(*Continued from page* 1)

Members	*Representing*
DR R. N. MEHROTRA	Synthetics and Chemicals Ltd, Bombay
DR B. SURYANARAYANAN (*Alternate*)	
DR W. MILLNS	Indian Rubber Manufacturers Research Association, Thane
SHRI N. NAGARAJAN	Sundram Industries Pvt Ltd, Madurai
SHRI P. VIJAYARAGHAVAN (*Alternate*)	
SHRI S. C. NANDY	Bata India Ltd, Calcutta
SHRI SUNIL SARKAR (*Alternate*)	
SHRI R. R. PANDIT	Bayer (India) Ltd, Bombay
SHRI D. J. BHARUCHA (*Alternate*)	
SHRI K. S. RADHAKRISHNAN	National Rubber Manufacturers Ltd, Calcutta
SHRI R. P. MATHUR (*Alternate*)	
SHRI B. C. SEN	Ministry of Defence (R & D)
SHRI V. BHATTACHARYA (*Alternate*)	
SHRI E. V. THOMAS	Rubber Research Institute of India, Kottayam
DR M. G. KUMARAN (*Alternate*)	
SHRI M. S. SAXENA, Director (P & C)	Director General, ISI (*Ex-officio Member*)

Secretary

SHRI T. R. RAJAGOPALAN
Deputy Director (P & C), ISI

Methods of Testing Vulcanized Rubbers Subcommittee, PCDC 13 : 9

Convener

DR S. N. CHAKRAVARTY	Modi Rubber Ltd, Modipuram

Members

SHRI L. K. MATHUR (*Alternate* to Dr S. N. Chakravarty)	
DR P. S. BHARGAVA	Alkali & Chemical Corporation of India Ltd, Rishra
SHRI N. C. SAMAJDAR (*Alternate*)	
SHRI B. CHAKRAVARTY	Escon Consultants Pvt Ltd, Calcutta
SHRI J. CHATTERJEE	Andrew Yule & Co Ltd, Calcutta
SHRI A. K. BISWAS (*Alternate*)	
DR C. K. DAS	National Rubber Manufacturers Ltd, Calcutta
SHRI R. P. MATHUR (*Alternate*)	
SHRI R. N. P. DUBEY	Directorate General of Technical Development, New Delhi
DR V. R. B. MATHUR (*Alternate*)	
SHRI C. S. INAMDAR	Cosmos India Rubber Works Pvt Ltd, Bombay
SHRI SEN GUPTA (*Alternate*)	
SHRI G. R. KAVISHVAR	Indian Rubber Regenerating Co Ltd, Bombay
SHRI P. S. VIRANI (*Alternate*)	
SHRI S. K. MAHESHWARI	Indian Petrochemicals Corporation Ltd, Vadodara
DR Y. N. SHARMA (*Alternate*)	

(*Continued on page* 17)

Indian Standard

METHODS OF TEST FOR VULCANIZED RUBBERS

PART 14 ADHESION OF RUBBER TO METAL

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 22 March 1984, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 The need for a standard method for the determination of bond strength between rubber and metal has been felt for the standardization of quality in different rubber products like resilient mounting and engine mounting. The reasons for failure of bonds in these articles are rather complicated in service, due to local stress concentration either by a suddenly imposed excessive shock or due to fatigue under different dynamic conditions. However, it was considered essential to formulate a standard method of test under Standard laboratory conditions to provide data for the development and control of rubber compounds and their methods of manufacture.

0.3 Three methods have been prescribed in this standard. Method A covers the procedure for testing the strength of rubber to metal bonds by separation where the rubber part is adhering to a metal surface while Method B covers the procedure for measuring the force required to cause the rupture of a unit of standard dimensions, comprising rubber bonded to two parallel metal plates. Method C which has been included in this revision specifies a method for measuring the static vulcanized adhesion strength of rubber compound where test piece is composed of two conical ends of a rigid material joined by a cylinder of rubber.

0.4 In the preparation of this standard, assistance has been freely drawn from the following publications:

ISO/813-1974 Vulcanized rubber — Determination of adhesion to metal — One plate method. International Organization for Standardization.

