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Advances of unilateral mobile NMR in nondestructive materials testing

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Abstract

Unilateral mobile NMR employs portable instrumentation with sensors, which are applied to the object from one side. Based on the principles of well-logging NMR, a hand-held sensor, the NMR-MOUSE (MObile Universal Surface Explorer) has been developed for nondestructive materials testing. In the following, a number of new applications of unilateral NMR in materials science are reviewed. They are the state assessment of polyethylene pipes, the characterization of wood, the in situ evaluation of stone conservation treatment, high-resolution profiling of rubber tubes and 2-D imaging for defect analysis in rubber products.

Keywords: Mobile NMR; NMR-MOUSE; Unilateral NMR imaging; Polyethylene; Cultural heritage

1. Introduction

NMR can be conducted in highly inhomogeneous fields by detecting echoes. The initial magnetization converted into magnetization detectable by one or more echoes can be modified by magnetization filters in the same way as contrast is introduced in NMR imaging [1]. By systematic variation of a filter parameter and evaluation of the transverse relaxation during detection of the echo train, 2-D data sets are obtained for correlation of parameters in a manner similar to multidimensional spectroscopy [2]. The use of this type of NMR is of great value in well logging [3] and it also offers interesting applications in materials science [1,4]. In solid materials, translational molecular diffusion is absent and higher field gradients can be tolerated. This implies that unilateral NMR can be conducted at higher fields (0.5 vs. 0.02 T) and higher field gradients (10 vs. 0.1 T/m) for materials investigations than for well logging. A compact unilateral sensor developed for nondestructive materials testing is the NMR-MOUSE (MObile Universal Surface Explorer, registered trademark of RWTH Aachen) [5]. The original NMR-MOUSE employs a U-shaped magnet with the radiofrequency coil situated in the magnet gap following earlier concepts of unilateral sensors with electromagnets for moisture detection in soil and road decks [6]. A simpler design is the bar-magnet NMR-MOUSE that consists of a figure-of-8-type surface coil mounted on one of the pole faces of a bar magnet [7]. This "Easy NMR-MOUSE" provides an excellent signal-to-noise ratio in a single-shot CPMG train from unfilled natural rubber (Fig. 1). By adjusting the magnet geometry of the original NMR-MOUSE, the "Profile NMR-MOUSE" is obtained with a thin and flat sensitive volume that is suited for measuring depth profiles with a spatial resolution of better than 30 μm. All measurements reported below were conducted with CPMG sequences.

2. Applications of the original NMR-MOUSE

The Easy NMR-MOUSE has a penetration depth of about 3 mm, whereas the maximum usable depth is 10 mm and more for the original NMR-MOUSE, depending on the physical dimensions of the components. Both devices can operate with dead-times less than 10 μs, providing minimum echo times of 20 μs, so that they are well suited for analysis not only of rubber but also of semicrystalline polymers like polyethylene (PE) and cellulose.

The CPMG echo decay of PE can well be fitted with the sum of two exponential functions, where the fast relaxing component $A_{\mathrm{short}}\exp\{-t/T_{2\mathrm{eff,short}}\}$ is assigned to the

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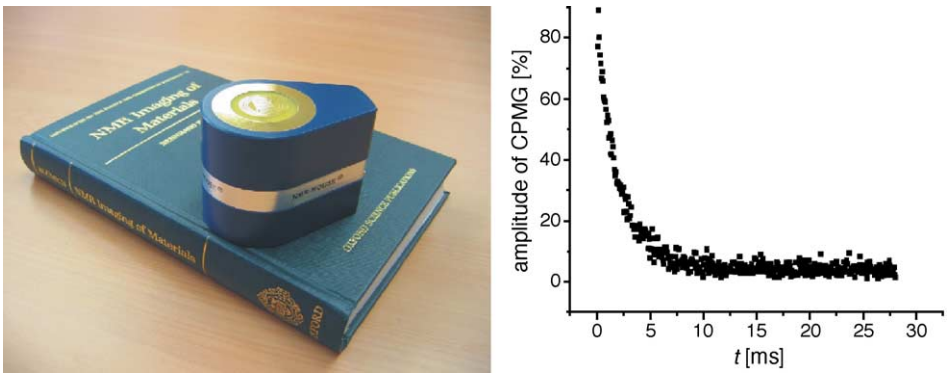

