Physical Testing of Rubber

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Introduction :

- Rubber has unique and wide range of properties, understanding of which is required to incorporate them in a variety of products and applications, like tires, hose, conveyor belt, bridge bearings, shoe sole, cable, mounts, gaskets, seals, rocket insulation etc. etc.
- Rubber differs considerably from other engineering materials for example, it is the most highly deformable material, exhibiting virtually complete recovery, and it is virtually incompressible with a bulk modulus some thousand times greater than its shear modulus.

REASONS FOR PHYSICAL TESTING :

- Quality control of incoming material
- Quality control of in-process material
- Quality control of finished goods
- Factory Technical Service
- Customer Technical Service
- Preparation of Samples for Sales department
- Development compounding
- Research
- Advertising assistance
- Product performance Testing

CLASSIFICATION OF TESTINGS :

- Testing of new materials
- Evaluation of processing behaviour of rubber compounds
- Testing of vulcanizates
- Testing of finished products

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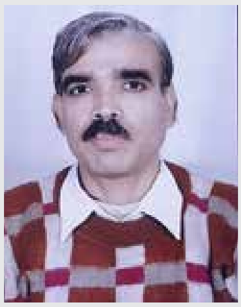


Brief Personal Biography:

- Completed M. Tech Degree in Plastics and Rubber Technology in 1992 from Department of Polymer Science and Technology, University of Calcutta, India
- Received Ph.D degree in Rubber Recycling during December 2007 from Mohan Lal Sukhadia University Udaipur, Rajasthan, India.
- Joined HASETRI in 1992 and worked for more than 23 years.
- Worked as Head (R&D) of Pidilite Industries Ltd. at Dahej, Gujarat for one and half years.
- Currently working as Business Head in Pukhraj Additives LLP for more than 5 years.
- Acting as a faculty member of IRI in PGDIRI – DIRI National Level Crash Course.
- Lead Assessor of National Accreditation Board of Testing and Calibration Laboratories (NABL) in the field of Testing and Proficiency Test Program.
- Fellow Member of Indian Rubber Institute (FIRI) and Institute of Engineers (India) Ltd (FIE).
- 60 numbers of Technical papers and 3 book chapters.

TESTING OF ELASTOMERS:

Viscous properties	Elastic properties
• PRI, Mooney , Rheo, Processing • Rebound Resilience • Deformation set • Relaxation and Creep test • Mechanical loss factor • Temperature increase (Flexometer test)	• Stress at 300% strain • Compression modulus • Shear modulus • Hardness • Dynamic modulus

PROCESSABILITY TESTS

- **PLASTICITY AND RECOVERY TEST**

International Standards :

ASTM D 926 - 93

Significance of testing :

It is a very rapid test and needs very small test piece. In this test the test piece is compressed between two parallel plates under a constant force and the compressed thickness is measured. The extent of compression gives a measure of plasticity of the rubber. This test has been found very useful for routine testing, particularly of raw rubber like Natural Rubber.

Procedure :

ASTM D 926 describes the above test based on William's Plastimeter with plates 4 cm in diameter. The test piece is 2.00 ± 0.02 cm^3 in volume and can conveniently be a cylinder of 16 mm diameter and 10 mm thick. The test piece is preheated for 15 min at a temperature of 100°C and compressed under a force of 5 kgf for around 3 - 10 mins.

Measurement and Calculation :

The thickness of the compressed test piece is measured in mm and this value is multiplied by 100 is quoted as the plasticity number.

After this the test piece is allowed to cool for 1 min and its height is measured. The 'recovery value' is reported as the difference between plasticity number and recovered height, it is intended to be a measure of the elastic component.

The plasticity measurements are made before and after ageing for the determination of Plasticity Retention Index (PRI). Test pieces are aged at 140 ± 0.2°C for 30 ± 0.25 min and cooled to room temperature before making the plasticity measurements. PRI is calculated as follows :

$$\text{PRI} = \frac{\text{aged rapid plasticity number}}{\text{unaged rapid plasticity number}} \times 100$$

Limitations :

Shear rate encountered in rubber processing operation is very high, ~ 10^4 sec^{-1}. But Plasticity tests operate at much lower rate, ~ 0.0025 to 1 sec^{-1}, thus having no correlation with injection moulding.

- **MOONEY VISCOSITY, STRESS RELAXATION, MOONEY SCORCH AND DELTA MOONEY TEST**

International Standards :

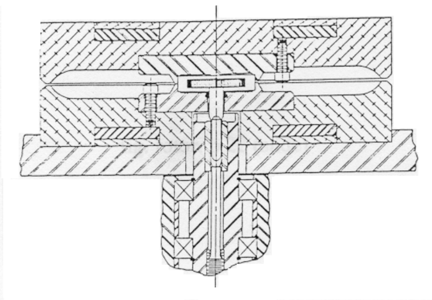
ASTM D 1646-96, SS-ISO 289, ASTM 3346-90

Significance of testing :

These tests are for predicting processing characteristics where correlation between viscosity values and molecular mass is present. Viscoelastic characteristics can be correlated with stress relaxation. It can also predict the rate of cure and incipient cure time during very early stages of vulcanization.