ISO/814-1974 Vulcanized rubbers — Determination of adhesion to metal – Two plate method. International Organization for Standardization.

ISO/5600-1979 Rubber — Determination of adhesion to rigid materials using conical shaped parts. International Organization for Standardization.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard covers preparation of test pieces and the procedure of testing the adhesion of rubber to metal where these have been bonded and vulcanized.

1.1.1 These tests may be used after suitable modification for knowing the comparative bond strength of production parts whenever the design permits preparation of suitable test pieces. It is also useful for preparation of test pieces in the laboratory under standard conditions to provide data for the development and control of rubber vulcanizates for rubber-metal bonding purposes. Results obtained with one procedure will not necessarily be identical with those obtained with the other procedure.

2. TERMINOLOGY

2.1 For the purpose of this standard, definitions given in IS : 3434-1965†, shall apply.

3. METHOD A — RUBBER ASSEMBLED TO ONE METAL PLATE

3.1 Outline of the Method — The test consists of measuring the force required to cause separation of a rubber part adhering to a metal surface, the angle of separation and the width and thickness of the rubber being fixed within agreed limits. The angle of separation is 90°.

*Rules for rounding off numerical values (*revised*).
†Glossary of terms for adhesives and pressure sensitive adhesive tapes.

3.2 Apparatus

3.2.1 *Testing Machine* — Any suitable tensile strength testing machine capable of accurately registering maximum force in Newtons obtained during the test and of maintaining the specified constant rate of separation of the jaws of 50 ± 5 mm/minutes.

NOTE — Inertia (pendulum) type dynamometers are apt to give results which differ because of frictional and inertial effects. A low inertia (for example, electronic or optical transducer) type dynamometer gives results which are free from these effects and is, therefore, to be preferred.

3.2.2 *Fixture* — Any suitable fixture for holding the test piece to the upper head of the machine may be used provided the direction of pull to cause separation is at all times, during the test, as nearly perpendicular as possible to the plane of the rubber-to-metal bond, that is, making an angle of 90° with the top holding fixture. A typical fixture is shown in Fig. 1.

NOTE — The tensile force is applied at an angle of 90° to the plane of the rubber-to-metal bond.

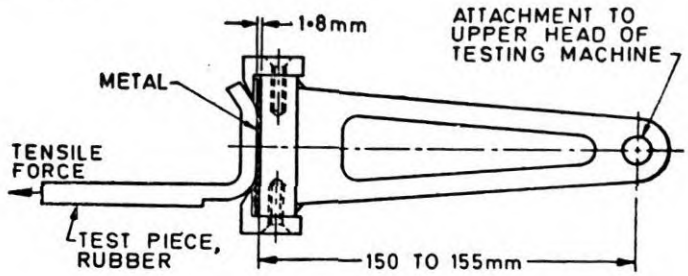


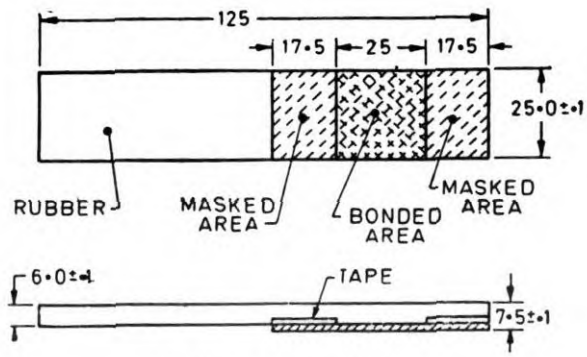
FIG. 1 TEST FIXTURE FOR THE ADHESION TEST

3.2.3 *Grip* — Any suitable grip may be used provided it does not allow the rubber to slip or cause it to rupture.

3.3 Standard Test Piece — The test piece shall consist of a 6 ± 0·1 mm thick strip of rubber 25 ± 0·1 mm wide and 125 mm long adhering to a 25 mm^2 area of the face of the metal. The dimensions of the metal strip shall be 1·5 ± 0·1 mm thick, 25 ± 0·1 mm wide and 60 ± 0·1 mm long. The test piece shall be so prepared that the bonded area of metal to rubber of 25 mm length and 25·0 ± 0·1 mm width is approximately in the middle of the metal strip as shown in Fig. 2

3.4 Preparation of Standard Test Piece

3.4.1 Single or multiple cavity moulds may be used for vulcanization depending on the number of test pieces.



All dimensions in millimetres.