Fig. 1. Easy NMR-MOUSE (left) with a bar magnet and single shot CPMG echo decay from unfilled natural rubber (right).

crystalline regions and the slowly relaxing component $A_{\text{long}}\exp\{-t/T_{2\text{eff,long}}\}$ to the amorphous regions. The fit parameters are interpreted in terms of the crystallinity $A_{\text{short}}/(A_{\text{short}}+A_{\text{long}})$, the average size of the crystallites $1/T_{2\text{eff,short}}$, and the molecular order in the amorphous domains $1/T_{2\text{eff,long}}$. Although this interpretation still needs to be verified in further investigations, the data obtained on PE pipes can consistently be explained in this way.

PE water pipes are squeezed tight to turn off the water flow for repair, and lifetime predictions are often based on measurements at 80°C. For this reason, we have subjected PE pipes to such treatment and analyzed the NMR signal at eight points symmetrically placed on the circumference in the deformation zone (Fig. 2). The data scatter from point to point is due to local strain induced by cooling and crystallization during the production. Upon deformation, the crystallinity decreases, and upon annealing 40°C below the melting temperature of the ideal PE crystal, it decreases further. During deformation macromolecular chains are drawn out of the small crystallites. At the same time, local order is induced in the amorphous domains by the deformation. This order is reduced upon annealing when

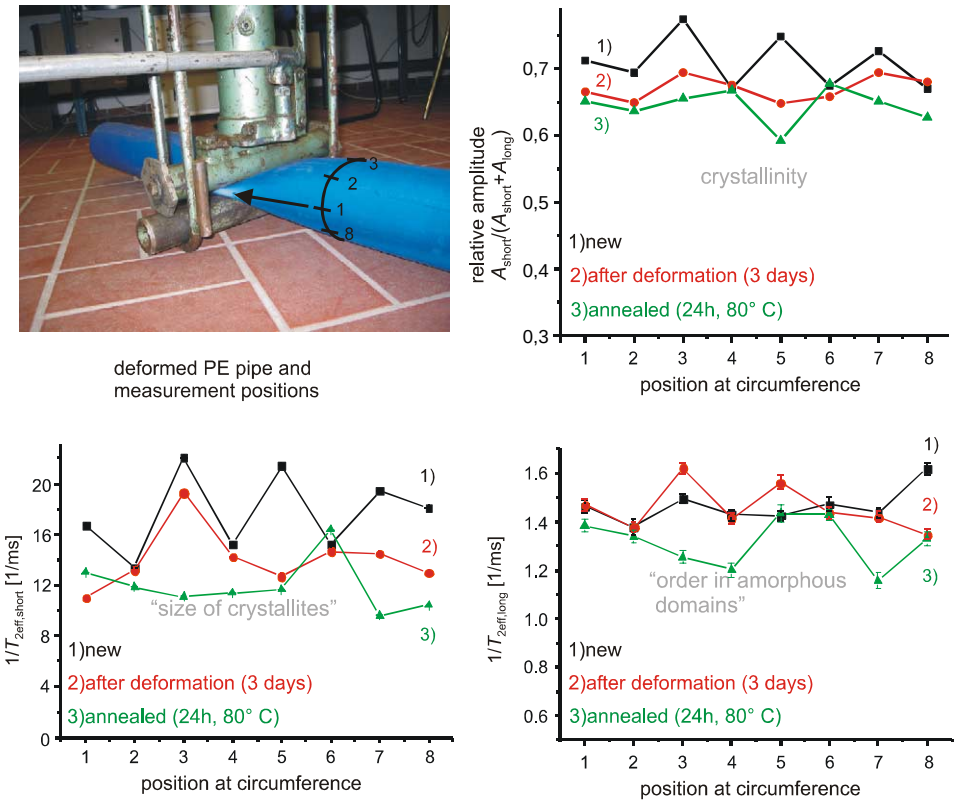

Fig. 2. Nondestructive morphology characterization of PE pipes. Top left: deformation and measurement positions. Top right: Relative amplitude ratio corresponding to the degree of crystallinity. Bottom left: Relaxation rate of the rigid component interpreted as the mean size of the crystallites. Bottom right: Relaxation rate of the soft component interpreted as the molecular order in the amorphous domains.