PHD THESIS : Non destructive ultrasonic characterization of polymer ageing

This proposed PhD thesis will be based in the Acoustic & Piezoelectricity of GREMAN laboratory pole in collaboration with the LMR laboratory and be supervised by P. Tran Huu Hue and J. Fortineau (GREMAN) and S. Boucaud Gauchet (LMR).

GREMAN laboratory (UMR CNRS 7347 - University of Tours - INSA Centre Val de Loire):

GREMAN, a research group on materials, microelectronics, acoustics & nanotechnology, is a laboratory of over 100 people with 42 permanent academic staff, 16 administrative & technical staff, 38 PhD students and 10 post-doctoral researchers. This joint research laboratory of Tours University and CNRS is in partnership with the French alternative energies and atomic energy commission (CEA) and INSA Centre Val de Loire. Since its creation in 2012, the main activities of GREMAN laboratory have focused on materials, components and devices for energy efficiency and microelectronics in 5 key topics:

- functional oxides for energy efficiency: combinatory synthesis and nanostructuration;
- magnetic and optical properties of ferroic and electronic correlation materials;
- innovative materials and components for power and RF microelectronics: wide bandgap and porous semiconductors and their applications;
- piezoelectric and capacitive micro and nano systems for ultrasound transducers and energy conversion;
- methods and instrumentation for ultrasonic characterization of complex media.

This PhD thesis concerns the last research topic, which is performed at the INSA Centre Val de Loire in the Acoustic & Piezoelectricity pole. The skills of the researchers are the non-destructive ultrasonic characterization using linear and nonlinear technics and the modeling (numerical and analytical) of the acoustic wave propagation in a complex medium [1,2,3,4]. The experimental equipment of the pole is composed of ultrasonic transducers (in contact and in immersion), pulse generator, function generator, oscilloscopes, spectrum and network analyzers, water tank, L.A.S.E.R interferometers and acoustic microscopy device.

LMR laboratory (University of Tours - INSA Centre Val de Loire):

Mechanical and rheological laboratory (LMR), University of François Rabelais of Tours, was created in 1990. LMR is located in Polytech'Tours school (depends on the University of Tours) and in INSA Centre Val de Loire (Blois). The main research theme is the dynamic characterization of materials and structures. This theme is divided into three sub topics included the characterization and the modeling of materials behavior, the fatigue and durability of materials, and structural vibrations. The studied materials are metals, ceramics, elastomers and polymers [5].

The laboratory LMR will participate to this work by bringing its knowledge in polymer materials, in particular the sample preparation, and the physical-chemical characterization and the analysis of mechanical properties under monotone and cyclic loading. Some research studies were conducted on the thermomechanical degradation of filled rubber

(amorphous polymer with rigid particles) used in automotive applications [2]. The thermal and mechanical degradations of formulations were analyzed and the contribution of structural factors on the fatigue behavior was determined. Two thermal degradation mechanisms were proposed. It was shown that the kinetic of theses mechanisms induced changes in their mechanical properties and fatigue damages.

Background and motivation:

Polymer materials are used in a wide field of industrial applications. Most of them require better mechanical properties and more specifically fatigue resistance. In high technological applications, such as storage tanks for compressed gas, polymer products must offer extremely high levels of durability under more and more critical conditions of use: complex mechanical cyclic solicitations and environmental conditions such as high temperature or high temperature variation cycles. Today, the study of polymer products degradation needs a use of several technical analysis that induce their destruction in order to collect samples.

The aim of the PhD thesis is to develop a specific non-destructive ultrasonic instrumentation in order to monitor the degradation of polymer material induced by both environmental conditions and mechanical solicitations. The final interest is to provide new tools for nondestructive technic to optimize quality process. This study consist of an original research approach based on both polymer material sciences and ultrasonic characterization due to the association of two research laboratories: GREMAN and LMR.

Objective and method:

The propagation of the acoustic waves depends greatly on the mechanical characteristics of materials. The measurement of the linear properties as celerity or attenuation or nonlinear parameter of the acoustic wave can be used to characterize the thermo-mechanical ageing of polymer materials.

Polyethylene samples will be prepared in order to allow ultrasonic, mechanic and physicochemical characterizations. They will undergo a varied temperature conditions in order to give a large ageing level. The microstructure of thermosetting polymers is composed of two phases: a soft part which is amorphous, and a rigid part which is crystalline. Polyolefin such as polyethylene or polypropylene presents different strain mechanisms as a function of the scale of observation and the mode of mechanical solicitations, i.e. tensile and compression mode. Principal breaking micro-mechanisms dependent on strain rate and direction are the cavitation and the crazing.

Microstructural evolutions of polymer caused by thermo-oxidative ageing and mechanical damages will be monitoring by microscopy and chemical analysis such as differential scanning calorimeter. The results obtained will be compared in order to deduce the thermomechanical degradation and the effect on the durability of polymer.

Firstly the linear ultrasonic parameters, such as celerity and attenuation of the acoustic wave will be measured using a calibrated insertion / substitution 3D spectroscopy method. The study of theses properties will be completed by mechanical and physicochemical parameters analysis. The comparison of the experimental data with theoretical ones based on the

modeling of an acoustic wave propagation in a polymer sample will give a degradation level of the polymer material. This approach will be completed by a characterization of the microstructure based on the acoustic microscopy.

Secondly, the measurements of the nonlinear acoustic parameters will be performed to characterize the presence of micro-cracks in polymer samples to add to linear measured parameters. The nonlinear parameter measurements will be performed by example using shift of the structural resonance frequency in function of the amplitude of solicitation.

The results obtained will open new prospects in order to further understand local damage mechanisms in thermosetting polymer products and to propose a real new device to help dimensioning calculation in complex structures.

References

[1] J. Bustillo, J. Fortineau, G. Gautier and M. Lethiecq, Ultrasonic characterization of porous silicon using a genetic algorithm to solve the inverse problem, NDT&E international, 62,93-98, (2014)

[2] G. Robin, F. Vander Meulen, N. Wilkie-Chancellier, L. Martinez, L. Haumesser, J. Fortineau, P. Griesmar, M. Lethiecq, G. Feuillard. Ultrasonic self-calibrated method applied to monitoring of sol-gel transition Ultrasonics (2012).

[3] Theoretical and experimental study of the electroacoustic behavior of lithium niobate under an initial mechanical stress, Domenjoud, M.; Lematre, M.; Gratton, M.; Lethiecq, M.; Tran-Huu-Hue, L.-P. In: IEEE Transactions on Ultrasonics, Ferroelectrics and Frequency Control, Oct. 2013, vol.60, no.10, pp. 2219-24

[4] Evaluation of the moisture content in phenolic resin via acoustic measurements, J. Fortineau, F. Vander Meulen, L. Haumesser, G. Feuillard, S. Barré and O. Bou Matar, J. Appl. Phys. 100, 074911 (2006)

[5] S. Gauchet, Study of the carbon back structure influence on fatigue strength of HNBR rubber, PhD thesis, University François Rabelais of Tours, (2007)

key words: polymer material, polymer ageing, ultrasonic characterization, mechanical and physicochemical characterization, acoustic propagation modeling

Required profile:

The applicant should have theoretical skills in materials science and in physical acoustics Good bases in signal processing and some experimental capabilities will also be essential.

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