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SII

Effect of Thickness on Phase Transitions of Organic Thin Film by DSC

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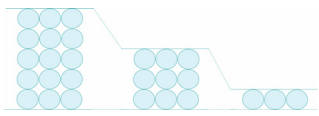
Purpose

- Structure and phase transition for ultra-thin film (thickness: < 1 μm)
- Efficacy of solid-solid interface

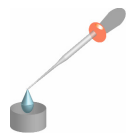
Sample

- n-alkyl alcohols $C_nH_{2n+1}OH$ (n=18)
  Recrystallization from hexane solution
  (purity: > 99.9 %)

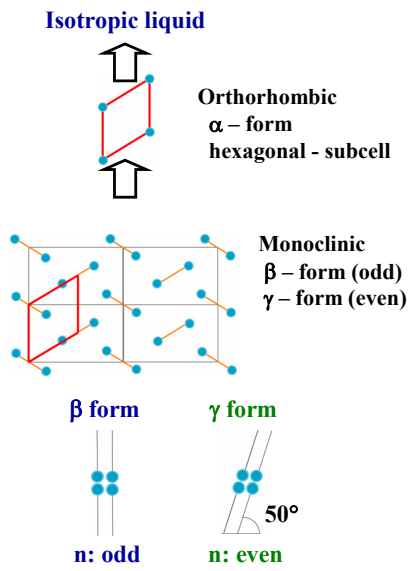


Preparations

- Hexane solution : 0.05 to 1 %
- 10μℓ on aluminum open pan for DSC
- 10μℓ on silicon substrate for AFM and GISAXS



As cast Cry : Crystallization by solution casting
Cool Cry : Crystallization by 5 K/min cooling from melting condition



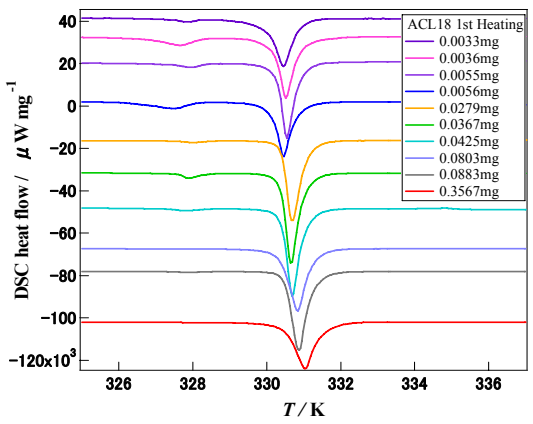
Experiments

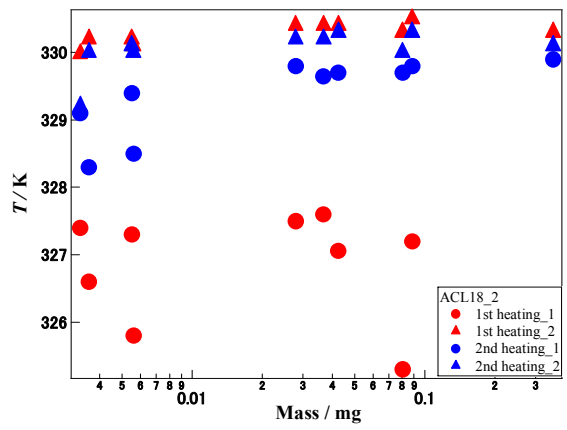
- **Electronic Balance :** Satrorius Supermicro ± 0.2 μg
- **DSC :** X-DSC7000 (SII NanoTechnology)
  Temperature : 290 → 350 → 290 → 350 → 290 K
  Scanning Rate : 5 K/min
- **AFM :** E-sweep (SII NanoTechnology)
  Dynamic force mode
  Operating speed : 0.9 Hz
  Cantilever
  Spring constant : 35 N/m
  Resonance frequency : 300 kHz
- **GISAXS :** SPring8 FSBL03XU
  Wave length : 0.1 nm^{-1}
  Camera length: 2031 mm
  Incidence angle: 0.17°
  Detector: Imaging Plate (R-AXIS)

