

Dynamic Mechanical Analysis on Polymer Composites – A Review

T.Anand¹, T.N.Sureshkumar², N. Anbarasan³, K. Karthick⁴, V. Mohan Babu⁵

¹Professor, Agni College of Technology, Chennai, Tamilnadu.

²Associate Professor, Agni College of Technology, Chennai, Tamilnadu

^{3,4,5}Student, Agni College of Technology, Chennai, Tamilnadu.

Corresponding Author: T.Anand

Date of Submission: 15-09-2020

Date of Acceptance: 26-09-2020

ABSTRACT: The technique Dynamic Mechanical Analysis (DMA) of polymers and its composites have been discussed in this paper. DMA of Natural Fibre Reinforced Plastics, Thermoplastic Polymers such as Nylon, Polypropylene, Acrylonitrile Butadiene Styrene and Thermosetting Polymers has been discussed in this work. Linear viscoelastic behaviour, storage modulus, loss modulus and frequency analysis of polymeric materials and composites of polymeric materials were explored. Glass transition temperature was also analysed with the use of Differential Scanning Calorimetry (DSC).

The polymer composites showed better results in storage modulus, glass transition temperature and loss modulus etc. The significance of enhanced properties has also been discussed.

KEYWORDS: Dynamic Mechanical Analysis (DMA), Polymer Composites, Storage Modulus, Glass Transition Temperature, Differential Scanning Calorimetry (DSC).

I. INTRODUCTION

Dynamic mechanical analysis is an important technique to measure the material properties when the periodic stress causes the deformation. A variable sinusoidal stress is applied on the materials and the corresponding resultant sinusoidal strain is measured. The phase difference is 0^0 between the sine waves of stress and strain, and then the material is purely elastic. The phase difference is 90^0 for purely viscous materials. In real time applications the mostly all materials including polymers are viscoelastic materials and the phase difference is between the extremes. The amplitudes of stress and strain waves and the phase difference together is used for determining the fundamental material properties like storage modulus, dynamic viscosity, $\tan \delta$, storage and loss compliance and transition temperatures.

Fig. 1 shows the relationship between several parameters such as stress, strain young's modulus and $\tan \delta$.

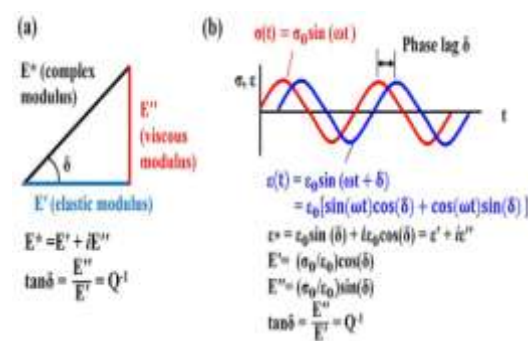


Fig.1. Viscoelasticity and Complex modulus

Most of the Dynamic Mechanical Analysis is carried out with a single frequency, constant strain amplitude and different temperatures. Multiple frequencies are used when the amplitude of deformation is a varying one.

Commercial equipments become more popular and user friendly from sixties. About 1966, Gillham developed the TBA and the modern period of DMA started. In 1971, Starita and Macosko built a DMA that measured normal forces and from this came the Rheometrics Corporation. In 1976, Bohlin also developed a commercial DMA and started Bohlin Rheologia. Torsional geometry was used in both. The early instruments were, regardless of manufacturer, difficult to use, slow, and limited in their ability to process data. In the late seventies, Murayani and Read and Brown wrote books on the uses of DMA for material characterization. Several thermal and rheological instrument companies introduced DMA's in the same time period, and currently most thermal and rheological vendors offer some type of DMA. Polymer Labs offered a dynamic mechanical

thermal analyser DMTA using an axial geometry in the early 1980s. This was soon followed by an instrument from Du Pont. Perkin-Elmer developed a controlled stress analyser based on their thermo mechanical analyser (TMA) technology, which was designed for increased low-end sensitivity. Very easy to use design that formalized both immersion and humidity methods. On the high force end of applications, Mettler Toledo also makes an instrument capable of 40 N loads and both Gabo and MetaVib work exclusively in high load instruments. Small vendors include Hitachi, Bosch, and others. The competition between vendors has led to easier to use, faster, and less expensive instruments. The revolution in computer technology, which has so affected the laboratory, changed the latter, and DMA of all types became more user-friendly as computers and software evolved.

