

## NUCLEATION OF HIGH PRESSURE GAMMA FORM IN ISOTACTIC POLYPROPYLENE

Przemyslaw Sowinski<sup>a</sup>, Ewa Piorkowska<sup>a</sup>, Severine A.E. Boyer<sup>b</sup>,  
Jean-Marc Haudin<sup>c</sup>, Kinga Zapala<sup>a</sup>

<sup>a</sup>Centre of Molecular and Macromolecular Studies, Polish Academy of Sciences, Sienkiewicza 112, 90 363 Lodz, Poland, przem\_so@cbmm.lodz.pl, epiorkow@cbmm.lodz.pl

<sup>b</sup> P PRIME Institute, ISAE-ENSMA, UPR CNRS 3346, Department of Physics and Mechanics of Materials, 1 Avenue Clément Ader, 86961 Futuroscope, severine.boyer@ensma.fr

<sup>c</sup> MINES ParisTech, Centre for Material Forming, UMR CNRS 7635, 1 Rue Claude Daunesse, 06904 Sophia Antipolis, France, jean-marc.haudin@mines-paristech.fr

### ABSTRACT

Isotactic polypropylene (iPP) crystallizes in the forms: monoclinic alpha, hexagonal (trigonal) beta, orthorhombic gamma or in the smectic form. Under atmospheric pressure iPP crystallizes usually in the alpha or beta phase; crystallization in the beta phase requires appropriate nucleating agents.<sup>1</sup> Crystallization of the gamma form is facilitated by high pressure. Owing to its unique structure with nonparallel chain alignment, the gamma form exhibits different mechanical properties than the alpha form.<sup>2</sup> Recently, Zapala et al.<sup>3</sup> demonstrated, by means of the droplet experiment, that heterogeneous nucleation is beneficial for crystallization of iPP in the gamma form under high pressure.

Our study focused on nucleation of the gamma form in iPP under high pressure. IPP utilized in the study was 3250MR1 (Arkema) with  $M_w = 213$  kg/mol,  $M_w/M_n = 5$ , MFI = 25 g/10 min. Three nucleating agents were used: Hyperform HPN-20E (Milliken Chemicals) and poly(tetrafluoroethylene) submicron particles, Dispersez 200W2 (Polysciences Inc.) which nucleate efficiently crystallization of iPP in the alpha form under atmospheric pressure,<sup>4</sup> and calcium pimelate, which is known to nucleate the beta form of iPP under atmospheric pressure,<sup>5</sup> (synthesized at CMMS, Lodz). The high pressure crystallization of neat iPP and iPP compositions with 0.2 wt% of the nucleating agents was carried out in a cell that consisted of a barrel and a piston<sup>2,3</sup> and a loading frame of the tensile testing machine (Instron), under pressure ranging from 100 to 300 MPa, nonisothermally during cooling and isothermally at 200°C. The crystallized specimens were analysed by differential scanning calorimetry (DSC), wide angle X-ray scattering (WAXS), and polarized light microscopy PLM. Crystallinity level, contents of crystallographic forms, number and size of polycrystalline aggregates in each specimen were estimated.

Both alpha-nucleating agents accelerated crystallization of iPP under high pressure in the gamma form whereas the beta-nucleating agent did not. The alpha-nucleating agents possibly acted by nucleating the alpha "seeds" facilitating further crystallization of the gamma crystals. Especially, poly(tetrafluoroethylene) is known to nucleate the iPP alpha phase through epitaxy involving (110) alpha plane, which does not apply for the gamma form<sup>6</sup>. The results show the possibility to nucleate the gamma form of iPP under high pressure.

**Acknowledgement:** Statutory funds of CMMS PAN, PAN/CNRS PICS Project n°4702 'Crystallization under shear and interface behaviors of polymers and polymer nanocomposites' and PAN/CNRS Collaborative Project n°179990 'High pressure crystallization and structure of polymers based nanocomposites'.

### References:

- <sup>1</sup> Varga, J. in *Polypropylene: structure, blends and composites*, vol 1, Karger-Kocsis J. Ed, Chapman & Hall, London, 1993, pp 56–115.
- <sup>2</sup> Lezak, E., Bartczak, Z., Galeski, A. *Macromolecules* **2006**, 39, 4811.
- <sup>3</sup> Zapala, K., Piorkowska, E., Hiltner, A., Baer, E. *Coll. Polym. Sci.* **2012**, 290, 1599.
- <sup>4</sup> Masirek, R., Piorkowska, E. *Eur. Polym. J.* **2010**, 46, 1436.
- <sup>5</sup> Lezak, E., Bartczak, Z., Galeski, A. *Polymer* **2006**, 47, 8562.
- <sup>6</sup> Mathieu, C., Thierry, A., Wittmann J.C., Lotz, B. *Polymer* **2000**, 41, 7241.