Results

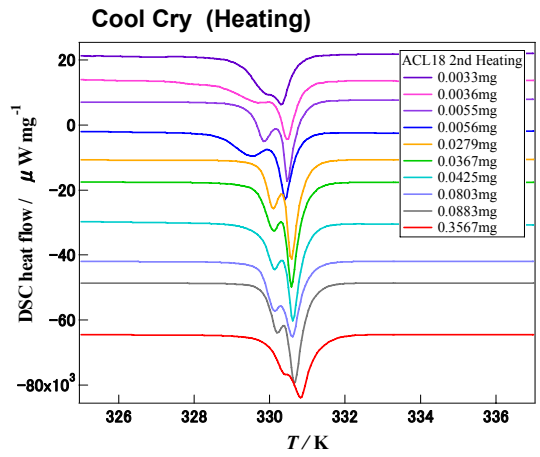
《 DSC measurements 》

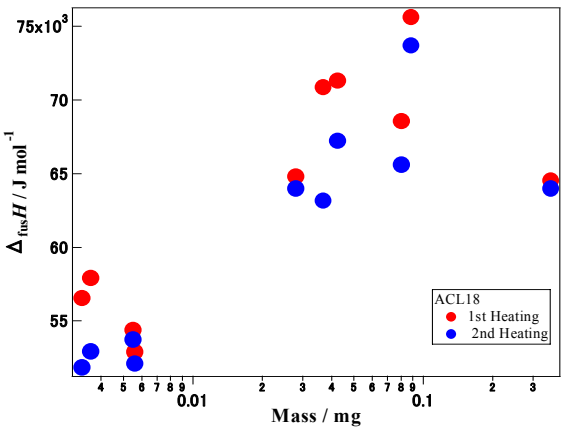
As cast Cry (Heating)



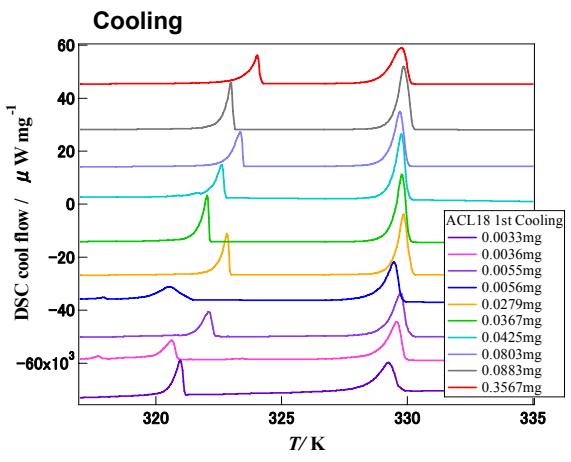


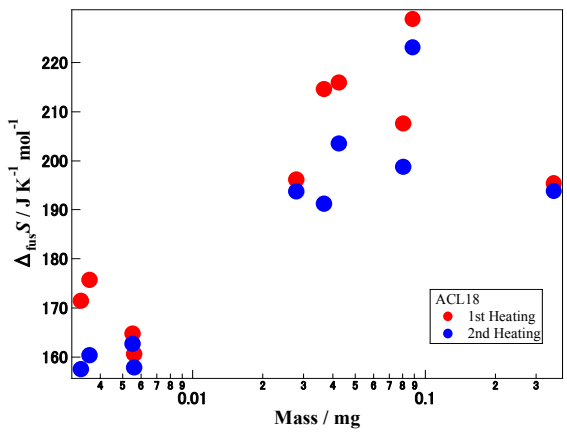
Cool Cry (Heating)