In the automotive industry for interior covering parts both light and stiff, natural fibre reinforced plastics are used. The material should meet the demand requirements related to temperature resistance because the inside temperature of the car surface may be reached up to 100°C under extreme conditions.

In this review paper the dynamic mechanical analysis of polymer composites have been discussed.

II. DYNAMIC MECHANICAL TESTING

In an oscillatory measurement the material is subjected to a sinusoidal stress or strain and the strain or stress response is measured shown in Fig.2. The dynamic mechanical analysis (DMA) analyses both elastic and viscous material response simultaneously. In this type of experiment, a motor is used to either apply a sinusoidal strain or stress to a material (in tension, bending, or shear) and the resulting stress is measured with a force transducer or the resulting strain is measured with a position sensor.

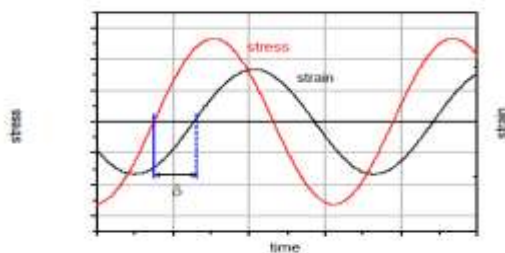


Fig.2. Signals of stress and strain in oscillation test.

The rheological material behaviour can be measured as a function of time, temperature, strain or stress amplitude and frequency. The results obtained provide information about the sample structural properties such as MW, MWD, concentration, cross linking density for polymers or particle/domain size, shape, interface properties, etc. for multiphase fluids. This information is important in product development (formulation) to predict product performance and processing behaviour of new or modified materials.

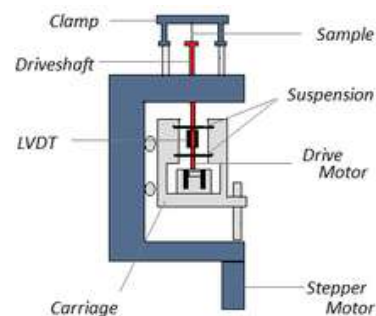


Fig. 3. Dynamic Mechanical Analyser

The Dynamic Mechanical Analyser (DMA) consists of a linear variable differential transformer; it is a kind of displacement sensor. It measures the change in voltage. The clamps hold the samples to be tested. An instrument probe which is moving through a magnetic core, a drive motor and shaft support and a guidance system are used. Fig. 3 shows the schematic arrangement of Dynamic Mechanical Analyser.

The dynamic mechanical analysis is used to study the viscoelastic property of a polymer where a sinusoidal force (stress σ) is applied to a material and the resulting displacement (strain) is measured. For a perfectly elastic solid, the resulting strain and the stress will be perfectly in phase. For a purely viscous fluid, there will be a 90 degree phase lag of strain with respect to stress. Viscoelastic polymers have the characteristics in between where some phase lag will occur during DMA tests.

$$\text{Stress } \sigma = \sigma_0 \sin(\omega t + \delta)$$

$$\text{Strain } \varepsilon = \varepsilon_0 \sin(\omega t)$$

Where t is time, ω is frequency of strain oscillation and δ is phase lag between stress and strain.

III. RESULTS AND DISCUSSION

Thomas Reussman et al.,[1] studied the dynamic mechanical properties of Fiber Reinforced Plastics. Natural Fiber Reinforced Plastics are used in automotive industry due its exceptional quality. The studies were performed at high temperature since the inside car temperature is high. The influence of fiber content, fiber length, fiber matrix adhesion, damping and stiffness of the natural fiber reinforced plastics were evaluated. The shear modulus of the composites is increased with increase in fiber length and fiber content. Flax fiber reinforced polypropylene has higher dynamic stiffness than glass fiber reinforced polypropylene. Greater mechanical damping is also achieved in the natural fiber reinforced composites in the temperature range $0\pm 50^{\circ}\text{C}$.

