

Nucleating agents towards crystallization under realistic conditions

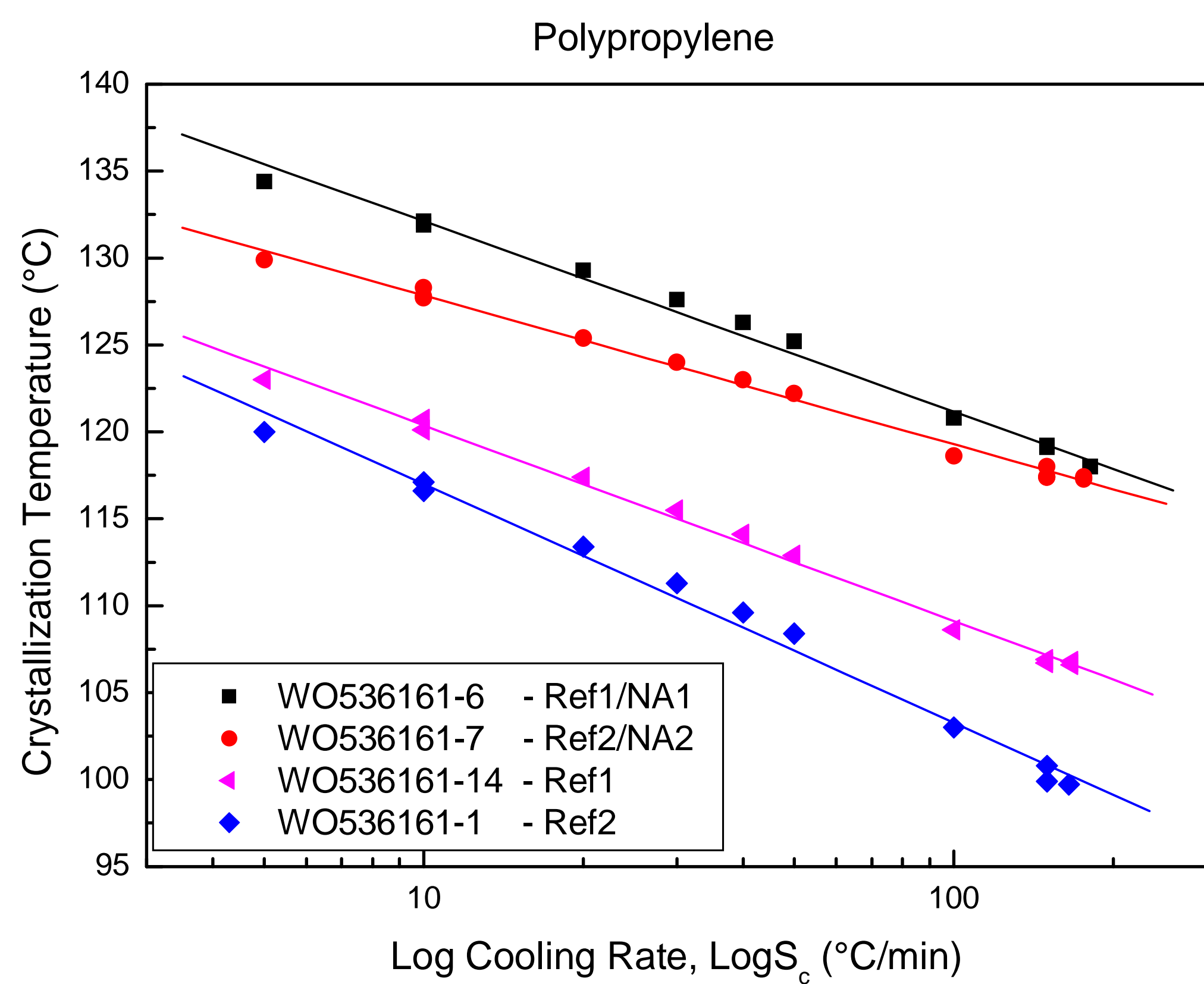
Albert Sargsyan^a, Geert Vanden Poel^a, Klaas Remerie^b, Evgeny Zhuralev^c, Christoph Schick^c
 a) DSM Resolve, b) SABIC, c) University of Rostock

Sharing knowledge...