FIG. 2 TEST PIECE FOR METHOD A

3.4.1.1 When the test pieces are to be made using one mix and one type of adhesive system, a mould for several test pieces may be used. The inside mould dimension parallel to the longitudinal axis of the metal strip shall be 125 mm. The dimension along the transverse axis of the metal strip may be altered according to the number of test pieces to be cured at one time. The dimension perpendicular to the longitudinal and transverse axis of the metal strip shall be 7·50 ± 0·05 mm.

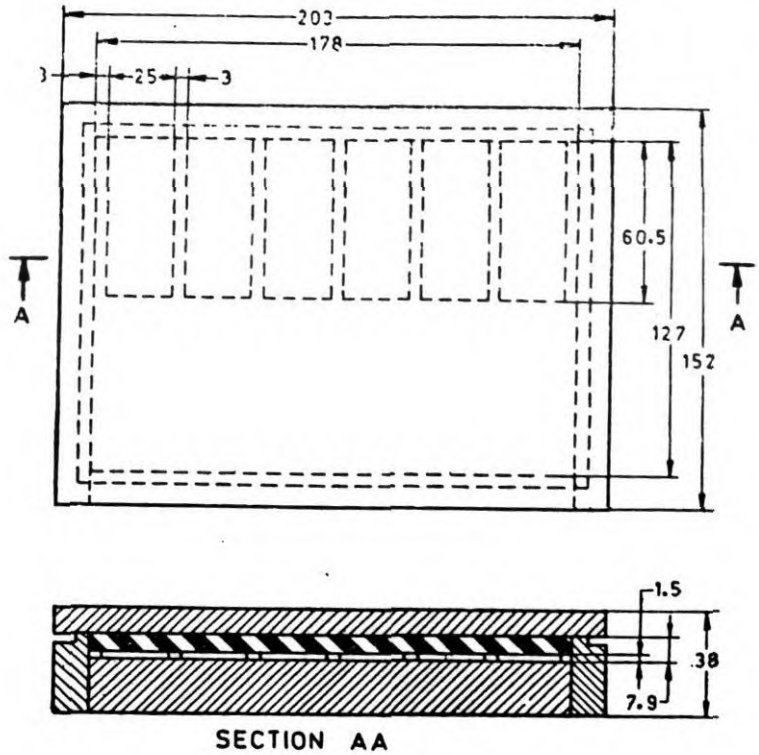
3.4.1.2 When only one test piece is to be made from a given mix, a mould as described in **3.4.1.1** shall be used except that the dimension along the transverse axis of the metal strip shall be restricted to the width of the test piece.

3.4.2 Unvulcanized rubber slabs 8·0 mm thick shall be cut to the dimensions of the required side for the mould (*see* Fig. 3) for suitable mould design so as to provide maximum pressure of the rubber against the metal surface during vulcanization (length 125 mm, width according to the number of test pieces to be vulcanized).

3.4.3 During assembly and vulcanization, great care shall be taken to keep the surfaces to be bonded clean and free from dust, moisture and other foreign material.

3.4.3.1 Rectangular metal strips of the standard dimensions described in **3.3** shall have the area to be bonded, prepared in accordance with the method for securing adhesion that may be under investigation. Both ends shall be marked with pressure sensitive tape so that the area described in **3.3** is available for adhesion.

3.4.3.2 The rubber surface to be bonded shall be solvent washed or treated by a method agreed to between the concerned parties.



All dimensions in millimetres.

