Analysis of Polymorphic Nucleation and Phase Transformation in Cooling Crystallization using Quartz Crystal Microbalance

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Based on capability of simultaneously characterizing both solid and liquid phases, quartz crystal microbalance (QCM) was applied to analyze the polymorphic nucleation and phase transformation in cooling crystallization. Sulfamerazine has two polymorphs of metastable phase of form-I and stable phase of form-II, with dominant face [020] and [002] respectively. The QCM sensor was self-assembled with 11-amino-1-undecanethiol, providing a layer of $-NH_2$. The formation of form-I crystal on sensor surface was prevented, however form-II was allowed. Thus, two polymorphs can be distinguished by QCM response. The mechanism lied on hydrogen bonding ability of each polymorph with the $-NH_2$ on sensor surface. Dominant group aromatic N of form-II can form hydrogen bond with $-NH_2$ on sensor surface, whereas difficult for form-I with dominant group of $-NH_2$. In cooling crystallization, QCM responded to the nucleation of form-I as a resonant frequency increase due to concentration depletion in liquid phase. As for form-II, a significant decrease of resonant frequency was induced at induction point due to crystal formation on sensor surface. The in-situ result of polymorphic nucleation and phase transformation profile by QCM were confirmed by Raman spectroscopy.