



Experiment title:

Kinetics of melting of the disordered α' -phase of poly (L-lactic acid) and of recrystallization of the melt to α -crystals

Experiment number:

MA2596

Beamline: BM26	Date of experiment: from: 27/07/2015 to: 29/07/2015	Date of report: 28/08/2015
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Report:

Samples of PLLA containing α' -crystals (obtained by crystallization at low temperature) have been heated at different rate in order to confirm their melting at about 150 °C if the heating rate is higher about 50 K/s, or their reorganization into α -crystals if the heating rate is lower. Detection of α' - and α -crystals of PLLA

advantageously is done by analysis of the X-ray pattern since both phases cause scattering at different angles (see Figure 1).

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Detection of α' - and α -crystals of PLLA advantageously is done by analysis of the X-ray pattern since both phases cause scattering at different angles (see Figure 1).

Figure 1: WAXS pattern of PLLA crystallized at 85 (red curve) and 145 °C (blue curve), containing α' - and α -crystals, for illustration of different peak positions. Data were collected using CuK α radiation (Eur Polym J 70 (2015) 215–220).

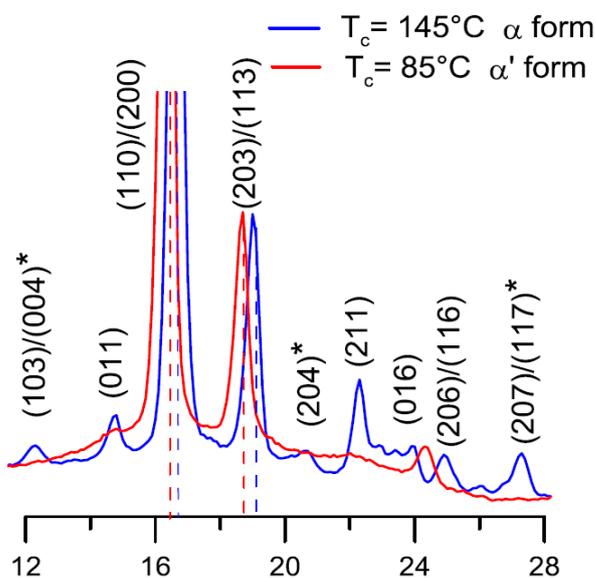


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A typical temperature-resolved WAXS experiment performed at ESRF is shown in Figure 2. It shows with the front curve, obtained at 23 °C, presence of α' -crystals, to be recognized with the 110/200 peak around 11° 2 θ . On heating at high rate of 85 K/s (effective at 140 °C) the peak disappears around 140 °C, indicating melting of the α' -crystals without prior reorganization into α -crystals. Similar sets of WAXS curves have been obtained on heating at lower rate, however, with the temperature of melting increasing. Though this observation, that is, increasing melting temperature with decreasing heating rate, indicates reorganization, it was impossible to clearly detect the transformation of α' - into α -crystals. Main reason is the strong noise of data, in combination with the low wavelength used in the experiments. The latter leads to a lower distance between the 110/200 peaks of the α' - and α -phase, complicating their identification, while peaks of lower intensity are not detected due to the low signal-to-noise ratio. Careful inspection of the sets of curves collected during slow heating, showed a broadening of the 110/200 peak indicating the phase transition, however, with great potential for further improvement on changing the instrumental setup.

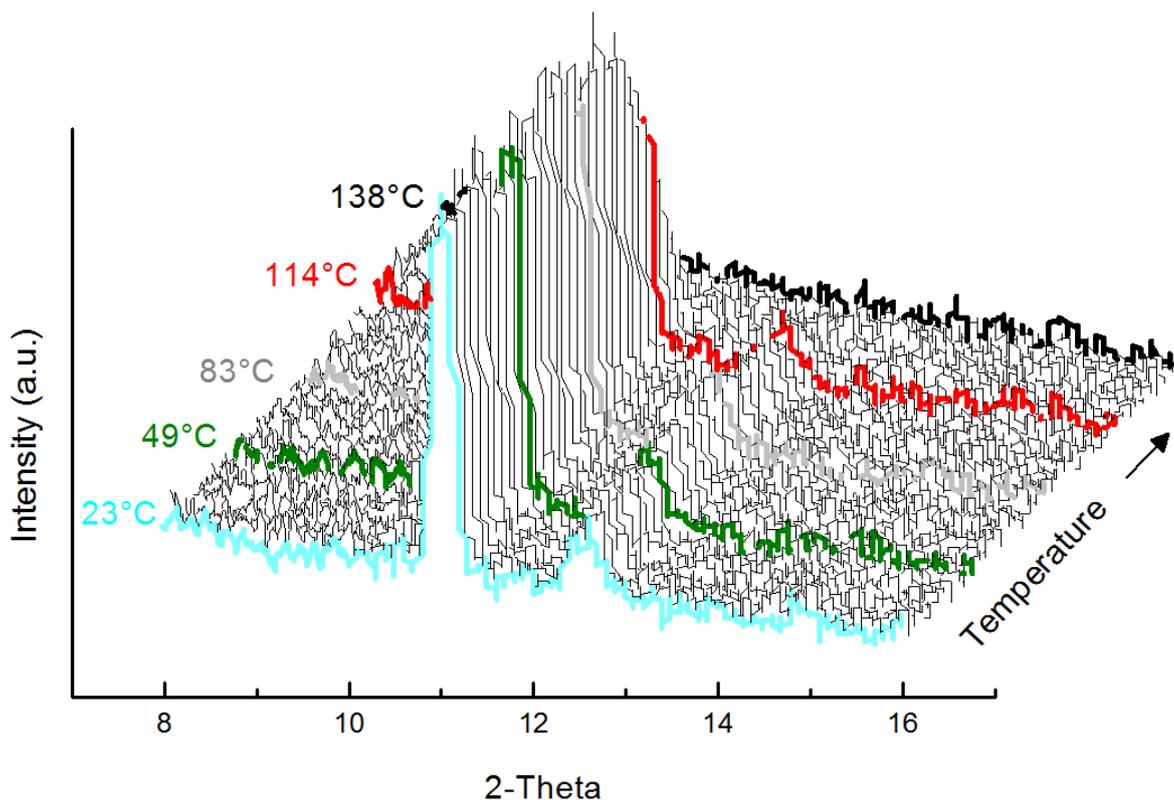


Figure 2: Series of WAXS pattern of PLLA crystallized at 85 °C, collected during heating at a rate of 85 K/s (effective around 140 °C).