FIG. 3 ADHESION TEST SPECIMEN MOULD

3.4.3.3 The metal strips and rubber slabs shall then be assembled for vulcanization. When more than one test piece is prepared at a time, the metal strips shall be placed approximately 3 mm apart to allow for separation of the test pieces. The assembly shall then be placed in the mould with the metal strips at the bottom. Vulcanization shall be carried out by heating the mould under pressure for a definite time at a controlled temperature in a suitable vulcanizing press. The time and temperature of vulcanization shall be as agreed to between the concerned parties. At the conclusion of the cure, care shall be taken in removing the test pieces from the mould to avoid subjecting the bonded surfaces to stress before the test pieces have cooled.

3.4.4 When more than one test piece is vulcanized at one time, the test pieces shall be separated from each other in preparation for testing. This shall be done by cutting with scissors, handknife or other suitable equipment. The edges of the test pieces may then be buffed on a belt sander to bring the edge of the rubber flash to the edge of the edge strip. Care shall be taken not to overheat the metal parts or the rubber and not to reduce the width of the test piece beyond the tolerance allowed.

3.5 Conditioning the Test Piece — The test piece shall be conditioned for at least 16 hours at 27°C ± 2°C. The time between vulcanization and test shall not be more than 6 days. Use the same temperature throughout any one test or series of tests.

3.6 Procedure — Place the test piece symmetrically in the fixture shown in Fig. 1 with the separating edge towards the operator. Before the load is applied, strip the rubber from the metal plate for a distance of approximately 1·5 mm by using a sharp knife. Then place the tab so formed in the grip, which is then moved at the rate of 50 ± 5 mm/min until separation is complete. Record the maximum force required to cause separation over the distance of 25 mm. Repeat this experiment with four test pieces.

NOTE — An autographic recording of the adhesion value over the full length of the piece may also be taken. During the test the operator shall cut the rubber back to the metal whenever the rubber stock tends to tear.

3.7 Expression of Results — Express the adhesion value in newtons per millimetre of width.

3.7.1 *Adhesion Failure Symbols* — The adhesion failures are denoted by symbols, which are defined as given below:

a) R Failure in the rubber;
b) RC Failure in the rubber/cover cement inter face;
c) CP Failure at the cover cement/prime cement inter face; and
d) M Failure at the metal/prime cement inter face.

3.8 Report — The following shall be included in the report:

a) All four test results in accordance with **3.7**;
b) A description of the type of failure in accordance with **3.7.1**, with expression of percentage failure of each type present;
c) A description of the test piece including the method of securing the adhesion;

d) Date of vulcanization;

e) Date of test;

f) Time and temperature of vulcanization; and

g) Temperature of test.

4. METHOD B — RUBBER ASSEMBLED BETWEEN TWO METAL PLATES

4.1 Outline of the Method

4.1.1 This method covers the procedure for testing the adhesive strength of rubber-to-metal bonds where the rubber is assembled between two parallel metal plates.

4.1.2 The test consists of measuring the force required to cause the rupture of a unit of standard dimensions comprising rubber bonded to two parallel metal plates, the direction of force being at 90° to the bonding surface.