Procedure :

A Mooney viscometer is the most common equipment for these tests. The instrument consists of a motor driven disk within a die cavity formed by two dies maintained at specified conditions of temperature and die closure force. Two rotors are used, larger one with 1.5 inch dia and smaller one is of 1.2 inch dia. The test consists of determining the torque necessary to rotate the disk in the chamber filled with rubber. A number proportional to this torque is taken as an index of the viscosity of the rubber where 8.3 ± 0.02 Nm equals 100 Mooney Units (MU). The rotor is driven at 2 rpm. Generally Mooney Viscosity, Stress Relaxation and Delta Mooney tests are carried out at 100°C and Mooney Scorch at 135°C. For stress relaxation test after completion of the test time (usually 4 min) the test is run for another 2 min while the motor is in rest.



Measuren

Mooney viscosity is usually expressed as, 50 ML_{1+4} @ 100°C,

where 50 is the viscosity in MU, L indicates the Large (standard) rotor, 1 is the pre-heating time and 4 is the test time and 100°C is the test temperature. Other parameters which can be calculated by a Mooney viscometer are Scorch time, Cure index, Optimum cure time, Delta Mooney etc. Typical Stress Relaxation and Mooney cure curves are shown below.

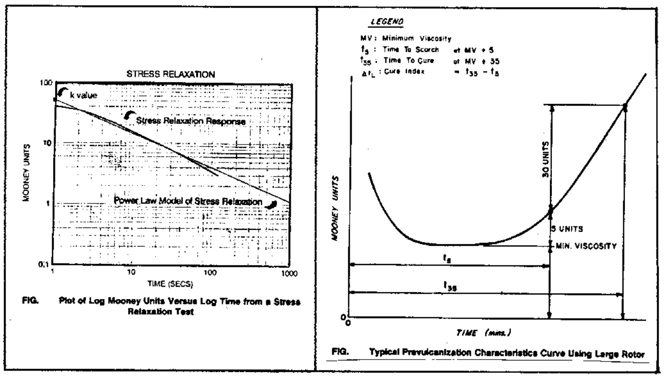


FIG. Plot of Log Mooney Units Versus Log Time from a Stress Relaxation Test

FIG. Typical Prevulcanization Characteristics Curve Using Large Rotor

Limitations :

The shear rates produced are not uniform but range from zero at the center to maximum at the periphery and the average shear rate of this m/c is ~ 2 sec^{-1} which is substantially below than encountered in extrusion and injection moulding.

This test method cannot be used to study complete vulcanization because the continuous rotation of the disk will result in slippage when the specimen reaches a stiff consistency.

- **EXTRUDABILITY**

International Standards :

ASTM D 2230 - 96

Significance of testing :

One objective in the extrusion of rubber compounds is to obtain a smooth extrusion that closely reproduces the contours of the extrusion die. This test method provides for a subjective determination of this under controlled conditions and it simulates to some extent the processing operations of extrusion and injection moulding. Apart from this it has the important advantages that much higher shear rates are possible than normally used other instruments and it is comparable with those encountered in actual processing at shop floor.

Procedure :

In this test rubber is forced through a small cylindrical die under a known pressure and the volume extruded in a given time is measured. Apart from the rate of extrusion it is also possible to visually estimate the quality of the extrusion, for which the cylindrical die could be replaced with one having more complicated cross section (Garvey die) which was designed to show up typical faults that can occur in a mix with poor extrusion characteristics.

Measurement and Calculation :

The Garvey die has an acute angled wedge portion (X). Mixes are rated by assigning a numerical value or score to each of the characteristics like; (a) surface, (b) sharpness of the 'edge' X, (c) the two 'corners' Y and Z, and (d) cross sectional dimensions, i.e., die swell. Rating figures can be seen from the above mentioned ASTM standard.

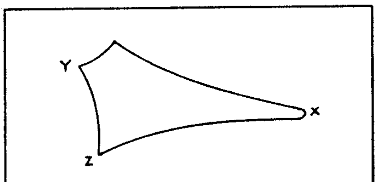


Fig : Cross section of the Garvey Die

Limitations :

As the rating is subjective, it does not lend itself readily to incorporation as a specification requirement. It does not measure other aspects of extrudability such as rate of extrusion or die swell in a quantitative manner. The short period of shearing is insufficient to break down thixotropic structures completely.

- **VULCANIZING CHARACTERISTICS**

International Standards :

ASTM D 2084-95, ASTM D 5289-95, SS-ISO 3417-91, SS-ISO 6502-91

Significance of testing :

This test is used to determine the vulcanization characteristics of vulcanizable rubber compounds and mainly used in quality control process, R&D activity.

The effect of compound changes on the viscosity and scorch characteristics can be determined from the initial portion of the curve while the effect on the rate of the cure and the modulus of the cured compound can be detected in the latter portion of the curve. This is a very rapid test and hence evaluation of the stock prior to final fabrication is easy. A good agreement between cure meter results and tensile modulus measurements make the cure meter an alternative method for controlling the production step and for testing of raw materials like polymers and fillers. Even the batch consistency can be checked easily with this equipment.

Procedure :

In the oscillating disk type rheometer (like, Monsanto R100) a bi-conical disk is embedded in the rubber in a closed cavity and the disk is oscillated through constant angular displacement (1,3,5°). In case of rotorless rheometer (like, Monsanto Moving Die Rheometer, MDR 2000) the sample is sealed within a cavity formed by the upper and the lower dies, which oscillates through and arc of ± 0.5, 1, 3°. In both the cases torque is monitored and if needed can be plot against time scale.

