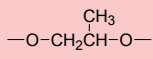


Phase separation analysis by the various microscopes method of polyurethane

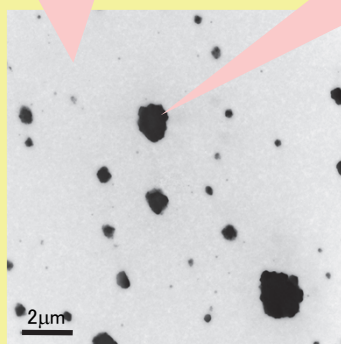
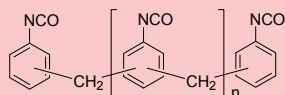
Analysis of phase separation of soft and hard segments in polyurethane resin is possible by TEM observation and the AFM phase imaging. Furthermore, the identification of hard segment is enabled by STEM-EDX elemental map of N element.

Analysis of polyurethane gel

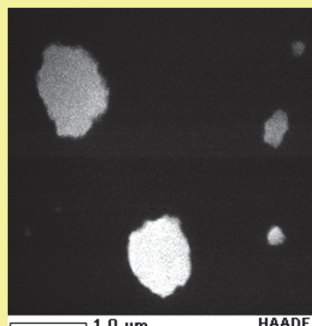
Polyether polyol
(PO/EO copolymer)



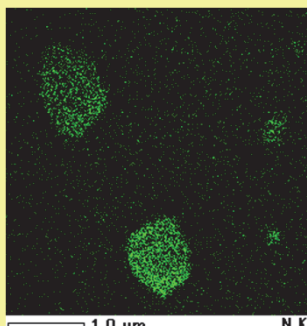
Polyisocyanate (polymeric MDI)



TEM image



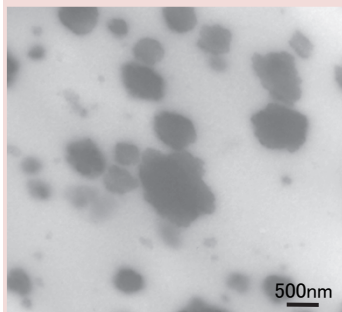
HAADF-STEM image



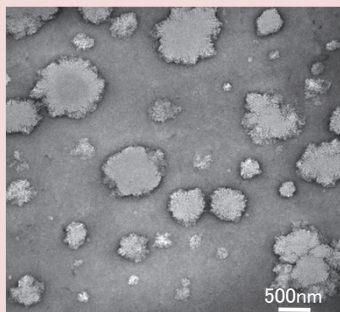
EDX map of N

- Polyurethane gel can be evaluated by TEM imaging cross-sections prepared using a cryo-ultramicrotome.
- The contrast of the phase separation depends on electron density differences between hard segment and soft segment.
- The clear distribution of the N is got in EDX by large-sized (around 100 mm²) silicon drift detector (SDD).
- Island phase is hard segment by EDX map of N can be confirmed and it is revealed that MDI is localized in island phase.

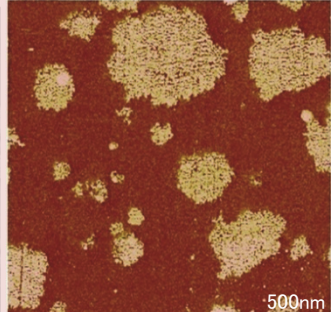
Analysis of general polyurethane



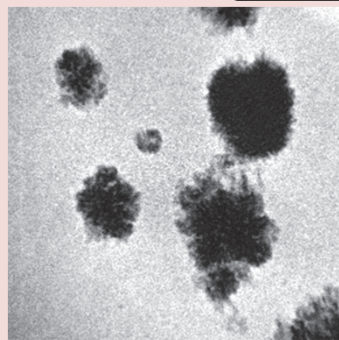
TEM image



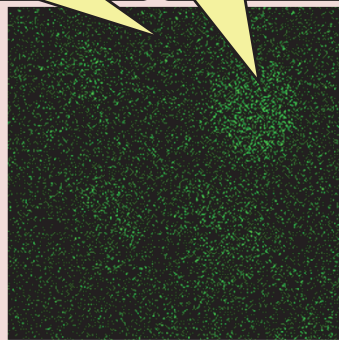
TEM image (RuO₄ staining)



AFM phase image



HAADF-STEM image (RuO₄ staining)



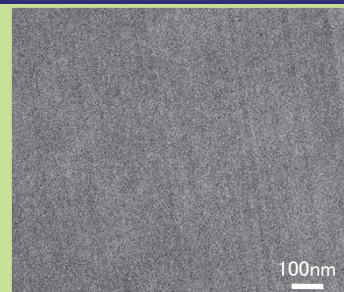
EDX map of N

A small amount of N is detected in matrix.

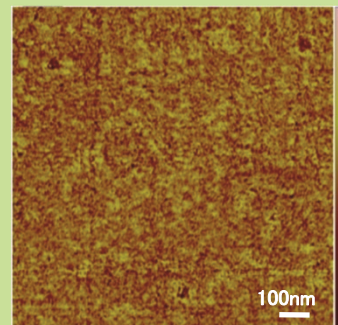
N is detected in island phase.

- Phase separation is indistinct by non-staining TEM observation, but becomes apparent by RuO₄ staining.
- Island phase is hard segment by AFM phase imaging can be confirmed.
- A small amount of N is detected in matrix by N map of EDX and existence of MDI is suggested.

Analysis of hard type polyurethane



TEM image (RuO₄ staining)



AFM phase image

Phase separation cannot be observed by TEM, but fine structure is confirmed by AFM phase imaging.