## Instruments for Dynamic Testing of Polymer Materials

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## **Contents**

| 1 | Dynamic Testing of Polymer Materials |   |    |
|---|--------------------------------------|---|----|
|   | 1.1                                  | Instruments for Dynamic Testing of Polymers | 1  |
|   | 1.2                                  | ASTM D5992 and ISO 4664-1                   | 9  |
|   | 1 3                                  | References                                  | 12 |

### Chapter 1

#### **Dynamic Testing of Polymer Materials**

#### 1.1 Instruments for Dynamic Testing of Polymers

There are five (5) main classes of experiments for measurement of viscoelastic behaviour

- 1. Transient measurements: creep and stress relaxation
- 2. Low frequency vibrations: free oscillations methods
- 3. High frequency vibrations: resonance methods
- 4. Forced vibration non-resonance methods
- 5. Wave propagation methods

The frequency scale for the different test methods are shown in Figure (1.1)

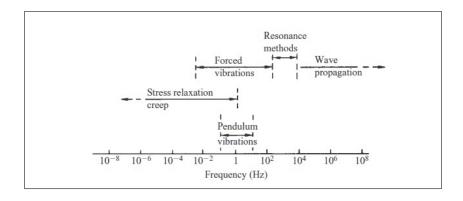


Figure 1.1: Frequency Scale for Different Test Methods

Dynamic characterization of elastomeric materials and polymers requires the use of sophisticated instruments with high fidelity load cells, displacement transducers and strain gauges to understand the deformations taking place in the material under dynamic frequencies. A Rheometer is used to test the dynamic properties during cure. A servo hydraulic fatigue testing machine and dynamic mechanical analyzer (DMA) instruments are the primary material testing instruments used in dynamic characterization of polymers. The sophistication of material testing instruments increase with needs for higher frequencies and higher loads. Strain gauge based and piezoelectric quartz based load cells are used in this instruments to study the load, stress and strain and record the test data.

Along with high quality hardware need also arises for a sophisticated and advanced software to carry out all the calculations. The software that calculates all the dynamic properties also needs to be as sophisticated and advanced as the hardware required to do the test



Figure 1.2: AdvanSES High Frequency and ElectroMechanical Testing Setup

Figure (1.2) shows the AdvanSES servo hydraulic tester used in material testing at high frequencies the servo hydraulic tester is capable of going up to 100 hertz under sine wave definition hydraulic actuator is the primary source of frequency generation in the instrument and the servo valve in the actuator controls the flow of hydraulic fluid into the actuator so as to apply a controlled displacement at controlled frequency. The load cell in the instrument measures the loads generated in the sample under the dynamic frequencies. The servo hydraulic tester is primary primarily used to study static and dynamic stiffness, loss and storage modulus and Tan-delta. Fatigue crack growth propagation of rubber samples can also be tested using a high fps camera integrated with the tester. Elevated temperature testing is also available with the use of a temperature chamber with automatic PID control.



Figure 1.3: AdvanSES High Frequency Testing Setup with Temperature Chamber

High temperature tests (e.g. tensile, compression, flexure and fatigue tests) are used to determine the thermal-elastic behavior, heat resistance, endurance and durability of metallic and polymer materials. Elevated temperatures is combined with mechanical testing, environmental aging, analytical solution methods to develop and provide a comprehensive test protocol to evaluate materials and components.

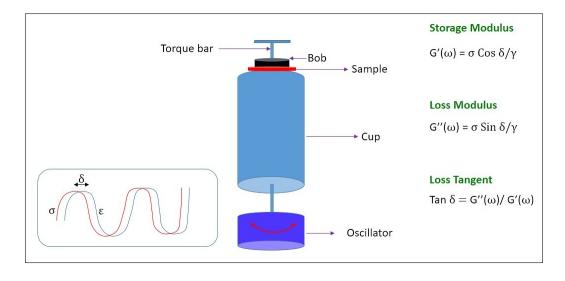


Figure 1.4: Oscillating Rheometer for Dynamic Testing

Figure (1.4) shows a rheometer instrument used for studying the dynamic properties of uncured and cured rubber compounds. The instrument consists of a torque applicator, an oscillator and a load measuring device. The sample is held between the torque bar and the cup. The oscillator supplies an oscillatory motion that is transferred to the sample by the cup. The angular torsional deformations of the sample and the load generated in the instrument are measured using advanced load cell, displacement transducers and rotary encoders. A sine wave is input into the sample and a sine wave is similarly output from the instrument, both the input and output waves are compared to calculate the storage modulus, loss modulus and the tangent delta.