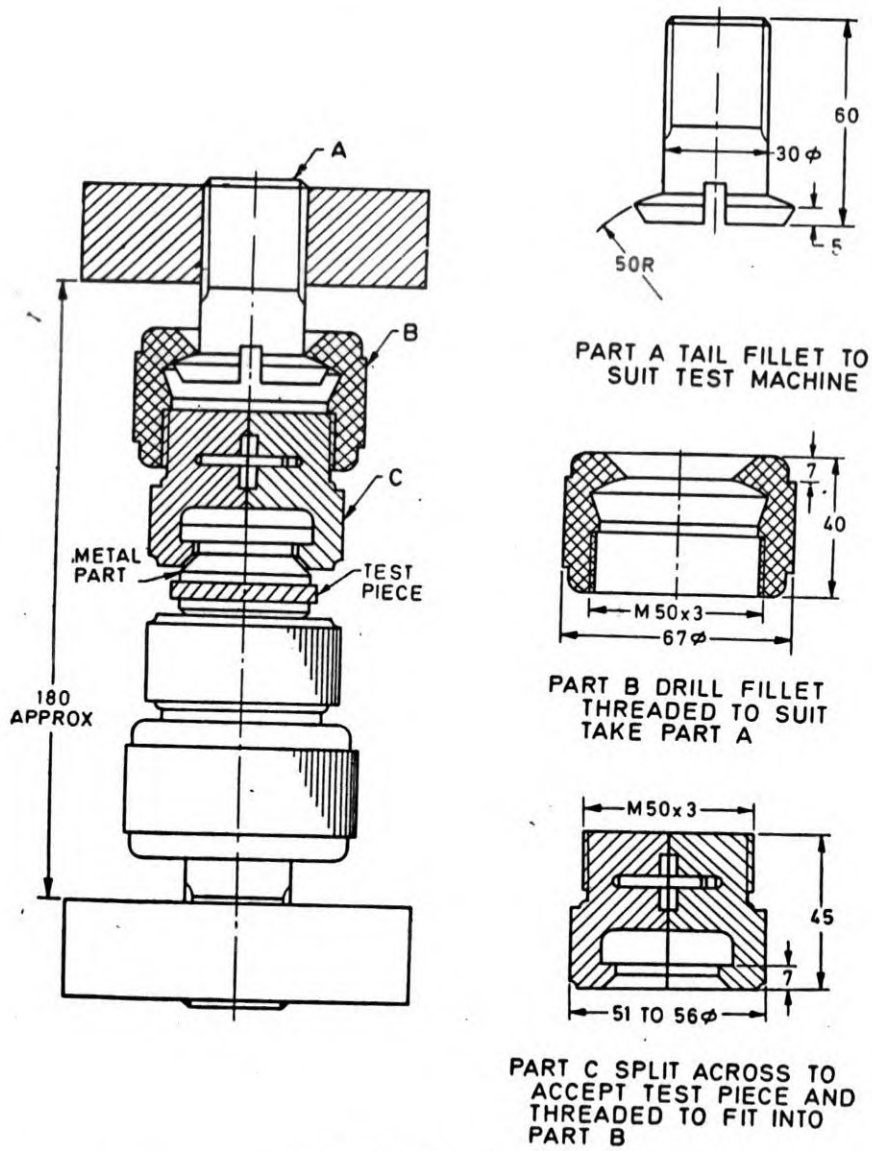
4.2 Apparatus

4.2.1 *Tensile Strength Testing Machine* — Any suitable tensile strength testing machine capable of accurately registering maximum force in newtons obtained during the test and of maintaining the specified constant rate of separation of the jaws of 25 ± 5 mm/min.

NOTE — Inertia (pendulum) type dynamometers are apt to give results which differ because of frictional and inertial effects. A low inertia (for example, electronic or optical transducer) type dynamometer gives results which are free from these effects and is, therefore, to be preferred.

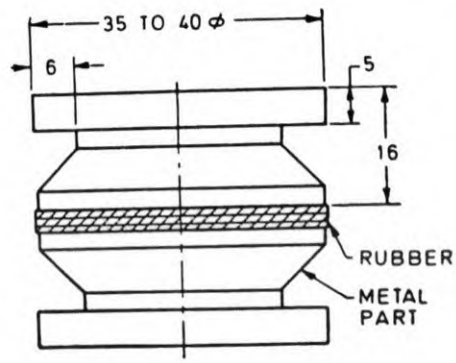
4.2.2 *Fixture* — The fixture for holding the test pieces in the testing machine shall permit accurate centering of the applied load during the test (*see* Fig. 4).

4.3 Test Piece — The test piece shall consist of a rubber cylinder 3·0 ± 0·2 mm thick and of any diameter between 35 to 40 mm with a tolerance of 0·1 mm between different test pieces having its circular ends bonded to the faces of two metal plates of equal diameter. The diameter of the metal plate shall be approximately 0·08 mm less than that of rubber cylinder. The thickness of the metal plates shall be not less than 9 mm. A typical test piece is shown in Fig. 5.



All dimensions in millimetres.

FIG. 4 FIXTURE FOR HOLDING RUBBER TO METAL BONDED TEST PIECES



All dimensions in millimetres.