1. Introduction

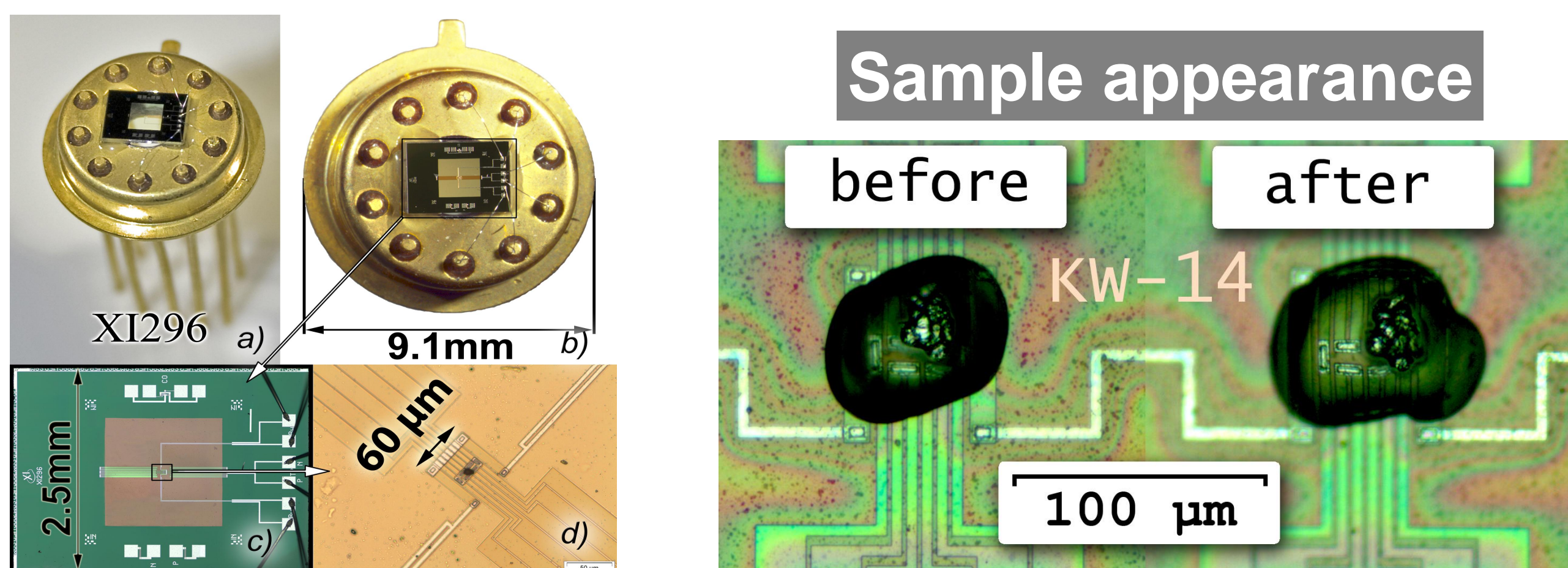
The crystallization behavior and morphology of isotactic polypropylene (iPP) is strongly dependent on the crystallization conditions; i.e. cooling rate, isothermal temperature, additives like nucleating agents. The influence of the cooling speed – rapid cooling by ultra fast scanning nanocalorimetry – on the crystallization behavior of iPP has been intensively studied by Prof. C. Schick et al. [1-3]. Fast scanning calorimetry makes it possible to cool in a controlled way. The critical rate for mesophase formation has been confirmed to be 100 K/s [4, 5]. Cooling rates of 0.1 to 160 K/s leads to the formation of the monoclinic α -crystal. At cooling rates of 1000 K/s and higher the mesophase formation can be suppressed, which leads to fully amorphous iPP.