The sample requirements for the rheometer testing are that the samples should be dimensionally stable of rectangular or cylindrical cross section. To test the material a sample is firmly gripped at both ends. the specimen is electromagnetically or servo driven into sinusoidal oscillations of defined amplitude and frequency. The viscoelastic properties of the material makes the torque lag behind the deformation. The lag between the input and output is the phase angle as shown earlier in Figure(??). The observed values for load, phase angle, and geometry constant of the specimen is used to calculate the complex shear modulus G\*, the storage shear modulus G', the loss shear modulus G'', and tan delta.

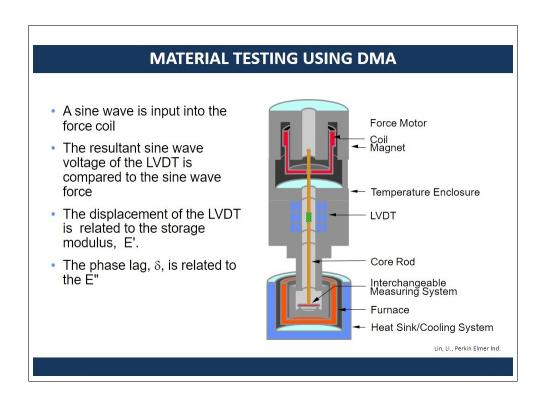


Figure 1.5: Axial Dynamic Mechanical Analyzer with Furnace

Figure (1.5) shows a DMA instrument manufactured buy TA Instruments. As shown in the figure, a force motor with a coil and magnet is used to apply a force and lvdt measures the displacement of the sample. Furnace is provided for elevated and low temperature measurements. The sample is kept inside the furnace and a sine wave is input into the force motor, the resultant sine wave voltage of the lvdt is now compared to the input sine wave and the storage modulus, loss modulus, phase and tan delta are calculated from the test results data.

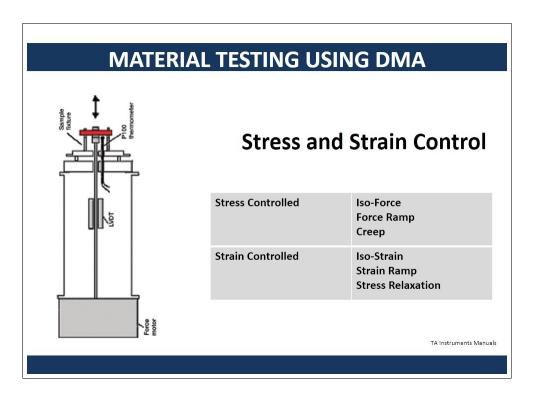


Figure 1.6: Axial Dynamic Mechanical Analyzer with Interchangable Sample Fixtures. Image Courtesy: Perkin Elmer Industries

Figure (1.6) shows a DMA instrument from Perkin Elmer. When compared to the TA Instruments, both the machines have similar performance. Both the instruments can be operated under stress control and strain control. Creep and stress relaxation experiments can be carried out in all the instruments along with frequency sweep, strain sweep and temperature sweep studies

A DMA instrument is very versatile instrument able to apply different deformation modes on the sample. Different deformation modes can be chosen based upon the quality of the material and the material properties under study. Figure(1.7) shows the different deformation modes available for application in a DMA instrument. Single and double cantilever beam, tensile, shear, compression and three point bending tests can be carried out using different kinds of fixtures.