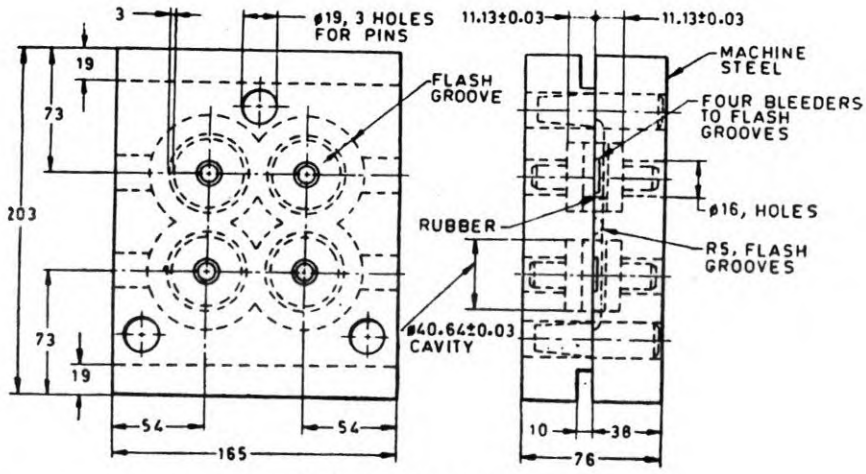
FIG. 5 TEST PIECE FOR METHOD B

4.4 Preparation of Test Pieces — Prepare the test pieces as follows:

Prepare circular metal parts of the standard dimension from rolled carbon steel bar. Treat the smooth metal parts with the adhesion system agreed to between the concerned parties. Cut unvulcanized rubber discs using a circular die of such size that a limited amount of flash is obtained on moulding. Treat the surface of the rubber to be bonded to the metal by the method agreed to between the concerned parties. Assemble the rubber discs and metal pieces for vulcanization in the mould. A typical mould is shown in Fig. 6. Construct the mould so that the rubber projects beyond the edges of the metal end pieces by approximately 0·04 mm in order to prevent tearing of the rubber by the edge of the metal during test.

NOTE — Other metals may be used if the parts are in conformity with essential dimensions.

During the preparation of the test piece, take care to keep the exposed surfaces of the rubber and metal free from dust, moisture and foreign matter. Do not touch the surface by hand during assembly. Carry out the vulcanization by heating in the mould for a definite time at a controlled temperature under pressure in a suitable vulcanizing press. Adjust the time and temperature of cure for the rubber compound used in accordance with the system agreed to between the concerned parties. At the conclusion of the cure, take care in removing the test pieces from the mould to avoid subjecting the bonded surfaces to undue stress before the test pieces have cooled.



All dimensions in millimetres

FIG. 6 DETAILED DRAWING OF A MOULD FOR ADHESION TEST PIECES

4.5 Conditioning the Test Piece — The test pieces shall be conditioned as specified in **3.5**.

4.6 Procedure — Mount the test piece in the testing machine. Carefully centre and adjust the test piece so that the tension is uniformly distributed in the cross section during the test. Apply tension by separating the jaws at a rate of separation of 25 ± 5 mm/min, until the test piece breaks. Record the maximum force. Repeat the test with four test pieces.

4.7 Expression of Results — The adhesion shall be calculated by dividing the maximum force by the cross-sectional area of the test piece. It shall be expressed in meganewtons per square metre.

4.8 Report — The report shall include the following:

a) All four results calculated in accordance with **4.7**;

b) A description of type of failure in accordance with **3.7.1** with expression of percentage failure of each type present;

c) A description of the method of securing the adhesion, and whether the test piece was prepared in the laboratory or taken from a production line;

d) Date of vulcanization;

e) Date of test;

f) Time and temperature of vulcanization;

g) Temperature of test; and

h) Metal used, if other than the specified steel.

5. METHOD C — MEASURING ADHESION OF RUBBER TO METAL WITH A CONICAL SPECIMEN

5.1 Outline of the Method

5.1.1 The test consists in measuring the force required to cause the rupture of a test piece of standard dimensions, comprising a cylindrical rubber body bonded to two conical metal parts. The particular geometry of the specimen produces, in most cases, an interfacial failure between rubber and conical parts, because of a stress concentration at the tip of the cones.