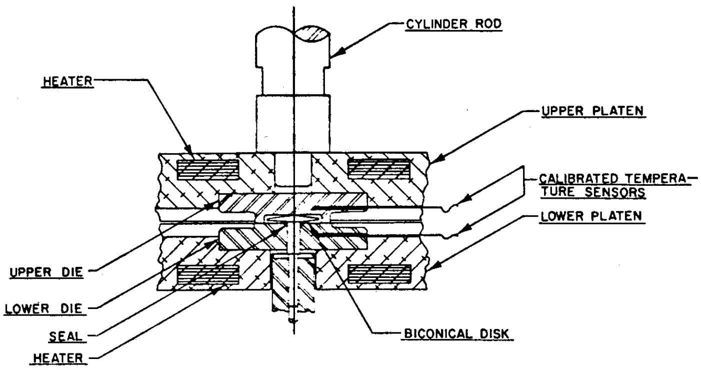


FIG. Cure Meter Assembly

Measurement and Calculation :

Typical curves obtained from an oscillating disk rheometer are shown below.

The information that can be deduced from a typical rheo curve are :

Minimum (M_L) and Maximum torque (M_H); Scorch time (t_s2) - time for two units rise in torque value from the minimum torque; Optimum cure time (T_c90) - time corresponding to the optimum torque value which determines the time of moulding (minutes to ML + 90(MH - ML)/100 torque).

Limitations of ODR over MDR :

The major advantages of MDR over ODR are: high precision m/c, unique die design, less amount of sample requirement, rapid thermal recovery, digital temperature control, integral data system etc.

- **TACK**

International Standards :

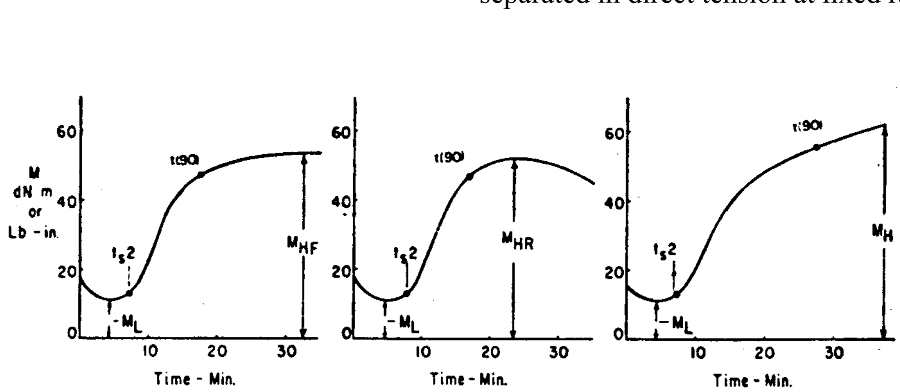
Not Available, generally measured as per instrument manufacturer's instruction manual

Significance of testing :

Tackiness is the property of two pieces of rubber to stick when pressed together and is also known as auto-adhesion. This is an important property as far as the built-up products such as tyres, foot wears, belts are concerned. Solvent based glues generally leave residues which may cause blow holes after moulding, it is therefore desirable to have a better self adhesion in the compound itself, that's why it is an important properties that decides the consolidation of components of a product.

Procedure :

In Monsanto Tel-tack meter two strip test pieces (10 mm) are placed together at right angles to give a known contact area (100 mm^2). After a set contact time (usually 30 sec) under known load (8 ozs), the two test pieces are separated in direct tension at fixed rate of grip travel.



Measurement and Calculation :

Maximum separation force (ozs) and separation time (sec) needed to separate the two specimens is noted from the dial gauge and stop watch respectively. Then 'tack index' is calculated by using the formula below.

$$\text{Tack Index} = \frac{\text{Separation force X (Separation time)}^{1/3}}{\text{Contact force X (Contact time)}^{1/3}}$$

Limitations :

Tack is basically a qualitative test and much depends upon the operator. It is very much sensitive to variations in temperature, pressure, rate of application and removal of pressure and time of contact and surface area of the specimens. So during testing of tack special care is to be taken.

- **SWELLING INDEX**

International Standards :

ASTM D 3616-95

Significance of testing :

Swelling index is a measure of the type of gel. Gel with a low swelling index is referred to as 'hard gel' and usually indicates the presence of material that does not break down readily. A high swelling index normally indicates the presence of a 'loose gel' that may be broken down easily.

Procedure :

A weighed sample is allowed to stand in a suitable solvent till equilibrium swelling (~ 48 hours). The sol or soluble portion is removed from the sample. Sample is taken out from the solvent and the swollen weight is taken. Swelling index is measured by dividing swollen weight with the original weight.

Measurement and Calculation :

Swelling index = Swollen weight / Original weight

Limitations :

Equilibrium swelling is necessary to get informative results. Swelling index can not gives accurate state of cure, it provides only the apparent values.

VULCANIZATE TESTING

INTRODUCTION :

Tests on rubber products can be generally classified into two categories - compound tests and tests on actual products. The former is mainly intended for compound evaluation which is useful in quality control and developmental studies. Tests on actual finished products are designed to give performance characteristics of the product.

Rubber must be tested under carefully prescribed conditions. When products are made to a customer's satisfaction, it is essential that when the customer tests the product he is able to obtain the same results, within narrow limits, as the manufacturer obtained when the goods were produced. Similarly raw materials are tested in the producer's laboratory and subsequently in the customer's laboratory, the results must agree closely.

The international organisation which serves to standardise the test methods for rubber in the American Society for Testing and Materials (ASTM) which is supported by committees made up of industry representatives. In India the standardisation work is carried out by the Bureau of Indian Standards (BIS) formerly known as ISI.