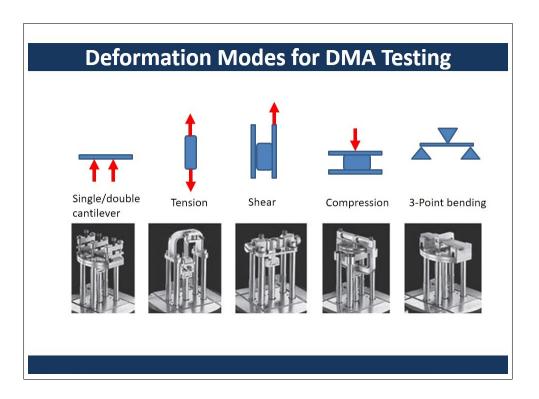


Figure 1.7: Deformation Modes Available in a Typical DMA Machine, Image Courtesy: TA Instruments and Perkin Elmer Industries

Materials such as hard polymers or soft viscoelastic elastomers are ideal materials to be tested on a DMA machine for dynamic properties. The testing conditions and parameters such as applied frequency range, temperature and available sample sizes and shape dictate the machine required for the testing. To carry out frequency and strain sweep studies on automotive and aerospace components, it becomes imperative to use a servo hydraulic machine. While the necessity to study the dynamic property of a material during processing makes it imperative to use a moving or oscillating die rheometer.

The importance of dynamic testing comes from the fact that performance of elastomers and elastomeric products such as engine mounts, suspension bumpers, tire materials etc., cannot be fully predicted by using only traditional methods of static testing. Elastomer tests like hardness, tensile, compression-set, low temperature brittleness, tear resistance tests, ozone resistance etc., are all essentially quality control tests and do not help us understand the performance or the durability of the material under field service conditions. An elastomer is used in all major applications as a dynamic part being able to provide vibra-

tion isolation, sealing, shock resistance, and necessary damping because of its viscoelastic nature. Dynamic testing truly helps us to understand and predict these properties both at the material and component level.

#### 1.2 **ASTM D5992 and ISO 4664-1**

ASTM D5992 covers the methods and process available for determining the dynamic properties of vulcanized natural rubber and synthetic rubber compounds and components. The standard covers the sample shape and size requirements, the test methods, and the procedures to generate the test results data and carry out further subsequent analysis. The methods described are primarily useful over the range of temperatures from cryogenic to 200°C and for frequencies from 0.01 to 100 Hz, as not all instruments and methods will accommodate the entire ranges possible for material behavior.

Figures (1.8 and 1.9) show the results from a frequency sweep test on five (5) different elastomer compounds. Results of Storage modulus and Tan delta are plotted.

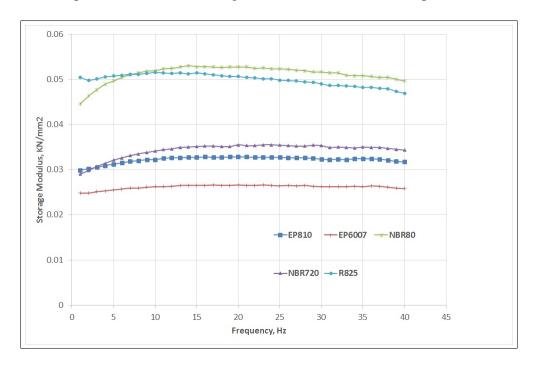


Figure 1.8: Plot of Storage Modulus Vs Frequency from a Frequency Sweep Test

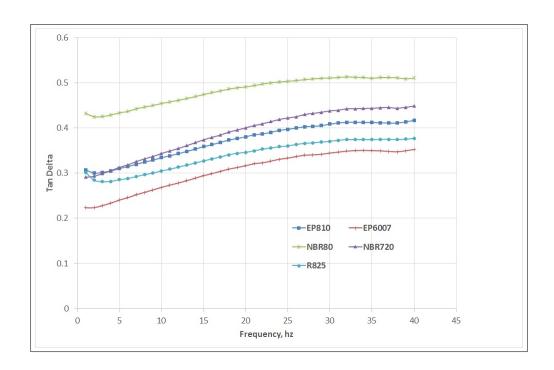


Figure 1.9: Plot of Tan delta Vs Frequency from a Frequency Sweep Test

The frequency sweep tests have been carried out by applying a pre-compression of 10 % and subsequently a displacement amplitude of 1 % has been applied in the positive and negative directions. Apart from tests on cylindrical and square block samples ASTM D5992 recommends the dual lap shear test specimen in rectangular, square and cylindrical shape specimens. Figure(1.10) shows the double lap shear shapes recommended in the standard.

