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Rubber, vulcanized or thermoplastic — Low temperature testing — General introduction and guide

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Foreword

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The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

Rubber, vulcanized or thermoplastic — Low temperature testing — General introduction and guide

1 Scope

This International Standard provides a general introduction to, and guidance on, the methods of test for low temperature properties of vulcanized and thermoplastic rubbers.

It is intended to provide an understanding of the significance of the various low temperature properties and to assist in the selection of an appropriate test method.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
low temperature test
test to measure any property at a temperature below standard laboratory temperature

3 Types of low temperature test

3.1 General

With reduction of temperature, rubbers become stiffer, until finally becoming hard and brittle, and also recovery from an applied deformation becomes more sluggish. The point of becoming hard and brittle is the glass transition. Any physical test can be made at sub-normal temperatures and, for particular purposes, it might be desirable to follow changes in, for example, tensile strength, dynamic modulus, resilience, or electrical resistivity as the temperature is lowered, depending on what is relevant to service. The glass transition temperature (T_g) is most commonly determined from differential scanning calorimetry (DSC) or dynamic thermal mechanical analysis (DTMA) testing. In this respect, it should be noted that the measured T_g will depend on the frequency of the test used, a “fast” test yielding a higher T_g than a “slow” one. Further information can be found in Reference [6].

Largely for practical convenience, a number of specific low temperature test procedures have evolved for measuring these general trends in behaviour and have been widely standardized.

These low temperature tests can be grouped as follows:

— change in stiffness;

— brittleness point;

— rate of recovery (set and retraction).

In addition, some rubbers, for example natural rubber and polychloroprene, stiffen at low temperatures by partial crystallization. This is a gradual process continuing over many days or weeks and is most rapid at a particular temperature characteristic of each polymer, for example −25 °C for natural rubber. Hence, tests intended to measure the effect of crystallization have to detect changes in stiffness or recovery after periods of “ageing” at a low temperature.

3.2 Change in stiffness

Historically, before the introduction of thermal analysers, it was relatively difficult and expensive to measure tensile or compressive modulus at sub-ambient temperatures. Torsion tests, although rarely used to measure stiffness at ambient temperature, were found to be very convenient for measuring change of stiffness as temperature is lowered. The system standardized for rubber as the Gehman test in ISO 1432 uses a torsion wire to provide the torque to twist a strip test piece. The stiffness is measured as a function of rising temperature. The measurements may be made in a liquid or gaseous environment, either at intervals or with continuous rise of temperature. In principle, these alternatives yield equivalent results.

The normal expression of results is to give the temperatures at which the relative modulus (calculated with respect to that at 23 °C) is 2, 5, 10, and 100. If required, the apparent torsional modulus at any temperature may be calculated but, because it depends on the test conditions, should not be taken as an absolute value.

With modern universal test machines with environmental cabinets and, in particular, dynamic thermal mechanical analysers, it can be argued that essentially the same data could be better obtained using the more commonly used modes of deformation, and which would match data obtained at ambient and elevated temperatures. However, the traditional ISO 1432 method continues to be widely used.

3.3 Brittleness point

A somewhat arbitrary method for determining the point at which the material becomes brittle is described in ISO 812. It operates on the basis of striking a test piece in the form of a simple cantilever with the striker moving at 2 m/s. The result is either pass or fail, failure being any crack or complete separation of the test piece. Various forms of apparatus are possible and the test may be conducted in a liquid or gaseous medium but, in principle, the results are equivalent.

Two procedures are specified: determination of brittleness temperature and determination of 50 % brittleness temperature. The former gives the lowest temperature at which there are no failures, while the latter finds the temperature at which 50 % of test pieces fail. It has been suggested that the 50 % method is more reproducible but might use more test pieces. For specification purposes, the test may be used as a go/no go procedure at one given temperature.

3.4 Recovery tests

The most straightforward way to measure recovery from a deformation at a low temperature would be to use an adaptation of the normal compression set or tension set tests. A test in compression is given in ISO 815-2. Essentially, it is the usual compression set procedure but with the deformed test piece being released and the recovery taking place at the specified low test temperature.

Although simple in principle, the test is practically rather difficult because of the need to unload the test piece and measure its recovery within a low temperature enclosure. Variability can be large and the test is best made with specially designed apparatus.

Traditionally, an alternative approach based on recovery in tension and known as the low temperature retraction test (TR test) has been widely used and is described in ISO 2921. The stretched test pieces are put into the heat transfer medium at a low temperature below the glass transition temperature. The test pieces are then allowed to recover as the temperature is raised. From a graph of percentage retraction against temperature, the temperatures corresponding to 10 %, 30 %, 50 %, and 70 % retraction are noted. This way of expressing the results is similar in principle to the results of the Gehman test for stiffness.

3.5 Crystallization

In principle, any of the low temperature tests can be used to study crystallization effects by holding the test piece at the low temperature for much longer than in the standard tests. A clause suggesting that this can be done is included in, for example, the TR test standard. However, a procedure based on hardness measurement has been standardized as ISO 3387. The hardness of a test piece is measured soon after being place in the low temperature cabinet and after it has been held at the low temperature for a period of time, usually 24 h and 168 h. This procedure has a similar practical difficulty, of making a measurement at low temperature inside an enclosure, as for determining low temperature compression set. One reason for introducing the hardness procedure was because it can be used with unvulcanized compound as well as vulcanized rubber. This would not be feasible with the other standard low temperature tests.

4 Significance of low temperature tests

None of the standard low temperature tests yields absolute values, as the results depend on the particular test conditions. Different conditions can give different results. Consequently, the results can be used as guidance as to performance at low temperature but might not precisely equate to the performance of a particular product. However, in the Gehman and TR tests, the results are essentially a measure of the change in a property, which is less dependent on the conditions.

It is obvious that the different tests measure different aspects of low temperature behaviour and are, hence, complementary. Which test is preferable in a given situation will depend on which aspect or aspects are relevant to service. For example, if remaining ductile is the only consideration then the brittleness test is appropriate. If recovery from deformation, as in a seal, is important then set or TR tests are more relevant. As mentioned earlier, a property other than those in the standardized low temperature tests might be the most appropriate for a particular circumstance.

5 Comparison of tests

Notwithstanding the fact that the various tests measure different aspects of behaviour, the question is often asked as to how the results compare. Published reports on comparisons of methods are discussed in Reference [6]. The indications are that the ranking of compounds is not always the same for all the tests but reasonable correlations can be expected between similar measures (hardness and Gehman modulus) or between measures at the same degree of stiffening [for example brittleness and TR10 or T10 (Gehman)].

Bibliography

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[4] ISO 2921, *Rubber, vulcanized — Determination of low-temperature retraction (TR test)*

[5] ISO 3387, *Rubber — Determination of crystallization effects by hardness measurements*

[6] Brown R.P. *Physical Testing of Rubber*. Springer, 2006

