
**Plain bearings — Dynamic adhesion
test method for DLC coated parts
under lubricated condition**

*Paliers lisses - Méthode d'essai d'adhérence dynamique sur des
composants revêtus de DLC dans des conditions de lubrification*

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 123, *Plain bearings*, Subcommittee SC 7, *Special types of plain bearings*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Reducing the mechanical loss in engines is one of the effective ways to improve the fuel efficiency of automobiles. To reduce the friction coefficient between engine parts, diamond-like carbon (DLC) coatings can be applied for plain bearings as well as other sliding parts such as valve lifters and piston rings.

Since DLC hard coatings have high internal stress due to its coating process under high temperature and the difference of the coefficient of thermal expansion between DLC and engine parts, the quality control of the adhesion strength between coatings and base materials of specific parts after the coating process is very important. As there is no appropriate official test, automobile manufacturers or part suppliers must validate the adhesion strength by using a specially designed test facility using actual engine parts. In order to further spread the DLC coatings to mechanical parts of engines in the future, a test method can be used to easily evaluate the adhesion strength of the coatings.

The purpose of this document is to provide a test method for evaluating adhesion strength of DLC coatings. The test method specified in this document is a test apparatus equipped with a simple ball-on-disk that has a correlation with conventional engine evaluation results and a relatively inexpensive acoustic emission measurement machine without using actual parts.

The positioning of this test method in the process of parts evaluation performed by automobile manufacturers or part suppliers is shown in [Annex A](#). The correlation between results obtained by the test method specified in this document and results obtained by the test method using conventional engine parts is described in [Annex B](#).

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Plain bearings — Dynamic adhesion test method for DLC coated parts under lubricated condition

1 Scope

This document specifies a procedure to measure the adhesion strength of diamond-like carbon (DLC) coatings specified in ISO 20523 by detecting acoustic emission signals using ball-on-disk method under the lubricated condition. DLC is normally coated on metal or ceramic.

The test results are not applicable when the DLC coated parts operate in an unlubricated environment.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 683-17, *Heat-treated steels, alloy steels and free-cutting steels — Part 17: Ball and roller bearing steels*

ISO 1101, *Geometrical product specifications (GPS) — Geometrical tolerancing — Tolerances of form, orientation, location and run-out*

ISO 21920-2, *Geometrical product specifications (GPS) — Surface texture: Profile — Part 2: Terms, definitions and surface texture parameters*

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

ball-on-disk method

test method in which the sliding contact between fixed balls and rotating disk specimen

3.2

acoustic emission

AE

physical vibration caused by sudden movement within a material subjected to physical stress, often symptomatic of a defect

3.3

adhesion strength

load of diamond-like carbon (DLC) coating delamination detected by sharp signal increment of *acoustic emission* (3.2)

4 Specimens

4.1 General

Ball and disk specimens having the dimensions described later are prepared. To load equally on each ball, three balls shall be applied. In the ball-on-disk test, a bearing ball is fixed and used. The disk can be used as a substrate for a DLC film for testing. All appropriate details regarding the ball and disk specimens shall be provided, such as their dimensions, surface finish, material type, composition, microstructure, and processing.

4.2 Ball specimen

The ball specimen shall be 9,525 mm in diameter, made of 100Cr6 material as specified in ISO 683-17, and is recommended to be the equivalent to G28 or more as specified in ISO 3290-1. The hardness of the ball is HRC 62-67.

4.3 Disk specimen

The thickness of the disk specimen is 2,5 mm or more. The thickness of the DLC film is 0,1 μm to 10 μm . In order to set the sliding radius of the ball to 9 mm, the diameter of the test specimen is 28 mm or more. If the disk has a hole inside, the acceptable inner diameter is 12 mm or less. The surface of the disk shall be flat and parallel within 0,02 mm as specified in ISO 1101. The roughness of the test surface shall be less than 0,02 μm Ra as defined in ISO 21920-2. The hardness of the substrate is recommended to be HRC 60 or more, or 700 HV or more.