Cooling



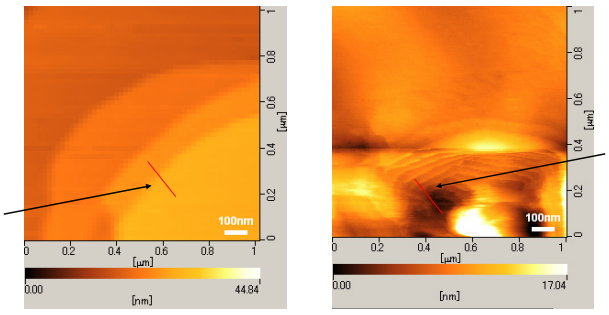


《 AFM 》

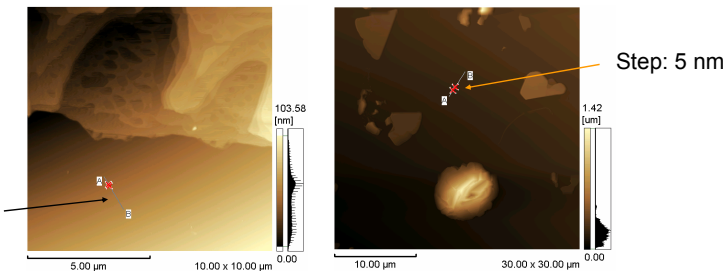
Topography

As cast Cry | Cool Cry

Multilayer crystal

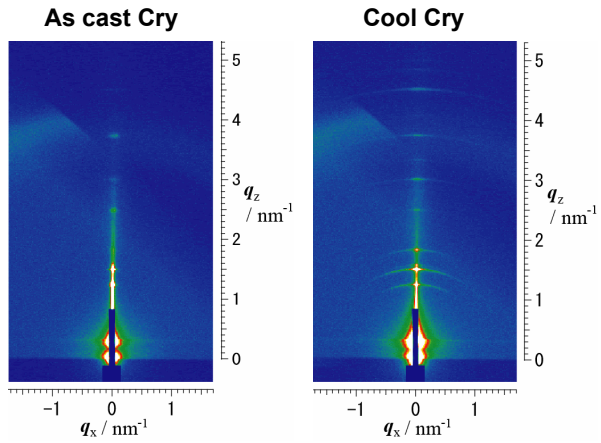


Monomolecular crystal

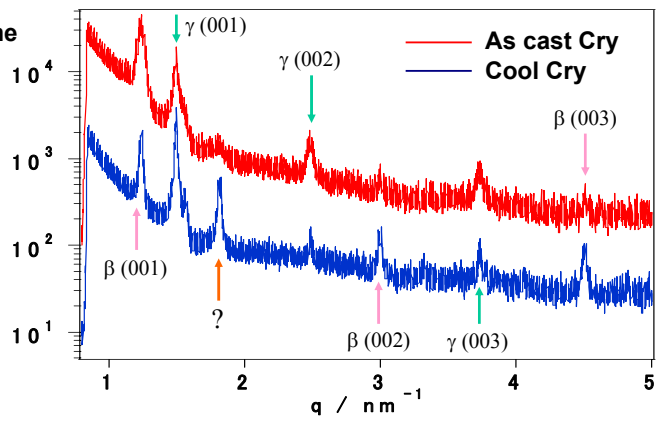


《 GISAXS 》

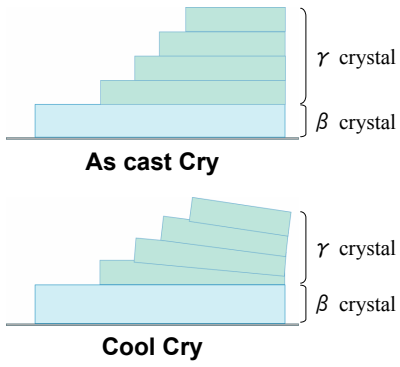
As cast Cry | Cool Cry



Out-of-plane



Conclusion

The phase transitions and the structure of the minute sample (μg- mg) have been observed.

- The thickness of the As cast Cry and the Cool Cry was uneven.
- The monomolecular layer crystal and multi-molecular layer crystal was observed for both samples by AFM.
- The smaller the sample masses of As cast Cry and Cool Cry became, the decrease the melting temperature, the melting enthalpy, and the melting entropy became.
- Two kinds of melting of $C_{18}H_{37}OH$ were observed. It was clear that the metastable β crystal and the stable γ crystal coexisted.
- It was indicated that the metastable β crystal was formed in the monomolecular layer crystal which directly contacted the substrate interface.