

## Preparation of nano-sized particles for improved toughness

***Citation for published version (APA):***

Casteren, van, I. A., Goossens, J. G. P., & Meijer, H. E. H. (2001). *Preparation of nano-sized particles for improved toughness*. Poster session presented at Mate Poster Award 2001 : 6th Annual Poster Contest.

***Document status and date:***

Published: 01/01/2001

***Document Version:***

Publisher's PDF, also known as Version of Record (includes final page, issue and volume numbers)

***Please check the document version of this publication:***

- A submitted manuscript is the version of the article upon submission and before peer-review. There can be important differences between the submitted version and the official published version of record. People interested in the research are advised to contact the author for the final version of the publication, or visit the DOI to the publisher's website.
- The final author version and the galley proof are versions of the publication after peer review.
- The final published version features the final layout of the paper including the volume, issue and page numbers.

[Link to publication](#)

***General rights***

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal.

If the publication is distributed under the terms of Article 25fa of the Dutch Copyright Act, indicated by the "Taverne" license above, please follow below link for the End User Agreement:

[www.tue.nl/taverne](http://www.tue.nl/taverne)

***Take down policy***

If you believe that this document breaches copyright please contact us at:

[openaccess@tue.nl](mailto:openaccess@tue.nl)

providing details and we will investigate your claim.

# Preparation of Nano-Sized Particles for Improved Toughness

I.A.van Casteren, J.G.P.Goossens, H.E.H.Meijer

Eindhoven University of Technology, department of mechanical engineering,  
P.O. Box 513, 5600 MB Eindhoven, the Netherlands

## Introduction

For brittle amorphous polymers, the *maximum impact strength* and *minimum loss in stiffness* can be obtained by using easy cavitating nano-sized modifiers. The self-assembly process of diblock-copolymers in micelles can be used to prepare such systems [1]. These micellar structures should be formed in monomers, like styrene or methyl methacrylate (MMA), which after polymerisation form the continuous phase.

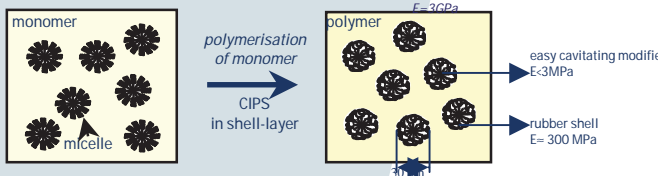


Figure 1: Schematic representation of the proposed morphology.

## Material preparation

The diblock-copolymers were synthesized by the Atom Transfer Radical Polymerisation of methylacrylate (MA) and butylacrylate (BA) using hydrogenated poly(butadiene) (PB) ( $M_n = 3840$  g/mol) macroinitiators.

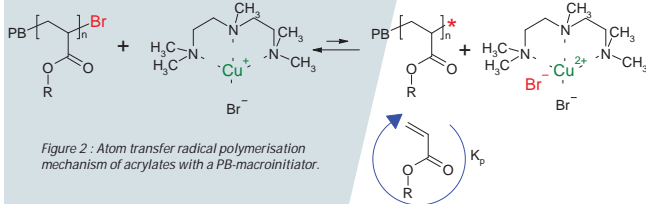


Figure 2: Atom transfer radical polymerisation mechanism of acrylates with a PB-macroinitiator.

## Results

Diblock copolymers were synthesized with a  $M_n$  of 10000 g/mol,  $M_w/M_n \sim 1.10$  and lamellar bulk morphology.

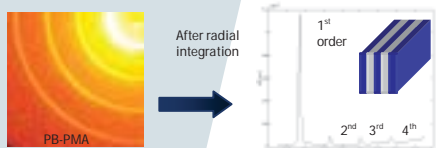


Figure 3: SAXS pattern of PB-PMA presenting the lamellar morphology, d-spacing 179 Å.