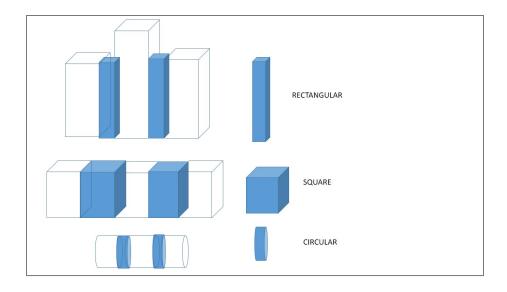


Figure 1.10: Double Lap Shear Shapes

#### 1.3 References

- 1. Sperling, *Introduction to Physical Polymer Science*, Academic Press, 1994.
- 2. Ward et al., Introduction to Mechanical Properties of Solid Polymers, Wiley, 1993.
- 3. Seymour et al. *Introduction to Polymers*, Wiley, 1971.
- 4. Ferry, Viscoelastic Properties of Polymers, Wiley, 1980.
- Goldman, Prediction of Deformation Properties of Polymeric and Composite Materials, ACS, 1994.
- 6. Menczel and Prime, *Thermal Analysis of Polymers*, Wiley, 2009.
- 7. Pete Petroff, Rubber Energy Group Class Notes, 2004.
- 8. ABAQUS Inc., ABAQUS: Theory and Reference Manuals, ABAQUS Inc., RI, 02.
- 9. Attard, M.M., *Finite Strain: Isotropic Hyperelasticity*, International Journal of Solids and Structures, 2003.
- 10. Bathe, K. J., Finite Element Procedures Prentice-Hall, NJ, 96.
- 11. Bergstrom, J. S., and Boyce, M. C., *Mechanical Behavior of Particle Filled Elastomers*, Rubber Chemistry and Technology, Vol. 72, 2000.
- 12. Beatty, M.F., Topics in Finite Elasticity: Hyperelasticity of Rubber, Elastomers and Biological Tissues with Examples, Applied Mechanics Review, Vol. 40, No. 12, 1987.
- 13. Bischoff, J. E., Arruda, E. M., and Grosh, K., *A New Constitutive Model for the Compressibility of Elastomers at Finite Deformations*, Rubber Chemistry and Technology, Vol. 74, 2001.
- 14. Blatz, P. J., *Application of Finite Elasticity Theory to the Behavior of Rubberlike Materials*, Transactions of the Society of Rheology, Vol. 6, 1962.

- Eberhard Meinecke, Effect of Carbon-Black Loading and Crosslink Density on the Heat Build-Up in Elastomers, Rubber Chemistry and Technology, Vol. 64, May 1991.
- 16. Boyce, M. C., and Arruda, E. M., *Constitutive Models of Rubber Elasticity: A Review*, Rubber Chemistry and Technology, Vol. 73, 2000.
- 17. Callister Jr., W. D., *Introduction to Materials Science and Engineering* John Wiley and Sons, NY, 1999.
- 18. Myers, M. A., Chawla, K. K., *Mechanical Behavior of Materials* Prentice Hall, 1998.
- 19. Terrill., E. R. et al., *Modulus-Fatigue Resistance of Silica-filled Tire Tread Formulations* Fall 184th Technical Meeting of the ACS, Rubber Division, 2013.
- Silva, A. J., Berry, N. G., Costa, M. F., Structural and Thermo-mechanical Evaluation of Two Engineering Thermoplastic Polymers in Contact with Ethanol Fuel from Sugarcane Material Research Vol.19 No.1, Jan/Feb 2016.
- 21. Menard, Kevin, P., Dynamic-Mechanical Analysis, CRC Press, Boca Raton, 1999.
- 22. Nielsen, L. E., Mechanical Properties of Polymers, Marcel Dekker, New York, 1974.
- 23. Tschloegl, N. W., *The Theory of Viscoelastic Behaviour*, Academic Press, New York, 1981.
- 24. Leyden, Jerry., *Failure Analysis in Elastomer Technology: Special Topics*, Rubber Division, 2003.
- 25. Baranwal, Krishna., Elastomer Technology: Special Topics, Rubber Division, 2003.
- 26. Srinivas, K., and Pannikottu, A., Material Characterization and FEA of a Novel Compression Stress Relaxation Method to Evaluate Materials for Sealing Applications, 28th Annual Dayton-Cincinnati Aerospace Science Symposium, March 2003.

