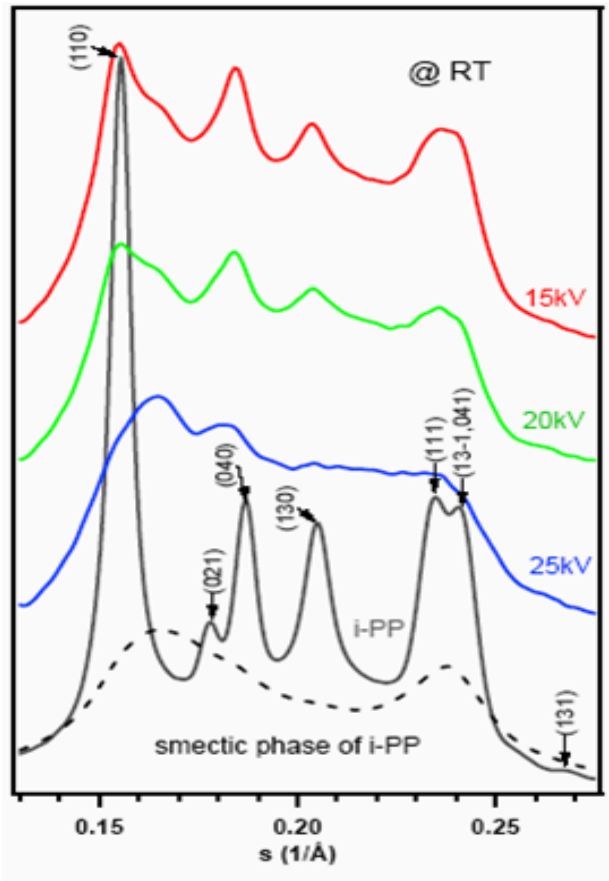

Figure 1: WAXS curves corresponding to bulk iPP and iPP electrospun fibers formed at 15kV, 20kV, and 25kV. The solid and dashed black curves correspond to the α-phase and smectic phase of iPP.

phase fraction. The change in the WAXD curve during heating at a rate of 10°C/min was monitored from 55°C to 180°C (cf. Figure 2A). In this temperature range, an increase in crystallinity was observed. The smectic phase of iPP recrystallizes during the heating process before the melting process sets in.

The onset of the smectic phase recrystallization for all three samples is at about 90°C, whereas the maximum of crystallinity was observed at 145°C. At temperatures above 145°C the samples enter the main melting region. All three samples showed similar recrystallization and melting behaviour. Independently of the electrospinning conditions, similar maximum crystallinities were observed. Even though directly after melt electrospinning the ratio of the smectic phase to alpha phase differs, similar crystallinities were achieved upon heating to 145°C. Figure 2B shows two WAXD patterns of the melt electrospun polypropylene fiber, produced at 25 kV and monitored at 55°C and at 145°C. As this fiber exhibits the lowest crystallinity at room temperature, the recrystallization process in this case was mostly pronounced. The intensity of 040-reflection in the temperature range from 55°C to 200°C is shown in Figure 2C. The onset of crystallization can be identified to be at about 90°C, reaching a maximum at 145°C which is followed by beginning of melting. Figure 2C shows the temperature derivative of the 040 peak intensity (–dI/dT), which allows identifying the dynamics of the temperature evolution of crystallinity.

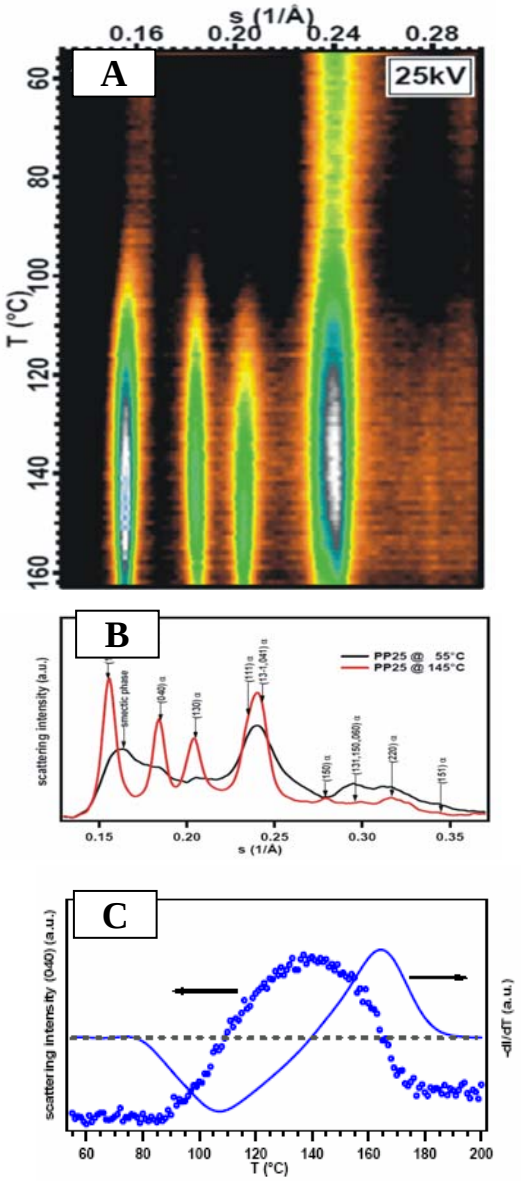

Figure 2: A) Variable temperature WAXS corresponding to iPP fibers electrospun at 25kV. B) The same as in A measured at 55°C and 145°C. C) Intensity of the 040 diffraction peak and its temperature derivative dI/dT measured between 55°C to 200°C (The onset of crystallization is at 90°C and the maximum crystallinity is at 145°C).

Conclusion:

Electrospun fibers of isotactic polypropylene contain a mixture of the crystalline α-phase and smectic phase. Distinct differences in crystallinity and the ratio of α-phase to smectic phase were observed, depending on the processing parameters. The increase of the voltage during electrospinning resulted in a decrease of crystallinity and an increase of the smectic phase fraction. The observed onset of recrystallization of the smectic phase for all three samples was at about 90°C, while the maximum crystallinity was reached at 145°C. Similar maximum crystallinities were obtained for all electrospun fibers, independently of the processing parameters and fiber diameter. Recrystallization was most pronounced for the polypropylene fibers produced at the highest voltage of 25kV, as they showed the lowest crystallinity at room temperature.

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