5.1.2 The adhesion is obtained by a bonding system which may include not only the metal and the rubber compound, but other elements such as thin alloy coatings or chemical treatments of metal parts and either a single cement or both primer and cover cements. The bonding system for preparing the test pieces should be adequately specified by the user.

5.1.3 The method is designed primarily to apply to test pieces prepared in the laboratory under standard conditions in order to provide data for development and control of bonding systems and their components, such as cements or other compounds, and of methods of manufacture.

5.2 Apparatus

5.2.1 *Testing Machine* — capable of recording within 2 percent the maximum force obtained during the test, and of maintaining the specified constant rate of separation of the jaws.

NOTE — Inertia (pendulum) type dynamometres are apt to give results which differ because of frictional and inertial effects. A low-inertia type dynamometer (for example, using electronic or optical transducer) gives results which are free from this effect, and is, therefore, to be preferred.

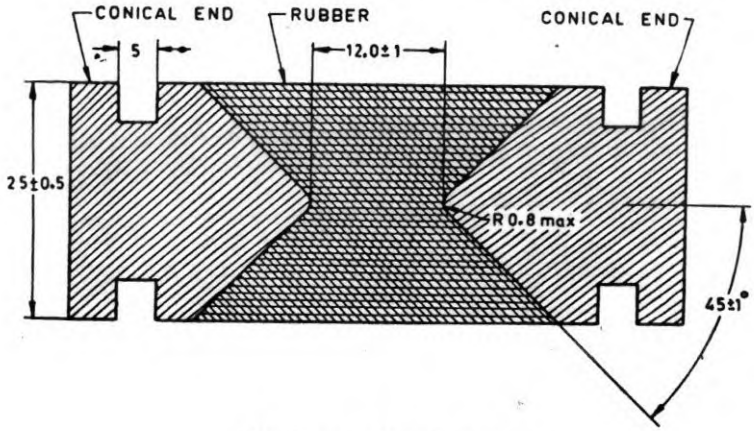
5.2.2 *Grip* — For holding the test pieces in the testing machine which permit accurate centring of the applied load during the test.

5.3 Test Piece

5.3.1 *Form and Dimensions* — The standard test piece (*see* Fig. 7) is formed by two cylindrical metal parts terminated by opposite conical ends, and cylinder of rubber bonded to the conical ends. The diameter

of this cylinder and of the cylindrical portion of the metal parts shall be 25 ± 0·5 mm. The distance between the tips of the conical ends shall be 12 ± 1 mm; the half-angle of the cone vertex shall be 45 ± 1° and the tip shall not be rounded to a radius greater than 0·8 mm.

5.3.1.1 The cylindrical portion of each metal part shall be not less than 5 mm in length and shall be terminated so as to match with the holding grips (*see* **5.2.2**) of the testing machine (**5.2.1**).



All dimensions in millimetres.

FIG. 7 STANDARD TEST PIECE

5.4 Materials

5.4.1 The materials used shall conform to the specifications for the bonding system to be investigated. If no specification is given for the material for the metal parts, they shall be made from low carbon steel bar and their conical ends shall be grit-blasted.

5.5 Preparation of Test Pieces

5.5.1 Clean the surface of the conical ends or treat in accordance with the adhesion system under investigation and, if so specified, coat with primer and/or cover cement. Spread the adhesive coating over the conical area only.

5.5.2 During the preparation of the test piece, take great care when handling the materials to keep the bonding surfaces of the rubber and metal parts free from dust, moisture and foreign matter. Do not touch the treated conical surfaces by hand during assembly.

5.5.3 Vulcanize the test pieces in a suitable transfer mould, properly insulated, provided with heaters and compression devices. Place the metal parts and the rubber compound in the pre-heated mould for vulcanization. Use sufficient unvulcanized compound to fill the pot and provide some excess after filling the mould cavities.

NOTE — The mould design should take account of the fact that machining the rigid parts for re-use will gradually reduce their size.

5.5.4 Carry out the vulcanization under the specified conditions of time, temperature and pressure. At the conclusion of the cure, take great care when removing the test pieces from the mould to avoid subjecting the bonded surfaces to undue stress before the test pieces have cooled.

