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Field-cycling NMR relaxometry as a tool of molecular rheology–applications on polymer melts

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NMR relaxometry provides rich information on the dynamics in complex liquids, such as polymer melts. Using the field-cycling (FC) technique, the spin-lattice relaxation rate R_1, reflecting the spectral density, can be measured over a broad range of frequencies ω, appreciably reaching below the earth field, with sophisticated low-field equipment. Assuming frequency-temperature superposition, the effective window extends about ten decades in the NMR susceptibility representation $\chi''(\omega):=\omega R_1(\omega)$, when master curves are constructed. In high-M polymers, the local (a-process), Rouse and entanglement dynamics is covered. The broad frequency range allows for a transformation into the time domain; a time auto-correlation function is gained. Concerning ^{1}H, relaxation is caused by fluctuations of the dipolar interaction, comprising intra- as well as inter-molecular contributions. The isotope dilution technique allows for a separation yielding both, the re-orientational correlation function $C_2(t)$ as well as $C_{inter}(t)$, providing the segmental mean square displacement (MSD). Complementing the FC data with such of field-gradient NMR reaching even longer times, the full MSD is probed in highly entangled polymers. All power-law regimes of the tube-reptation model are reproduced. Concerning $C_2(t)$, however, the predictions are only confirmed in parts. Comparing FC NMR with shear rheology, much similarity is found, regarding the local, the Rouse and the terminal regime. This renders FC NMR as a powerful tool of molecular rheology. As NMR relaxometry addresses molecular correlation functions, the slow dynamics in the entanglement regime is resolved. In particular the constrained Rouse, the reptation regime as well as the corresponding crossover times. In contrast, the shear data is governed by the rubber plateau.

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Structural, electrical and optical properties of Cr doped ZnO thin films: Influence of Cr concentration and annealing temperature

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The pure ZnO and Cr doped ZnO (Cr:ZnO) thin films (thickness: 200 nm) were grown on both side polished silica (SiO_2) substrates by RF magnetron sputtering at room temperature. As- deposited samples were annealed at 400°C, 500°C and 600°C for 45 min in quartz annealing furnace system, respectively. The structural and chemical composition analyses were carried out by X-ray diffraction (XRD), scanning electron microscope (SEM) and energy dispersive X-ray spectrometry (EDS). XRD studies revealed that the almost single crystalline hexagonal Wurtzite structure of pure ZnO film disappears with increasing Cr concentration and annealing process contributes the long range crystal order of films. SEM images show that average grain size is around 30 nm. EDS results indicate that only Zn, Cr and O elements are present in the Cr:ZnO thin films. The electrical properties were investigated by using the Four Point Probe (FPP) method. The smallest electrical resistivity for doped samples were obtained at 600°C annealing temperature and specifically as 5.34×10^{-4} Ω.cm belonging to $Cr_{8.21}ZnO$. The electrical conductivity and carrier concentration of the films are increased while mobility carriers are decreased with increasing Cr content. The optical properties were studied in the wavelength region of 200-1000 nm by employing UV-Vis spectroscopy. Pure ZnO and Cr:ZnO films that include 3.22 at.% Cr content (or less), have transmittance above 70% between 400-1000 nm before annealing. It was observed that all annealed samples have higher average transmittance in the range of 200–1000 nm as compared to as-deposited films. Tauc plots were drawn to specify the optical energy band gap (E_g) of as-deposited and annealed samples. The E_g increases from 3.24 eV to 3.90 eV with increasing Cr content from x:0 at.% to 3.22 at.% and then decreases to 1.60 eV for 11.80% Cr concentration.

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