Preparation and Conditioning of the Test Pieces :

Except in case of complete products, a test piece must be formed and properly conditioned before the test can be carried out. The specimens are generally directly moulded or cut and/or buffed or sliced to some particular geometric shape. The specimen preparation and conditioning is done under standard specification to yield a more meaningful and repeatable test data. ASTM D 3182 covers the same.

The properties of rubber depend on its history before test and the atmospheric conditions under which the test was carried out. That is results are affected by the age of the rubber, the conditions such as temperature and humidity under which it was stored, any mechanical deformation before test and humidity at the time of the test. Hence, to produce consistent results it is essential that these factors are controlled within suitable limits. Virtually all test methods specify a conditioning period prior to test, in a standard atmosphere. The standard conditions are $23 \pm 2^{o}C$ and 50 ± 5 % RH.

When both temperature and humidity are controlled, the standard conditioning time is a minimum of 16 hours and where temperature only is controlled, conditioning requires minimum of 3 hours.

- **HARDNESS**

International Standards :

ASTM D 2240-95, ASTM D 1415-88, SS-ISO 7619, SS-ISO 48

Significance of testing :

A hardness measurement is a simple way of obtaining the mechanical properties. The real purpose of running hardness test is to determine the elastic modulus of a rubber under conditions of small strain by determining its resistance to rigid indentor to which a force is applied. The hardness test can also predict the degree of cure. IRHD hardness bears a known relation to Young's modulus also.

Procedure :

In determining the hardness of a piece of rubber, the indentor is pressed down firmly under an applied force. The surface of the specimen must be flat enough for applying uniform load. The sample must be ~ 8 - 10 mm in thickness and have sufficient surface area for the base of the indentor, otherwise wrong value may be obtained.

Measurement and Calculation :

In case of Shore-A durometer the pointer reads at its maximum in a scale of 0 to 100. The internationally accepted standard dead load hardness (IRHD) method covers rubbers in the range of 30 to 85 IRHD. For the normal test a ball indentor of either 2.38 mm or 2.5 mm diameter is used, acting under a total force of 5.53 or 5.7 N respectively. The indentation of the ball is made relative to the surface of the test piece after a fixed time of 30 secs after applying the force.

Limitations :

Hardness is criticized on two counts. (1) the measured values are unreliable because of mechanical considerations and the failure of operators to follow the standard technique of operation; and (2) the characteristic that is measure, surface indentation, rarely bears any relation to the ability of a rubber product to function properly.

- **TENSILE STRESS AND STRAIN INCLUDING TEAR**

International Standards :

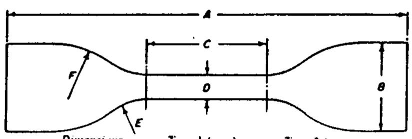
ASTM D 412-92, ASTM D 624-91, SS-ISO 1798, SS-ISO 37, SS-ISO 34-1

Significance of testing :

To the experienced rubber technologist, these tests are useful in many ways - for compound development, for manufacturing control and for determining a compound's susceptibility to deterioration by oil, heat, weathering etc. These tests are almost universally used as a means of determining the effect of various compounding ingredients and are particularly useful when such ingredients affect the rate and state of vulcanization of the composition. The 'retention' properties are much more significant than the absolute values before and after exposure to deteriorating influences. Tear test can predict qualitatively the service performance of the product against any cut.

Procedure :

The determination of tensile properties starts with a piece taken from the sample and includes the preparation of the specimen and testing of the specimen. Specimens may be in the shape of dumbbell, ring or straight piece of uniform cross section. Test pieces are strained at a



Dimensions	Type 1 (mm)	Type 2 (mm)
A	115 minimum	75 minimum
B	25 ± 1	12·5 ± 1
C	33 ± 2	25 ± 1
D	$6{\cdot}0\,^{+0{\cdot}4}_{-0{\cdot}0}$	4 ± 0·1
E	14 ± 1	8 ± 0·5
F	25 ± 2	12·5 ± 1·0

Fig. : Dumb-bell specimen for tensile test

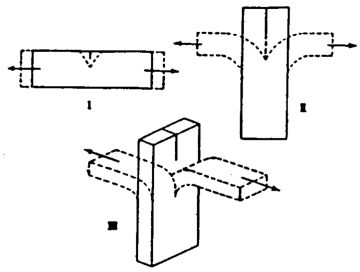


Fig. : Types of tear test pieces
original shape ------- shape after tearing

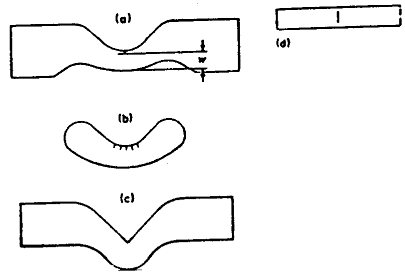


Fig. : Forms of tear test piece
(a) crescent with enlarged ends for gripping w = initial untorn width (b) simple crescent (c) angle (d) Delft

constant rate of traverse and force vs. extension is recorded. Different types of dumbbell and tear test specimens are used for this testing as follows.

Measurement and Calculation :

Tension tests shall be made on power driven machine equipped to produce a uniform rate of grip separation of 500 ± 50 mm/min. The force readings are expressed as stresses by reference to the original cross-sectional area of the test piece. The exact elongation is recorded by the extensometer and the load is monitored by the load cell attached with the Universal Testing Machine (UTM). After completion of the test modulus values at different elongation, tensile strength, elongation at break can be determined or can obtained directly from the computer, if the instrument is equipped with a computer and proper software. Similarly tear results can also be obtained after completion of the test.