2. HPer DSC

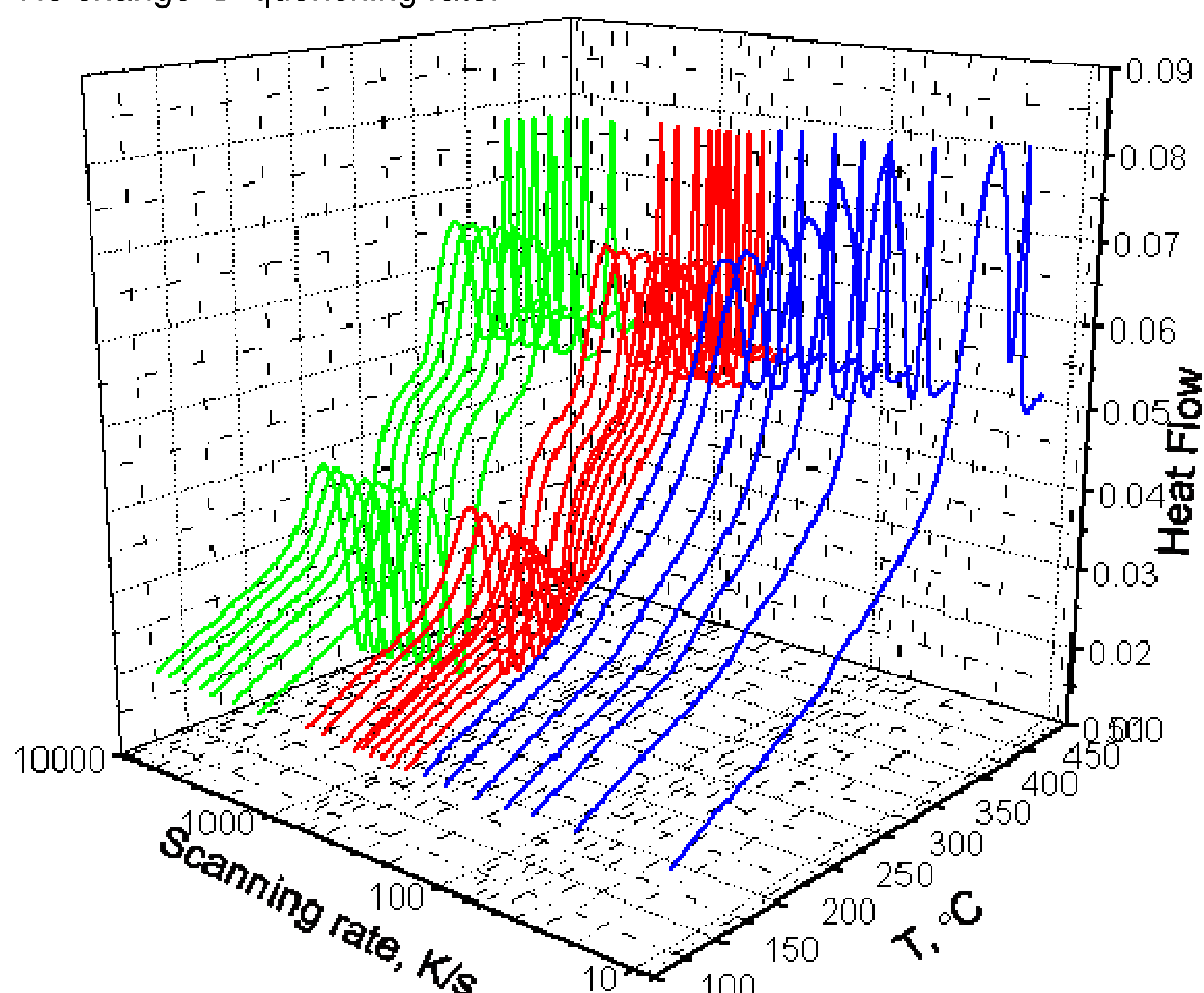


- ❖ Nucleated versus virgin PP material
- ❖ Different crystallization kinetics; dependent on Nucleating Agent

