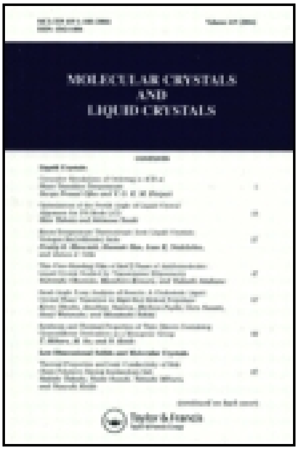

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Phase Transition Phenomena of an Amphiphile-coating Film on a Quartz Crystal Analyzer

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Thermal phase transitions of a cast film of an imidazole-containing amphiphile on a quartz crystal were monitored by the in situ measurement of the resonant frequency and the resonant resistance together with differential scanning calorimetry. The change of phase transition behaviors with scanning times could be confirmed by using the resonant resistance technique which was much more sensitive to the viscoelastic phase transitions than was the change of the resonant frequency.

Keywords: phase transition; amphiphile; quartz crystal analyzer

INTRODUCTION

The quartz crystal has been employed as a sensitive mass detecting device[1] and a liquid viscosity monitoring device[2]. In particular, phase transition phenomena of liquid crystals[3], lipid multilayer films[4], and Langmuir-Blodgett (LB) films[5] have been investigated by using the quartz crystal and the surface acoustic wave device. In the above studies, however, only the resonant frequency change was measured to examine the phase transition phenomena, but it is not enough to clarify up to the detail of the viscoelastic phenomena. Specifically, the viscoelastic phenomena on the coating films can be an important cause of the resonant frequency change, thus it can have an effect on the reliability of correlation between mass change and resonant frequency change[6]. Herein, we report the phase transition phenomena of a viscoelastic coating film of an imidazole-containing amphiphilic molecule, n-octadecyl urocanate (OU), which is thought to form photochemically active film of imidazole-coordinated metal complexes. Dependance of the film state on thermal history could be efficiently characterized by differential scanning calorimetry (DSC) together with a quartz crystal analyzer by the in situ measurement of the resonant frequency and the resonant resistance.

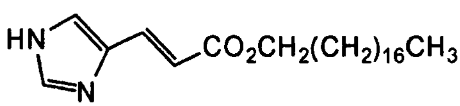
EXPERIMENTALS

Materials

The amphiphilic compound OU was synthesized by heterogeneous reaction of urocanic acid with n-octadecanol using dicyclohexylcarbodiimide as coupling agent in dichloromethane.

Measurement

We made an impedance analyzer for measuring the resonant frequency and the resonant resistance of the AT-cut quartz crystal (9 MHz), as described previously[7]. The compound OU was dissolved in chloroform to a concentration of 30 mg/ml and cast onto the quartz crystal to form 183 kHz coating after solvent evaporation.



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RESULTS AND DISCUSSION

DSC thermograms for the amphiphilic compound OU were obtained in bulk state. Figure 1a and 1b were respectively obtained from the first and the second heating scans. The thermograms obtained from more than the third scan were almost coincided with the Figure 1b. The points a to i on the thermograms are indicated for the comparison with the isotherms of the resonant frequency and

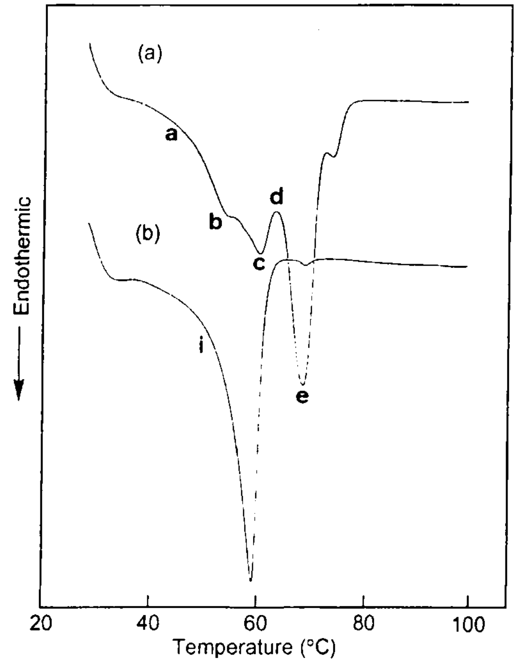

FIGURE 1 DSC curves of OU obtained from bulk measurement. (a) The first and (b) the second heating scans of the same sample. The scan rate of 10 °C/min was applied.

the resonant resistance of the OU-coating film on a quartz crystal.

Figure 2a and 2b respectively show the responses of the resonant frequency and the resonant resistance for the OU-coated quartz crystal on the heating and cooling cycle. At the first heating run, the resonant frequency and the resonant resistance changes can be separated into four parts from point a to point e. Together with Figure 1a the isotherms show that more than two kinds of phase transitions are involved in the first heating process.

In the cooling process from the point e to the point h, the resonant frequency and the resonant resistance showed much simpler change. The points c' and d' corresponding to the points c and d were detected in the resonant resistance change during the cooling process, while not discernible in the resonant frequency change. In the second heating run, the resonant frequency and the resonant

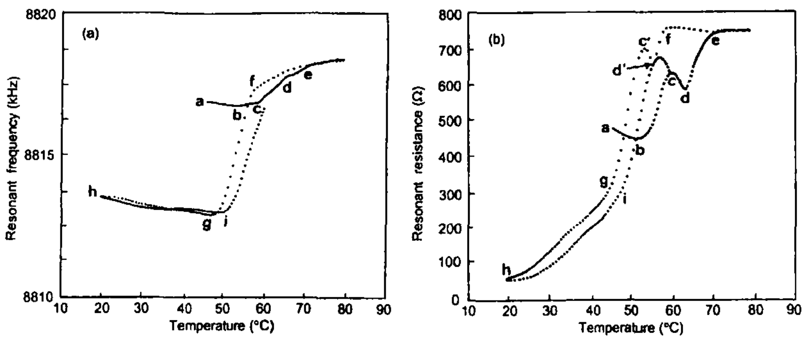

FIGURE 2 (a) The resonant frequency and (b) the resonant resistance responses for the OU-coated quartz crystal (183 kHz coating) with heating and cooling cycle of 20-80 °C. The point a indicates the starting point.

resistance followed the cooling curves with a slight difference.

The comparison of the points a to e of Figures 1a, 2a, and 2b shows that change of the resonant resistance is clearly represented by the turning point of the isothermic curve, while only the slight inflections were observed in the isotherm of the resonant frequency. This indicates that the isotherm of the resonant resistance can be a sensitive tool to investigate the thermal property of organic films.

Acknowledgments

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