Limitations :

Tensile and tear tests have limited significance to the design or application engineer who is interested in determining the suitability of a composition for use in a specific product. They can not be used in design calculations and they bear little relation to a rubber part's ability to perform its function because the rubber products are seldom approach to their ultimate strength or elongation and find no similarity in service life.

- **ABRASION RESISTANCE**

International Standards :

DIN 53516, ASTM D 5963-96, ASTM D 1630-94, ASTM D 2228-88, SS-ISO 4649

Significance of testing :

Abrasion resistance is a performance factor of paramount importance for many rubber products such as tyres, conveyor belts, poser transmission belts, hoses, footwear and floor covering. These tests may be used to estimate the relative abrasion resistance of different vulcanized rubber compounds and are suitable for comparative testing, quality control and R&D work.

Procedure :

There are various type of abraders are available where either loose or solid abradant is used. A loose abrasive powder can be used either in the manner of a shot blasting machine or between two sliding surfaces. Solid abradant consists of abrasive wheels, abrasive papers or cloth and metal knives. Some of the configurations of test piece and the abradant are shown below. The most common used instrument is the Din Abrader which uses a disc test piece of diameter 16 mm and height 6-10 mm in a suitable holder under a load of 10 N which is traversed across a rotating drum of 150 mm diameter with a rotating speed of 40 rpm covered with a sheet of emery paper and provides a abrasion path of 40 m. Running of a standard rubber sample is required to know the abrasiveness of the emery paper.

Measurement and Calculation:

The weight loss is measured after running the experimental sample in the Din abrader. Knowing the specific gravity of the sample and the standard abrasiveness of the emery paper it is possible to calculate the volume loss of the experimental sample using the formula below.

$$\text{Abrasion loss (mm3)} = \frac{\text{Mass loss} \quad X \quad 200}{\text{Sp. gr. X Abrasiveness degree}}$$

Limitations :

Since conditions of abrasive wear in service are complex and vary widely, no direct correlation between these tests and actual performance can be assumed. The common mistake is to think of abrasion resistance as a single entity, like modulus of elasticity. That is not the case. Modulus is a constant and remains the same, no matter what product the compound is used for. Abrasion resistance, on the other hand, is a complex of properties - resilience, stiffness, thermal stability, resistance to cutting and tearing - and different applications require these component properties in widely differing proportions. There are also some purely mechanical testing difficulties which may produce misleading results. For example, an undercured composition (soft and gummy) or one with a wax or oil 'bloom' will appear to have excellent abrasion resistance merely because the abrasive has been clogged or lubricated by material removed from the surface of the specimen.

- **RESILIENCE**

International Standards:

ASTM D 2632-92, ASTM D 1054-91, SS-ISO 466

Significance of testing:

This is a very basic form of dynamic test and is a function of both dynamic modulus and internal friction of a rubber. Some knowledge of these dynamic properties are essential in understanding the performance of those products which in service are subjected to deformations of varying frequency and amplitude. These properties are important in engineering applications of rubbers including spring and dampers.

Procedure :

Rebound resilience can be measured by different type of equipment; one of them is Dunlop Tripsometer. It consists of 42 cm dia and 1.4 cm thick steel disc mounted on bearings and with an out of mass balance in the form a bracket carrying the 4 mm dia indentor attached to its periphery. The scale graduated in degrees to measure the angular displacement of the disc which can be held in any desired position. The test piece shall be a cylindrical block of 8 mm^2 and 4 mm thick. The test specimen shall be conditioned well before the test, specially if the testing is carried out at elevated temperature. After loading the specimen the disc is allowed to strike the specimen from an angle of 45^o and the angular displacement of the rebound is noted.

Measurement and Calculation :

Resilience is approximately related to the loss tangent by,

$R = E_R / E_T = \exp(-\Pi \tan\delta)$; or $E_A / E_R = \Pi \tan\delta$,

where E_R is reflected, E_T is the incident and E_A is the absorbed energy.

In case of Dunlop Tripsometer, the calculation of rebound resilience is as follows.

Rebound Resilience (%) = $(1 - \cos\alpha) / (1 - \cos 45^o)$,

where, α is the rebound angle and 45^o is the initial angle.

Limitations :

The magnitude of the resilience measurement depends upon the size and shape of the pendulum hammer, the size, shape and the stiffness of the rubber specimen and upon the effective height of fall of the pendulum. Besides, the frequency is not of the same order of magnitude as that of many applications involving vibration.

- **HEAT BUILD UP**

International Standards :

ASTM D 623-93, SS-ISO 4666

Significance of testing :

This test describes about the heat rise in a vulcanized rubber cylinder after repeated compressive flexing and allow a fairly accurate prediction of the equilibrium operating temperature of a rubber product like tyre, V belt, in service and operating temperature will have an important effect on its physical properties. The data obtained by this test can be used to estimate relative service quality of different compounds, and are often applicable to R&D studies.

Procedure :

In case of Goodrich Flexometer a definite compressive load (143 psi) is applied to a cylindrical test specimen (having a dia 17.8 ± 0.1 mm and a height of 25 ± 0.15 mm) through a lever system having high inertia, while imposing on the specimen an additional high-frequency (1800 ± 10 rpm) cyclic compression of definite amplitude (generally 4.45 mm). The performance of the machine is checked by studying a control compound.