The morphology of PS and PMMA blends was studied by:

- ❖ various microscopy techniques
- ❖ time resolved Small Angle X-ray Scattering (SAXS)

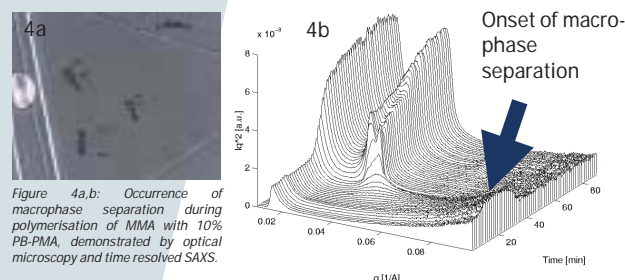


Figure 4a,b: Occurrence of macrophase separation during polymerisation of MMA with 10% PB-PMA, demonstrated by optical microscopy and time resolved SAXS.

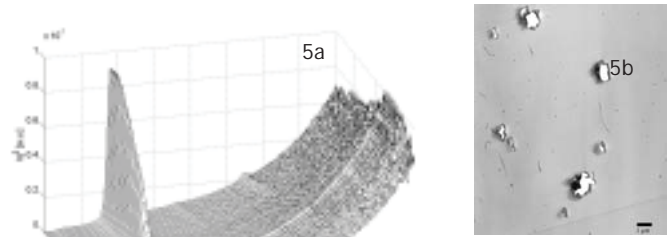


Figure 5a and b: Macrophase separation in PS blend with 10% PB-PMA, demonstrated by time resolved SAXS and TEM.

Incorporation of a strong proton-donating unit, i.e. p-(hexafluoro-2-hydroxy-isopropyl)styrene (HFS), into PS will result in *intermolecular hydrogen bonding* between PS matrix and acrylate-based copolymers and will avoid macro-phase separation [2].

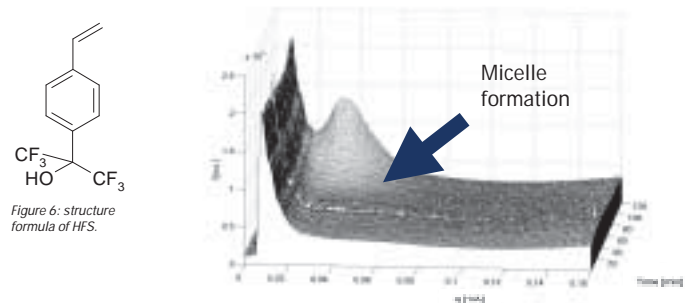


Figure 6: structure formula of HFS.

Figure 7: time resolved SAXS patterns of the polymerisation of styrene with 10% PB-PMA and 5% HFS

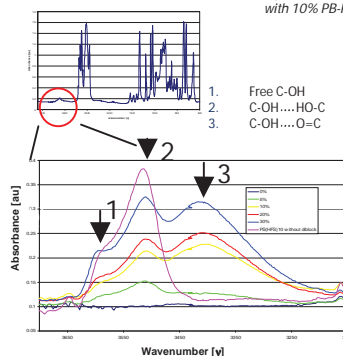


Figure 8: FT-IR plots of PS blends with 10% PB-PMA and various amounts of HFS.

Two different interactions can be detected by infrared spectroscopy:

- ❖ hydrogen bonding between the hydroxyl groups of HFS ( $3530 \text{ cm}^{-1}$ )
- ❖ hydrogen bonding between the hydroxyl groups of HFS and the carbonyl groups of the diblock ( $3390 \text{ cm}^{-1}$ )

## Conclusions

- The proposed nano-sized morphology can be made by the introduction of hydrogen bonding.
- Due to low molecular weight of the diblocks used a significant increase in mechanical properties could not be observed.
- Higher molecular weight diblocks or a crosslinked shell block should be used to increase the impact properties.

## References:

[1] Lipic, P.M., Bates, F.S., Hillmyer, M.A. *J. Am. Chem. Soc.* 120 8963 (1998)  
[2] Pearce, E.M., Kwei, T.K., Min, B.Y. *J. Macromol. Sci.-Chem.*, A21(8&9), 1181(1984)