Poster Presentation

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X-ray reflectivity study of the glass transition temperature of thin films

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The glass transition takes place in amorphous materials (like polymers) during heating or cooling, and can be described as reversible transition from a hard and brittle state into a rubber-like state. Although physical properties of the material change significantly during the glass transition, this is not a phase transition of the material. The temperature at which the transition between the glassy and rubbery state occurs is called the glass transition temperature, and this temperature is always lower than the melting temperature. Thermodynamically, the glass transition is associated with transfer of heat between the system and its surrounding and with an abrupt volume change. Previously it was shown that the glass transition temperature of nano-films is different from that of bulk materials [1], which signifies the importance of determining this parameter for such systems. In the current work, we use quick X-ray reflectivity (qXRR) measurements to determine the glass transition temperature of polyvinyl acetate (PVAc). PVAc is rubbery synthetic polymer with the formula (C4H6O2), a density of 1.18 g/cm3, and a glass transition temperature for bulk material of 30oC [2]. Regular X-ray reflectivity measurements are based on θ/2θ scans at grazing incidence and typically require 0.5-1.5 h for a single scan. The qXRR technique is based on simultaneous measurement of the whole angular x-ray reflectivity profile and is suitable for in-situ measurement without moving the sample and/or the x-ray optics. Thus, the qXRR technique allows for very fast measurement of the x-ray reflectivity curves (duration of each scan is typically 0.1–20 sec [3]), which permits studying the time evolution of chemical, thermal, and mechanical changes at the surface and interface of different materials. X-ray reflectivity measurements give information about both density and thickness of thin films, and are suitable for studying glass transition phenomena. Nano-thickness PVAc layers on a Si substrate were examined with the qXRR technique, with x-ray reflectivity scans (each 10-seconds in duration) being recorded while temperature was changed from 20 to 50oC (total of 331 scans over 7 hours and 46 minutes). In the current paper, the experimental setup, the data-processing, and the analysis of the results from the qXRR measurements will be presented.

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