Measurement and Calculation :

The increase in temperature at the base of the test specimen is measured after an interval (25 min) with a thermocouple to provide a relative indication of the heat generated in flexing the specimen. By comparing the change in height, the 'permanent set' can be calculated which indicates the degree of stiffening or softening of the test specimen. After completion of the test ΔT is calculated, which is commonly known as the 'heat build up'.

Limitations :

Because of wide variation in service conditions, no correlation between this accelerated test and service performance is given or implied. The results of flexometer tests are not suitable for control or classification purposes, nor are they applicable for use in purchase specifications.

- **FATIGUE TEST**

International Standards :

ASTM D 4482-85, ASTM D 813-95, SS-ISO 132

Significance of testing :

These tests are used to determine fatigue life at various extension, compression and repeated distortion by bending. These tests give an idea about the crack initiation behaviour of a rubber vulcanizate and only a very approximate measure of the crack propagation rate. It is possible to have an estimate of the ability of a rubber vulcanizate to resist crack growth of a pierced specimen when subjected to flexing. The information obtained may be useful in predicting the flex-life performance of a compound in active service, such as tyre side wall compounds. These tests are useful in R&D work, especially when an unknown compound or construction is compared in simultaneous test with a construction whose performance has been established in service.

Procedure :

Different types of fatigue test are carried out, like, Fatigue to Failure Test, De Mattia Fatigue test, Ross Fatigue etc. In each case the test specimens are cyclically strained at a fixed frequency and this cyclical straining action is called flexing. As a result of the flexing, cracks usually initiated by naturally occurring flaw, grow and ultimately cause failure which is defined as complete rupture of the test specimen. The number of cycles to failure, i.e., fatigue life, is recorded. Sometime a intentional cut is given on the specimen and cut growth rate is measured after certain interval. The test specimen for De Mattia tester is usually a 6” X 1” moulded strip of thickness 6.4 ± 0.1 mm with a circular groove at the center. For FTFT, the specimen is a dumbbell with two beads at each of the end.

Measurement and Calculation:

In case of FTFT, generally 6 specimens are tested and the 4 closest values are used for calculating the average fatigue life as follows.

JIS average = 0.5A + 0.3B + 0.1 (C + D),
Where, A > B > C > D

In case of De Mattia test generally three test specimens are tested; the data may be reported as the number of cycles required to reach a specified crack length, or as the average rate of crack growth over the entire test period or as the rate of cracking in millimeters (or inches) per kilocycle during a portion of the test.

Limitations :

No exact correlation between these test results and service is implied due to the varied nature of service conditions and are not suited for use in purchase specification requirements because the results from duplicate specimens do not check with sufficient precision.

- **DYNAMIC PROPERTY (FORCED VIBRATION)**

International Standards :

ASTM D 2231-94, SS-ISO 4664

Significance of testing :

Increased usage of rubber in vibrational and damping applications has resulted in a corresponding increased interest in the characterization of rubbers through dynamic mechanical testing. By this test one can measure Storage Modulus, Loss Modulus, Complex Modulus and which has a relation with tyre rolling resistance and in turn fuel economy of the vehicle. Heat Build Up results can also be correlated with the tanδ values. Characterisation of rubber by knowing its glass transition temperature (T_g) is also possible.

Procedure :

Rheovibron Dynamic Viscoelastometer is one of the equipment by which the above test can be carried out. The test can be carried out from sub-ambient temperature to high temperature. A strip of rubber sample (3 X 0.6 X 0.2 cc) is placed in between the chuck and by a servo hydraulic valve it is oscillated at four different frequencies, viz. 3.5, 11, 35 and 110 Hz. If the instrument is attached with a computer with proper software, continuous scanning of the data is possible with respect of temperature; otherwise calculate the dynamic parameters as follows (tanδ comes from direct reading of the scale)

Absolute Modulus, $|E^*|\ (\text{dyne/cm}^2) = \frac{L \times 10^{12}}{8ADS}$

Storage Modulus, $E'\ (\text{dyne/cm}^2) = |E^*| \cos\delta$

Loss Modulus, $E''\ (\text{dyne/cm}^2) = |E^*| \sin\delta$

where, L is the length of the sample (cm); S is the Sectional area (cm2); A is the amplitude factor and D is the dynamic force.

Limitations :

No exact correlation can be made between these test results and the service life due to the varied nature of service conditions.

AIR AGEING IN TEST TUBE ENCLOSURE

International Standards :

ASTM D 865-88, SS-ISO 188

Significance of testing :

Rubber and rubber products must resist the deterioration of physical properties with time caused by oxidative and thermal ageing. Accelerated ageing study can help the compound developers to predict the right composition which can withstand the actual service conditions. The isolation of specimens by the use of individual circulating air test tube enclosures prevents any cross contamination from volatile products and permits a more representative assessment of ageing performance. In these tests, the tensile properties and hardness of specimens are determined before and after exposure in order to establish the extent and rate of deterioration.

Procedure :

The tensile specimens are die out from the slabs and kept a large test tube which will accommodate only limited no. of specimens. Air circulation is properly maintained through out the ageing period. Different temperatures and period can be possible depending upon the application; but generally 70°C is used for natural rubber based compounds and 105°C is used for synthetic rubber based compounds. After the completion of the exposure period, the specimens are taking out and conditioned at room temperature for at least 8 hours before go for hardness and tensile testings.