- 27. Srinivas, K., Systematic Experimental and Computational Mechanics Failure Analysis Methodologies for Polymer Components, ARDL Technical Report, March 2008.
- 28. Dowling, N. E., Mechanical Behavior of Materials, Engineering Methods for Deformation, Fracture and Fatigue Prentice-Hall, NJ, 1999.
- 29. Hertz, D. L. Jr., Designing with Elastomers, Seals Eastern, Inc. 1983.
- 30. Srinivas, K., and Dharaiya, D., *Material And Rheological Characterization For Rapid Prototyping Of Elastomers Components*, American Chemical Society, Rubber Division, 170th Technical Meeting, Cincinnati, 2006.
- 31. Gent, N. A., *Engineering with Rubber: How to Design Rubber Components* Hanser Publishers, NY, 1992.
- 32. Horve, L., Shaft Seals for Dynamic Applications, Marcel Dekker, NY, 1996.
- 33. Pannikottu, A., Hendrickson, K. and Baranwal, K., *Comparison of Dynamic Testing Equipment and Test Methods for Tire Tread Compounds* ARDL Inc, OH, 1996.
- 34. Mars, W. V., and Fatemi, A., *A literature survey on fatigue analysis approaches for rubber*, International Journal of Fatigue, Vol. 24, 2002.
- 35. Belytschko T., Liu K.W, Moran B., *Nonlinear Finite Elements for Continua and Structures*, John Wiley and Sons Ltd, 2000.
- 36. Hasan O.A., Boyce M.C., A constitutive model for the nonlinear viscoelastic viscoelastic bahavior of glassy polymers, Polymer Engineering and Science 35, 1995.
- 37. Haward R.N., Young R.J., *The Physics of Glassy Polymers*, Chapman and Hall, 1997.
- 38. Simo J.C., On a fully three-dimensional finite-strain viscoelastic damage model: for-mulation and computational aspects, Computer Methods in Applied Mechanics and Engineering, Vol. 60, 1987.
- 39. Ogden, R. W. Non-linear Elastic Deformations, Dover Publications, New York, 1997.

- 40. Sasso, M., Palmieri, G., Chiappini, G., and Amodio, D. *Characterization of hypere-lastic rubber-like materials by biaxial and uniaxial stretching tests based on optical methods*, Polymer Testing, Vol. 27, 2008.
- 41. Guo, Z., and L. J. Sluys, *Application of A New Constitutive Model for the Description of Rubber-Like Materials under Monotonic Loading*, International Journal of Solids and Structures, Vol. 43, 2006.
- 42. Kaliske, M., L. Nasdala, and H. Rothert, *On Damage Modeling for Elastic and Viscoelastic Materials at Large Strain. Computers and Structures*, Vol. 79, 2001.
- 43. T.G. Ebbott, R.L. Hohman, J.P. Jeusette, and V. Kerchman., *Tire temperature and rolling resistance prediction with Fnite element analysis*, Tire Science and Technology, Vol. 27, 1999.
- 44. KV Narasimha Rao, R Krishna Kumar, PC Bohara, and R Mukhopadhyay. *A Fnite element algorithm for the prediction of steady-state temperatures of rolling tires* Tire Science and Technology, Vol. 34(3), 2006.
- 45. Menczel., J.D., and Prime, R. B., Thermal Analysis of Polymers, Wiley, 2009.
- 46. Douglas Loy, Class Lectures, University of Arizona.
- 47. Joseph Padovan, Class Lectures, *Tire Design and Performance Prediction*, University of Akron.
- 48. Goldman, Prediction of Deformation Properties of Polymeric and Composite Materials, ACS, 1994.
- 49. Netzsch Instruments, Class Notes and Machine Manuals, 2004.
- 50. T A Instruments, *Class Notes and Machine Manuals*, 2006.
- 51. Perkin Elmer Industries, Class Notes and Machine Manuals, 2007.
- 52. Matsouka, Relaxation Phenomena in Polymers, Hanser, Frankfurt, 1993.

- 53. Danko, D. M., and Svarovsky, J. E., An Application of Mini-Computers for the Determination of Elastomeric Damping Coefficients and Other Properties, SAE Paper No. 730263.
- 54. Silberberg, Melvin., *Dynamic Mechanical Properties of Polymers: A Review*, Plus-Tech Equipment Corporation, Natick, Massachusetts, 1965.
- 55. Lakes, Roderick., Viscoelastic Materials, Cambridge University Press, 2009.