Study on Viscoelasticity and dynamic mechanical testing was carried out by A.Franc. Oscillation frequency, amplitude of oscillation and temperature of the test are the three parameters controlled in each test. Maxwell model was used to describe the rheological behaviour obtained in oscillation experiment. The shear modulus of a material is independent of amplitude of the strain up to a particular strain applied. Dynamic mechanical analysis enables the characterization of the structural differences available between the materials. It also guides how the materials will be processed[2].

Jo-Yu Wang et al.,[3] investigated the dynamic mechanical analysis of the effect of water on glass bead-epoxy composites. The composites were fabricated by treating and not treating glass beads with a silane coupling agent. The interfacial adhesion of composites ostensibly good or poor and it has been shown in the SEM images.

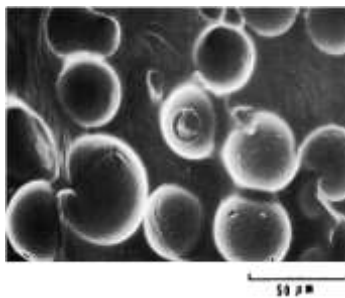


Fig. 4(a) SEM image of untreated composite specimen

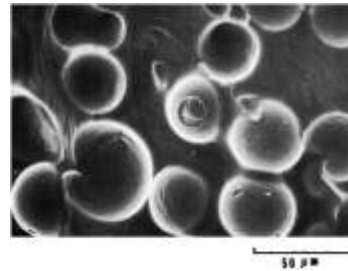


Fig. 4(b) SEM image of silane treated composite specimen

Material properties of dry and wet specimens were measured through dynamic mechanical tests. It is summarized that the loss tangent at T_{∞} is the best parameter for studying interfacial structure in dry composites. It was also identified that the magnitude of loss tangent in the ω -relaxation peak was most sensitive in wet composites.

Jyingsik Jang and Jievn Yi studied the mechanical and thermal analysis on Allylester Polymers and Composites filled with Alumina. Storage modulus and glass transition temperature were determined and compared with thermo-mechanical behaviour of pristine allylester polymers with that of composites filled with alumina. The glass transition temperature is high with more content of alumina. However, the glass transition temperature at 20 phr alumina was higher than at 40 phr. Viscosity will be high with increasing content of alumina. Compared with the AL-10, the similar viscosity of AL-20 helps the maximum cross linking of AL-20 [4].

Dynamic mechanical analysis of FRP composites based on different fiber reinforcements and epoxy resin as the matrix material was investigated by Gosh et al., Three-ply composite laminates prepared from E-glass or N-glass chopped strand mats (CSMs) and jute (J) fabrics as reinforcing agents and amine-cured epoxy resin as the matrix material were subjected to dynamic mechanical thermal analysis at a fixed frequency of 1 Hz over a temperature range of 30–180°C [5].

The volume fraction of fibers are ranging between 0.21 and 0.25. The reinforcing effect for the three fibers is in the order E-glass > N-glass > jute. Glass-reinforced composites show a higher storage modulus (E^*) than that of jute-reinforced composites. The E^* values of glass-jute hybrid composites lie in between those of glass-reinforced and jute-reinforced composites. The trends of variation in the loss modulus, glass transition temperature and damping parameter were interpreted and understood based on the difference

between the physical and chemical properties of three different fibres (E-glass, N-glass and jute).

The coefficient of thermal expansion of the main phase in the composites, the degree of matrix stiffening around the fiber-matrix interface and the extents of matrix softening in the zone next to interface.

Dayal Singh et al., performed the dynamic mechanical studies, impact resistance and SEM studies of ethylene propylene diene terpolymer-poly(vinyl chloride) (EPDM-PVC) and methyl methacrylates grafted EPDM rubber -PVC (graft contents of 4, 13, 21 and 32%) blends were undertaken. All the regions of viscoelasticity were present in the E9 curve, while the E0 curve showed two glass transition temperatures for EPDM-PVC and MMA-g-EPDM-PVC blends, and the T_g increased with increasing graft content, indicating the incompatibility of these blends. The tan δ curve showed three dispersion regions for all blends arising from the α, β, and γ transitions of the molecules. The sharp α transition peak shifted to higher temperatures with increasing concentration of the graft copolymer in the blends. EPDM showed less improvement while a sixfold increase in impact strength was noticed with the grafted EPDM. The scanning electron microscopy micrographs of EPDM-PVC showed less interaction between the phases in comparison to MMA-g-EPDM-PVC blends [6].