Measurement and Calculation :

After proper conditioning of the aged specimens, normal hardness and tensile testing are carried out as done for original testing from the same lot of specimens. Results may be reported as either percent loss or percent retained of the physicals. Often a percent is not used, both the original and aged properties are being reported.

$$\text{Retention (\%)} = \frac{\text{Aged values X 100}}{\text{Original values}}$$

Limitations :

Although the different ageing studies help a lot to simulate the service life, but no direct correlation between this accelerated ageing test and natural life of rubber is implied. Since the rate at which deterioration proceeds during the normal life of rubber varies widely, depending on conditions of exposure to heat, light, air, local climate and on the composition and state of cure of the rubber, this accelerated test is comparative only and must be evaluated against the performance of the rubber compounds of which both the natural and accelerated ageing characteristics are known.

- **OZONE RESISTANCE**

International Standards :

ASTM D 1149-91, ASTM D 3395-86, SS-ISO 1431-1,2

Significance of testing :

Ozone is a form of oxygen which occurs naturally in concentrations generally from 2 to 20 parts per 100 million (pphm). Ozone attacks unsaturated rubber, like NR, SBR, NBR very rapidly particularly when they are in stressed condition. To sort out this problem accelerated ozone ageing test is carried out at laboratory. This test is mainly used to differentiate, in a comparative sense, between different degrees of ozone resistance under the limited and specified conditions. These tests basically indicates the protective power of the waxes and other anti-ozonents used in rubber vulcanizates.

Procedure :

Different type of test specimens are used for different mode of test. For static test, 6" X 1" rubber strips are given 20% stretch; for dynamic test 4" X 0.5" rubber strips are given 25% stretch; for bend loop test 3.75" X 1" rubber strips form a loop of varying stretch form 0 - 25%. All of them are pre-conditioned at stretched for 24 hours in stretched condition so that waxes and other anti-ozonants may come out to the surface of the specimens. After the pre-condition time the specimens are exposed to an ozone atmosphere of 50 pphm at a temperature of 40°C for a definite period (~ 120 hr).

Measurement and Calculation :

After a certain interval the samples are checked by magnifying glass and the first appearance of crack is noted. Sometimes a gradation is also given to identify the nature of the crack, as 0 means no crack to 4 means extensive cracks. It is always better to use a reference compound alongwith the experimental one whose values are known.

Limitations :

Although these tests are helpful for comparative purpose, but may not give correlation exactly with the outdoor performance because service performance also depends on other conditions such as rainfall, ambient temperature, etc. A factor which complicates the ozone cracking problem is the effect produced by exposure to sunlight, known is 'crazing' which is different from ozone cracks. Crazing occurs whether or not the specimen is elongated and they are not in uniform direction but extend in all directions. This effect has been shown to be due to oxidation catalyzed by sunlight and rain water soluble products. These can not be judged by the indoor ozone testing.

- **EFFECT OF LIQUIDS**

International Standards :

ASTM D 471-95

Significance of testing :

Certain rubber articles, like, seals, gaskets, hoses, diaphragms may be exposed to oils, greases, fuels during service. Properties of these rubber articles deteriorate during exposure to these liquids, affecting the performance of the rubber part, which can result in partial failure. This test is suitable for specification compliance testing, quality control and R&D work.

Procedure :

This test method provides procedures for exposing test specimens to the influence of liquids under definite conditions of temperature and time. The resulting deterioration is determined by measuring the changes in physical properties, such as stress-strain properties, Hardness and changes in mass, volume and dimension, before and after immersion in test liquids, such as ASTM Oil No. 1 & 5, IRM 902 & 903; ASTM Reference Fuel A to I & K; ASTM Series liquids 101 to 104.

Measurement and Calculation :

$$\Delta Z, \% = \frac{(Z_2 - Z_1) \text{ X } 100}{Z_1}$$

where,

ΔZ = is the change in property, like Tensile Strength, Mass, Volume etc.,

Z_2 = Change in property of the specimen after immersion in test fluids

Z_1 = Original property of the specimen

Limitations :

This test method attends to simulate the service condition through controlled accelerated testing, but may not give any direct correlation with actual part performance, since service condition vary too widely. It yields comparative data on which to base judgement as to expected service quality.

PERMANENT SET

International Standards :

ASTM D 395-89

Significance of testing :

Permanent set tests are intended to measure the ability of rubber compounds to retain elastic properties after prolonged action of compressive and tensile stresses. These tests judge the ability to sustain stress or strain under specified conditions, like gaskets, O-rings, oil seals, tubes, bladders etc.

Procedure:

The test specimen is compressed or elongated to either a deflection (constant strain) or by a specified force (constant stress) and maintain under these condition for a specified time (generally 22 hours) and at a specified temperature (70^{o}C). For constrain strain test, usually 25% strain and for constant stress test, usually 1.8 kN (400 lbf) force is applied. In both the cases the thickness of the specimen (of proper dimension) is measured before and after testing.

To measure the tube / bladder growth, some users' developed test methods are also available, like Exxon's test method, where dumbbell specimens are elongated to 50% with respect to an one inch bench mark in a tensile set apparatus. The stretched specimens are kept in an air ventilated oven at 105^{o}C for 5 hours. After the completion of the ageing, the specimens are conditioned at room temperature at stretched condition for another 2 hours after which the stress is released. Final length is measured after 16 hours.

Measurement and Calculation :

The set at constant stress is calculated as a percentage of the original thickness.

$$C, \% = \frac{(C_1 - C_2) \text{ X } 100}{C_1}$$

where,

C = is the permanent set at constant stress

C_1 = original thickness

C_2 = final thickness

The set as per Exxon method is calculated as follows.

$$S, \% = \frac{(S_3 - S_1) \text{ X } 100}{S_2 - S_1}$$

where,

S = is the permanent set as per Exxon method; S_1 = original length (1 inch);

S_2 = elongated length (1.5 inch); S_3 = recovered length (in inch)

Limitations:

The actual stressing service may involve the maintenance of a definite deflection, the constant application of a known force, or the rapidly repeated deformation and recovery resulting from intermittent compressive forces, its effects as a whole can not be simulated by the above tests.