5.6 Conditioning of Test Pieces — The test piece shall be conditioned for at least 16 hours at 27°C ± 2°C. The time between vulcanization and test shall not be more than 6 days. Use the same temperature throughout any one test or series of tests.

5.7 Procedure

5.7.1 Mount the test piece in the grips (*see* **5.2.2**) of the testing machine (*see* **5.2.1**). Take extreme care in centring and adjusting the test piece so that the tension is symmetrically distributed in the cross-section during the test.

5.7.2 Apply the tension by separating the grips at a constant rate of 50 ± 5 mm/min until the test piece breaks. Record and note that maximum force.

5.7.3 Recover the broken test pieces and examine the failure surfaces.

5.8 Expression of Results — Express the adhesion value, in newtons, required to produce failure. In cases where the failure is in the rubber bulk, the adhesion value is recognised as being higher than that recorded.

5.8.1 *Type of Adhesion Failure* — Express the type of adhesion failure, as determined by examination of broken test pieces, by one or more of the following symbols:

a) R failure in the rubber bulk,

b) RC failure in the rubber cover cement interface,

c) CP failure at the cover cement/prime cement interface, and

d) M failure at the metal/prime cement interface.

Each symbol shall be followed by the percentage of the conical surface involved in that type of failure, estimated to the nearest 5 percent.

NOTE — The estimated percentage of the various types of failure may be expressed as in the following examples:

R — 50, RC — 50 means that roughly 50 percent of the area showed failure in the rubber and the other 50 percent showed failure at the rubber/cover cement interface.

R — 25, RC — 25, M — 50, means three types of failure present, with the M indicating 50 percent failure at the metal/primer cement interface.

5.9 Test Report — The test report shall include the following:

a) a description of the adhesion system used, including materials, treatments and rubber cure. If the materials are of undisclosed composition, sufficient references shall be given to identify them;

b) date of test piece preparation and testing;

c) temperature of test;

d) type of dynamometer used;

e) adhesion values for each test piece, in newtons;

f) type of failure for each test piece, expressed as in **5.8.1**;

g) time and temperature of vulcanization; and

h) metal used, if other than the specified steel.

5.10 Salvaging of Bonded Metal Parts — Bonded metal parts may be salvaged by the usual burning or chemical stripping techniques. Mechanical or chemical surface treatments may be used to re-establish a clean bonding surface. The sharpness of the conical tip may be reduced during salvaging; this affects reproducibility of the test results, and care must be taken to re-establish the sharpness of the cone to a radius of 0·8 mm or less.

(*Continued from page* 2)

Members	*Representing*
DR S. P. MANIK	Research, Designs & Standards Organization Lucknow
SHRI G. DORAISWAMY (*Alternate*)	
DR R. N. MEHROTRA	Synthetics and Chemicals Ltd, Bombay
DR B. SURYANARAYANAN (*Alternate*)	
DR W. MILLNS	Indian Rubber Manufacturers Research Association, Thane
DR M. S. BANERJI (*Alternate*)	
SHRI R. R. PANDIT	Bayer (India) Ltd, Bombay
SHRI N. D. DESAI (*Alternate*)	
SHRI V. D. PENDSE	Swastik Rubber Products Ltd, Pune
SHRI S. V. TATHAWADKAR (*Alternate*)	
SHRI C. R. RAO	National Test House, Calcutta
SHRI V. R. RAO	Sundaram Industries Ltd, Madurai
SHRI K. C. MADHUSUDHANAN (*Alternate*)	
SHRI S. SARKAR	Dunlop India Ltd, Calcutta
SHRI J. C. BOSE (*Alternate*)	
SHRI A. SEN	Bata India Ltd, Calcutta
SHRI D. BANERJEE (*Alternate*)	
SHRI B. C. SEN	Ministry of Defence (DGI)
SHRI P. L. NAG (*Alternate*)	
SHRI E. V. THOMAS	Rubber Research Institute of India, Kottayam
SHRI K. KOCHAPAN NAIR (*Alternate*)	