Simultaneous Dielectric and Dynamic Mechanical Analysis of Thermosetting Polymers was carried out by Jeffrey Gotro et al., This work presents a new method to measure simultaneously the dielectric loss factor and viscosity of thermosetting polymers during various cure cycles. A microdielectric sensor was mounted in the bottom plate of a parallel plate rheometer [7].

Three types of high performance laminating resins were investigated. During non-isothermal curing, dipole peaks were found to correspond to softening devitrification, the maximum in the loss factor followed the same heating rate dependence as the minimum in the viscosity, and dipole peaks were observed to correlate with vitrification when the cure temperature was below the ultimate glass transition temperature of the resin.

Simultaneous dynamic mechanical analysis and dielectric analysis of polymers was carried out by David D. Shepard et al., Dynamic mechanical analysis (DMA) and dielectric analysis (DEA) measurements were made simultaneously in a Perkin-Elmer DMA 7e connected to a Micromet Instruments Eumetric System III Microdiel ectrometer. Simultaneous DMA-DEA experiments

were performed during the non isothermal cure of an epoxy-glass prepreg. The results showed the techniques to be complementary. DMA provided gel point and vitrification information while DEA provided relative viscosity change, rate of reaction, and was highly sensitive to the end of reaction. The gel and vitrification points identified by DMA were correlated to the DEA data [8].

IV CONCLUSION

In this review paper the dynamic mechanical analysis of polymer composites have been studied and the following conclusions were made.

- To study and characterize the materials, dynamic mechanical analysis is used. The technique is also known as dynamic mechanical spectroscopy.
- Thermoplastic and thermosetting polymers having long molecular chains have good viscoelastic properties, which has the combination characteristics of Newtonian fluids and elastic solids.
- The composition of monomers and the cross linking characteristics add the functionality of the polymer.
- Storage and loss modulus are mainly depends on the material's ability to dissipate stress through heat.

REFERENCES

- [1]. Thomas Reussmann, Klaus-Peter Mieck; Dynamic mechanical analysis of natural fiber reinforced plastics. *Advanced Engineering Materials* 1999, 1, No.2
- [2]. A. Franck, *Viscoelasticity and dynamic mechanical testing*, TA Instruments Germany.
- [3]. Jo-Yu Wang' and Harry J. Ploehn. *Dynamic Mechanical Analysis of the Effect of Water on Glass Bead-Epoxy Composites*. *Journal of Applied Polymer Science*, Vol. 59,345-357 (1996).
- [4]. Jyoncsik Janc and Jieun Yi. *A Dynamic Mechanical Thermal Analysis on Allylester Polymers and Composites Filled with Alumina*. *Journal of Applied Polymer Science*, Vol. 61, 2157-2163 (1996)
- [5]. P. Ghosh, N. R. Bose, B. C. Mitra, S. Das. *Dynamic Mechanical Analysis of FRP Composites Based on Different Fiber Reinforcements and Epoxy Resin as the Matrix Material*. 1997 John Wiley & Sons, Inc. CCC 0021-8995/97/122467-06

- [6]. Dayal Singh, V. P. Malhotra, J. L. Vats The Dynamic Mechanical Analysis, Impact, and Morphological Studies of EPDM–PVC and MMA-g-EPDM–PVC Blends. *Journal of Applied Polymer Science*, Vol. 71, 1959–1968 (1999).
- [7]. Jeffrey Gotro, Michael Yandrasits. Simultaneous Dielectric and Dynamic Mechanical Analysis of Thermosetting Polymers. *Polymer engineering and science*, mid-march 1989, vol. 29, no. 5
- [8]. David D. Shepard, Benjamin Twombly Simultaneous dynamic mechanical analysis and dielectric analysis of polymers. *Thermochimica Acta* 272 (1996) 125-129