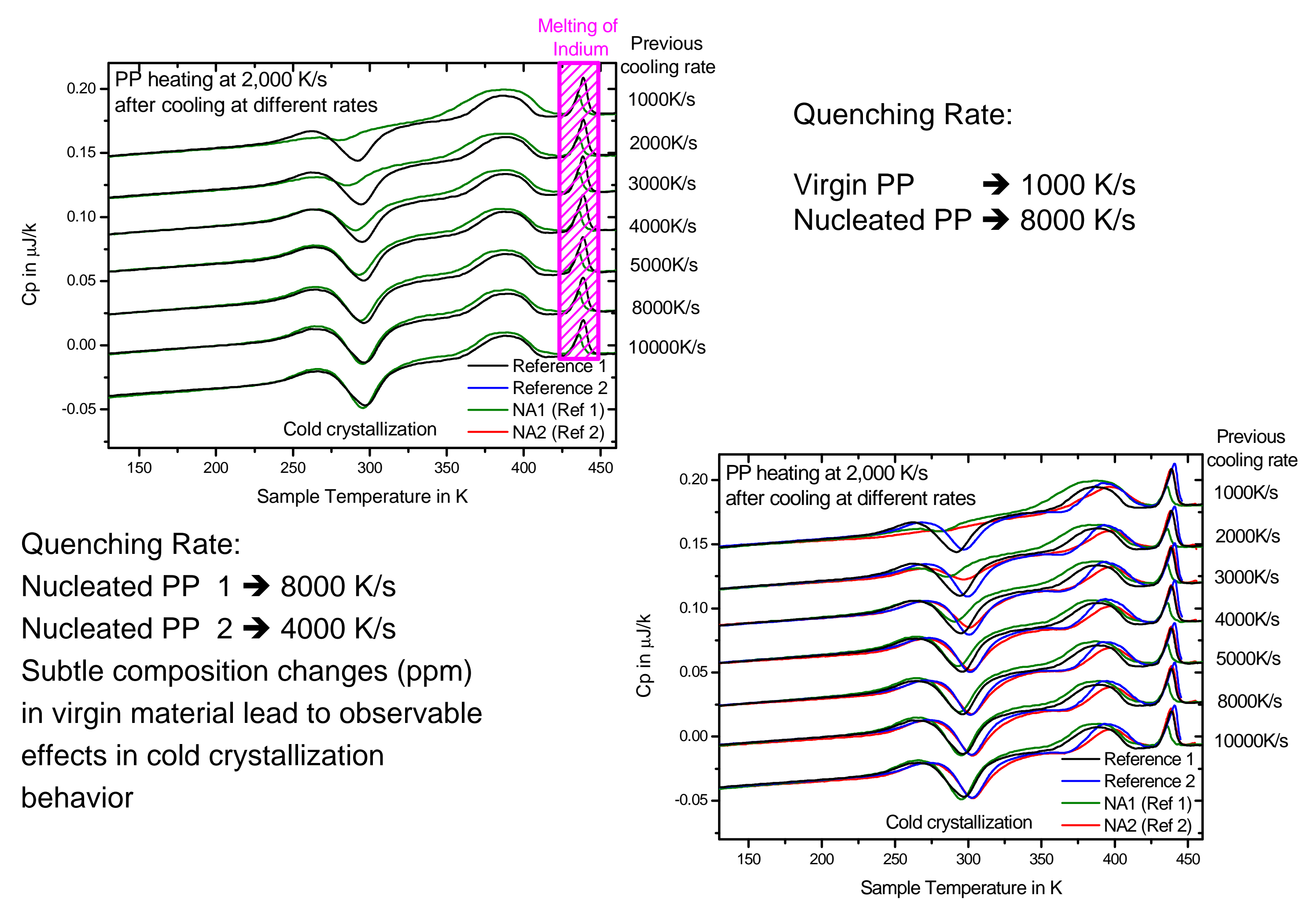
3. Ultra Fast Differential Scanning Chip Calorimeter (UFDSCC)



- Three well distinguishable regions:
- ❖ Change in melting behavior
 - ❖ Cold crystallization development
 - ❖ No change → quenching rate!