- **ADHESION**

International Standards :

ASTM D 413 - 82

Significance of testing :

These test methods cover the determination of the adhesion strength between plies of fabric bonded with rubber or the adhesion of the rubber layer in articles made from rubber attached to other material. These are applicable only when the adhered surfaces are approximately plane or uniformly circular as in belting hose, tyre carcasses etc.

These test methods are used to ensure the quality of a product by determining the force per unit width required to separate a rubber layer from a flexible substrate such as fabric, fiber or wire.

Procedure:

Strip Specimen : Type A, 180^o Peel - Measure the width of the strip cut to the nearest 0.22 mm (0.01 in.) and record. Separate the parts to be tested by hand at one end of the strip specimen and at a sufficient distance to permit the jaws of the testing clamp to be attached. Apply a specified or known mass by means of a clamp and mass carrier to the layer of which the adhesion is being determined.

Measurement and Calculation :

Report the adhesion value as separation force per unit width.

Limitations:

When the adhered surfaces contain sharp bends, angles, or other gross irregularities that cannot be avoided in preparing test specimens, then special test methods must be employed for evaluating adhesion.

The acceptable value of adhesion measured will, of course, vary from product to product due to different rubber formulations, flexible substrate and types of products.

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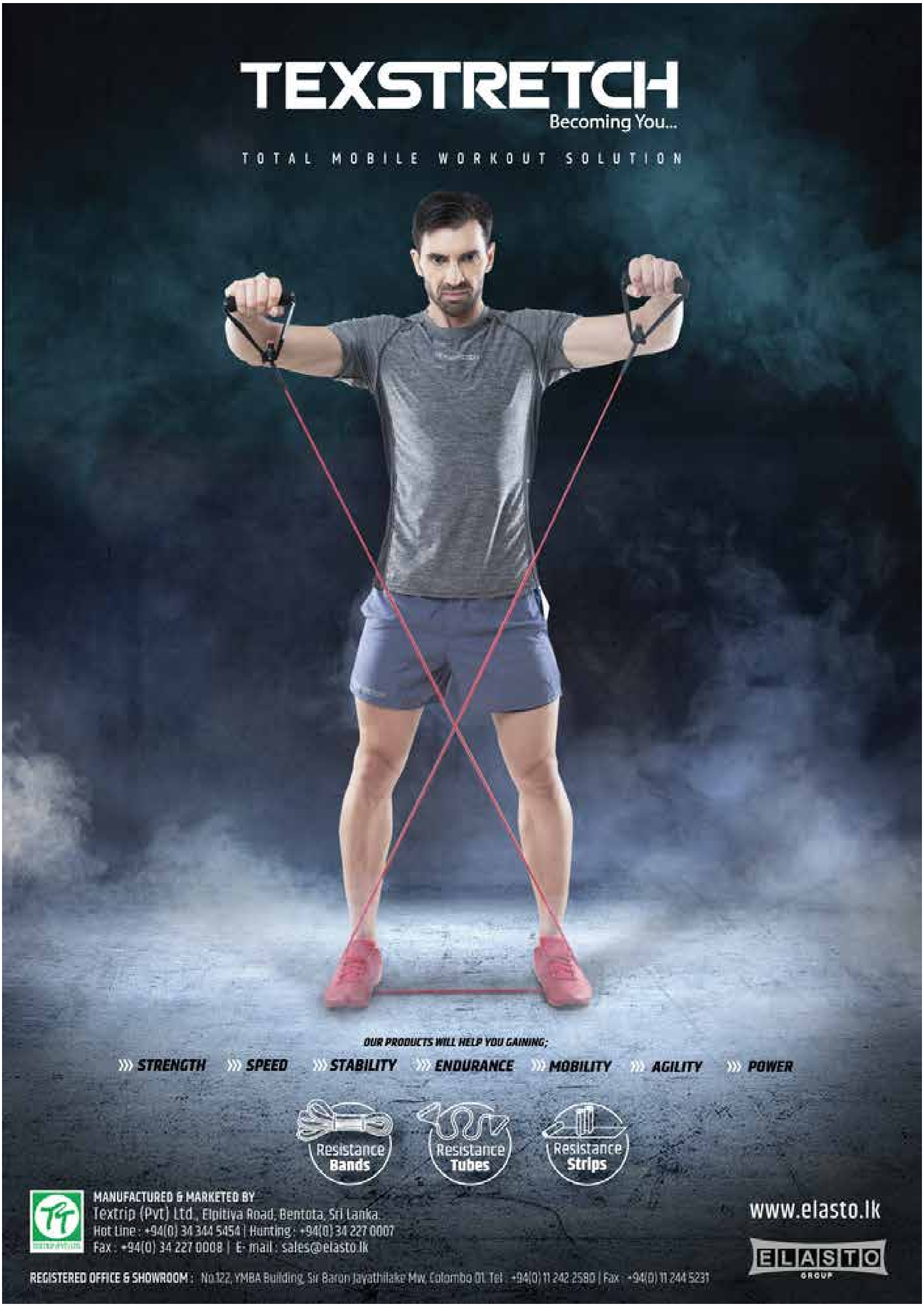
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