Ultrasonic dynamic mechanical analysis (UDMA) for polymer characterization

Subjects: Polymer Science

Contributor: Francesca Lionetto, Alfonso Maffezzoli

Ultrasonic wave propagation has been used for the high frequency dynamic mechanical analysis of polymers with the aim to monitor the changes of viscoelastic properties in polymers associated with glass transition, crystallization, physical or chemical gelation, crosslinking, and curing reactions.

Keywords: Ultrasonic wave propagation ; viscoelastic properties ; dynamic mechanical analysis

---

Ultrasonic dynamic mechanical analysis (UDMA) is based on the propagation of low intensity ultrasound, which in polymers strongly depends on the polymer viscoelastic behaviour and the measured properties are significantly affected by the phase transitions occurring with changing temperature and pressureor during chemical reactions. Therefore, the application of low intensity ultrasound, acting as a high frequency dynamic mechanical deformation applied to a polymer, can monitor the changes of viscoelastic properties associated with glass transition, crystallization, physical or chemical gelation, crosslinking, and curing reactions. Thanks to the non-destructive character (due to the very small deformation amplitude), low intensity ultrasound can be successfully used for polymer characterization. Moreover, this technique has a strong potential as a sensor for on-line and in-situ monitoring of production processes of polymers and polymer matrix composites.

Commercial equipments operating as those used for Dynamic Mechanical Analysis (DMA) at mechanical frequencies are still not available. However, some custom made ultrasonic devices for polymer characterization can be found in the literature [1][2]. Among others, Lionetto et al. [3] described a UDMA equipment operating up to 210 °C (Fig.1). It is able to propagate and receive low intensity longitudinal waves in the 1-10 MHz frequency range and to operate both in pulse-echo and in through-transmission mode. The ultrasonic equipment iscoupled with a rotational rheometer allowing simultaneous characterization of a sample by high frequencies (1-10 MHz) ultrasonic waves and low frequency (0.1-10 Hz) shear oscillations, providing information on its viscoelastic behaviour over a wider frequency range than a standard DMA. The ultrasonic velocity and attenuation, measured by UDMA, can be used to obtain the viscoelastic properties of tested polymers, i.e. storage and loss complex longitudinal moduli and the related loss factor, which can be correlated with the results of conventional DMA, carried out at mechanical frequencies (0.1-10 Hz).

UDMA has been successfully applied on different polymers in order to study:

- Curing of thermosetting resins (both epoxy and unsaturated polyesters), whose structural changes occurring at gelation and vitrification can be detected from the evolution of viscoelastic properties. Ultrasonic velocity was also correlated with the degree of reaction [3][4][5];
- Crosslinking ofsuperabsorbent hydrogel. TheUDMA results were compared to the degree of crosslinking measured by NMR and DMA analysis [6];
- Swelling kinetics of dry PVA hydrogels [7][8];
- Retrogradation of wheat starch gels on ageing in the rubbery state [9];
- Dispersion of nanofillers into polymer matrices [10][11];
- Crystallization kinetics of poly-lactic acid (PLLA) and linear low-density polyethylene (LDPE) [7][8];
- Gelation of waxy crude oils on cooling due to the precipitation of paraffin [12];
- Cure monitoring of composite matrices during manufacturing processes, adopting a non-contact (air-coupled) set up between sample and ultrasonic probe [13][14].

References

1. J. Appl. Polym. Sci. 2003, 88, 1665–1675; https://doi.org/10.1002/app.11822.
2. Polym. Test. 2009, 28, 561–566; https://doi.org/10.1016/j.polymertesting.2009.02.005.
3. Mater. Sci. Eng. A Struct. 2004, 370, 284–287; doi:10.1016/j.msea.2003.07.025.
4. J. Polym. Sci. Polym. Phys. 2005, 43, 596–602; doi: 10.1002/polb.20359.
5. Materials 2013, 6(9), 3783-3804; https://doi.org/10.3390/ma6093783.
6. Polymer 2005, 46, 1796–1803; doi:10.1016/j.polymer.2005.01.008.
7. Appl. Rheol. 2005, 15, 326–335.
8. Adv. Polym. Tech. 2008, 27, 63–73; DOI 10.1002/adv.
9. J. Food Eng. 2006, 75, 258–266; doi:10.1016/j.jfoodeng.2005.04.015.
10. IEEE Trans. Nanotechnology2016, 15, 731-737; doi:10.1109/TNANO.2016.2530697.
11. Materials 2019, 12(16), 2530; https://doi.org/10.3390/ma12162530.
12. Rheol. Acta2007, 46, 601–609; doi:10.1007/s00397-006-0144-9.
13. Ultrason. Ferrelectr. Freq. Control IEEE Trans. 2007, 54, 1437–1444; doi: 10.1109/TUFFC.2007.404.
14. Macromol. Symp. 2007, 247, 50–58; doi:10.1002/masy.200750107.