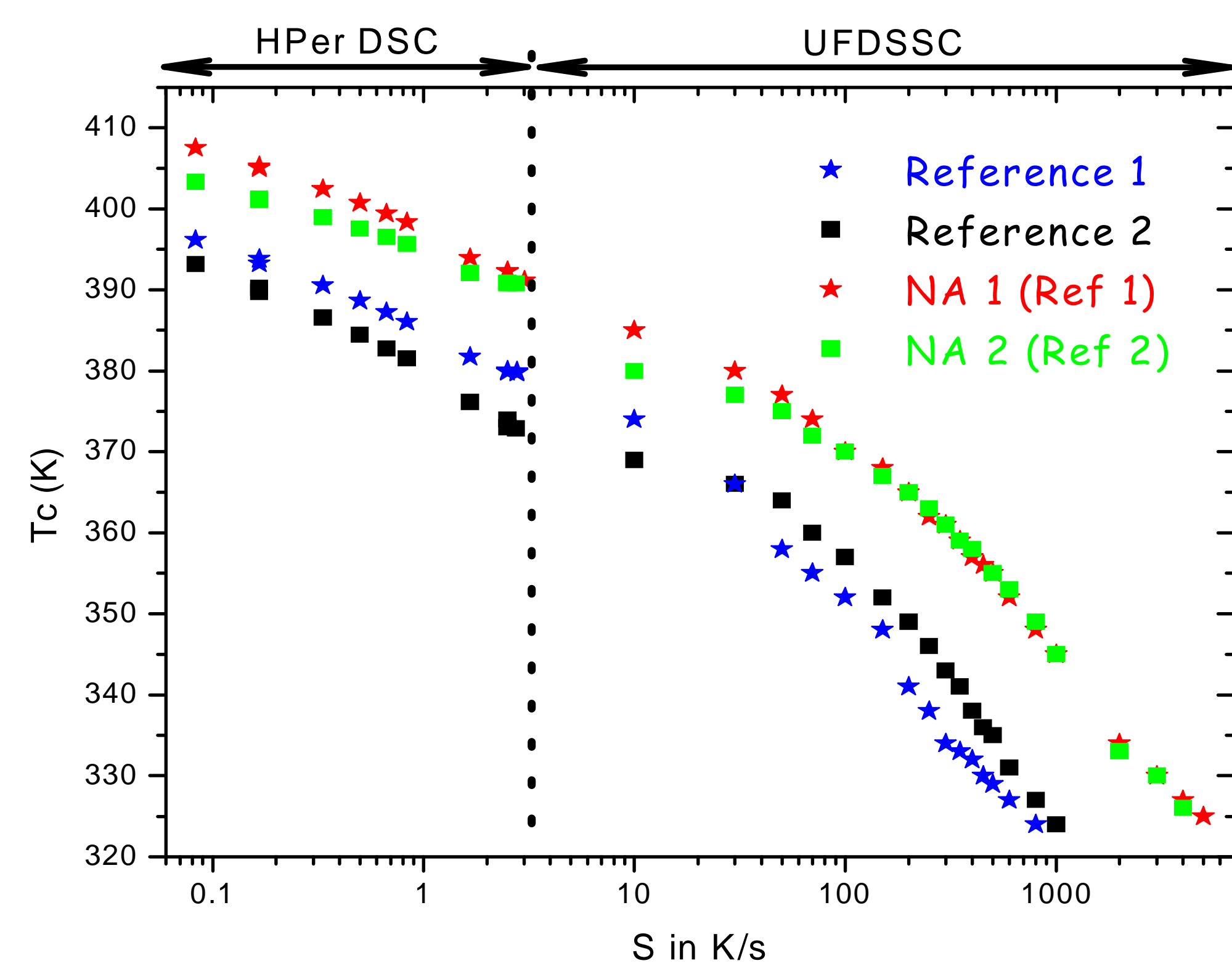


4. Melting of PP by UFDSCC



Quenching Rate:
 Nucleated PP 1 → 8000 K/s
 Nucleated PP 2 → 4000 K/s
 Subtle composition changes (ppm) in virgin material lead to observable effects in cold crystallization behavior

5. Synergy HPer DSC and UFDSCC



Good agreement HPer DSC and UFDSCC

6. Conclusion

In this study, the dependence of cold crystallization on cooling history has been investigated. The outcome of this study revealed elements which will be used for development of crystallization theory from the cold side. Crystallization kinetics of the PP samples with and without the nucleating agents has been studied on the very broad cooling rate range that includes all realistic (processing) speeds. The outstanding agreement between the results of two techniques as HPer DSC [6-9] and UFDSCC has been obtained due to a new calibration approach.

7. References

- [1] S.A. Adamovsky, A.A. Minakov, C. Schick, *Thermochim. Acta* 403 (2003) 55-63.
- [2] M. Merzlyakov, *Thermochim. Acta* 403 (2003) 65-81.
- [3] S. Adamovsky, C. Schick, *Thermochim. Acta* 415 (2004) 1-7.
- [4] A. Grady, P. Sajkiewicz, A.A. Minakov, S. Adamovsky, C. Schick, T. Hashimoto, K. Saijo, *Mater. Sci. Eng. A* 413-414 (2005) 442-446.
- [5] F. De Santis, S. Adamovsky, G. Titomanlio, C. Schick, *Macromolecules* 39 (2006).
- [6] High Performance DSC (HPer DSC; and its commercial equal: HyperDSC): T.F.J. Pijpers, V.B.F. Mathot, B. Goderis, R.L. Scherrenberg, E. van der Vegte, *Macromolecules* 32 (2002) 3601. See also recent Webcasts by V.B.F. Mathot, downloadable for free via www.scite.eu and www.hyperdsc.com;
- [7] G. Vanden Poel and V.B.F. Mathot, *Thermochimica Acta* 446 (2006) 41
- [8] G. Vanden Poel and V.B.F. Mathot, *Thermochimica Acta* 461 (2007) 107
- [9] V.B.F. Mathot, G. Vanden Poel, T.F.J. Pijpers: Chapter 8, Benefits and Potentials of High Performance Differential Scanning Calorimetry (HPer DSC) in Volume 5: Recent Advances, Techniques and Applications (Editors: Michael Brown & Patrick Gallagher); *Handbook of Thermal Analysis and Calorimetry* (Series Editor: Patrick Gallagher), 2008, p.269-297