5 Apparatus

5.1 Testing equipment

Adhesion strength test using ball-on-disk testing equipment shall consist of the following:

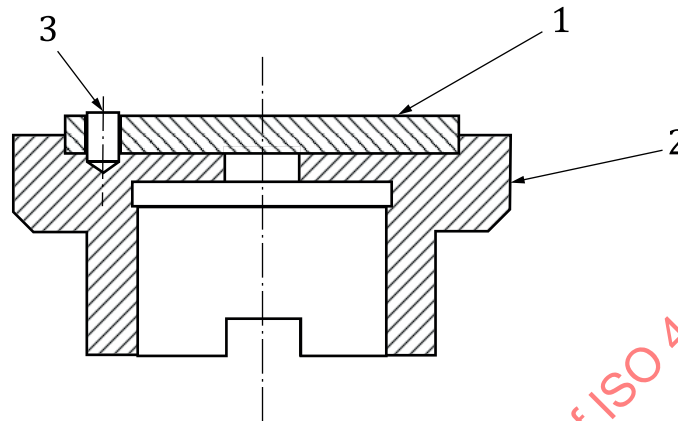
- a) the disk holder, for securing a disk specimen;
- b) the ball holder, for fixing ball specimens;
- c) the equipment, for driving the disk while pressing the ball against the disk with stepwise load;
- d) the equipment, for measuring the friction force;
- e) the oil bath maintaining lubrication and its temperature;
- f) the oil heater, for controlling the oil temperature;
- g) the acoustic emission (AE) sensor, for detecting delamination of DLC coating;
- h) the AE sensor holder, for detecting AE by way of ball specimens;
- i) the equipment, for amplifying AE signal;
- j) the equipment, for counting AE events;
- k) the ancillary devices for the above.

5.2 Specification of ball-on-disk equipment

The test equipment should have a capability to apply the load up to 5 kN, and speed at least 100 min^{-1} .

5.3 Sample of disk holder

The disk holder shall rotate in a horizontal plane. The eccentricity of the rotating axis shall be less than 0,02 mm and the fluctuation at the contact point in the direction perpendicular to the disk shall be less than 0,02 mm. The example of assembly is in [Figure 1](#). The concentricity between the upper frame centre and the centre of disk holder shall be aligned in the assembled condition.



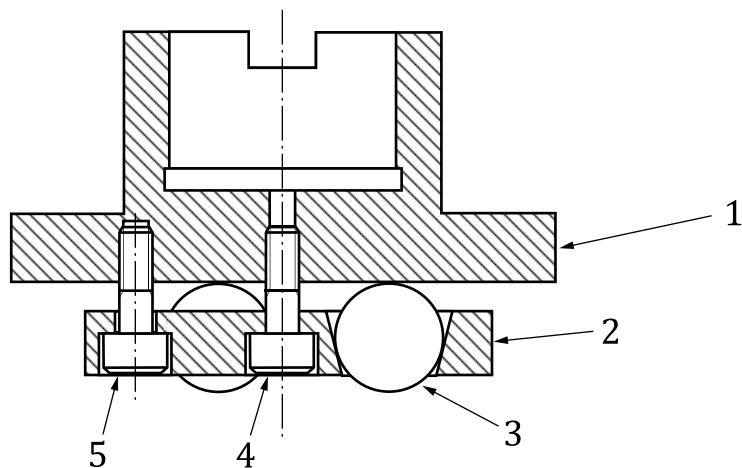
Key

- 1 disk
- 2 disk holder
- 3 pin to prevent from disk rotation

Figure 1 — Sample of disk holder

5.4 Sample of ball holder

A schematic of assembly is shown in [Figure 2](#). The ball holder shall fix the ball specimens to ensure the sliding motion between the ball specimens and the disk specimen. It is necessary to prevent from the ball specimen rolling. Bolt tension shall be given to bolts in [Figure 2](#) to fix the ball specimens to their holder in the test condition. The ball holder also should have a high rigidity because of the high stresses at the contact point between balls and their holder. In order to align orbitals of three balls, the concentricity of the upper frame centre and the centre of pitch circle diameter of the ball specimens shall be aligned in the assembled condition.



Key

- 1 upper frame
- 2 ball holder
- 3 ball specimen
- 4 bolt centre
- 5 bolt outer

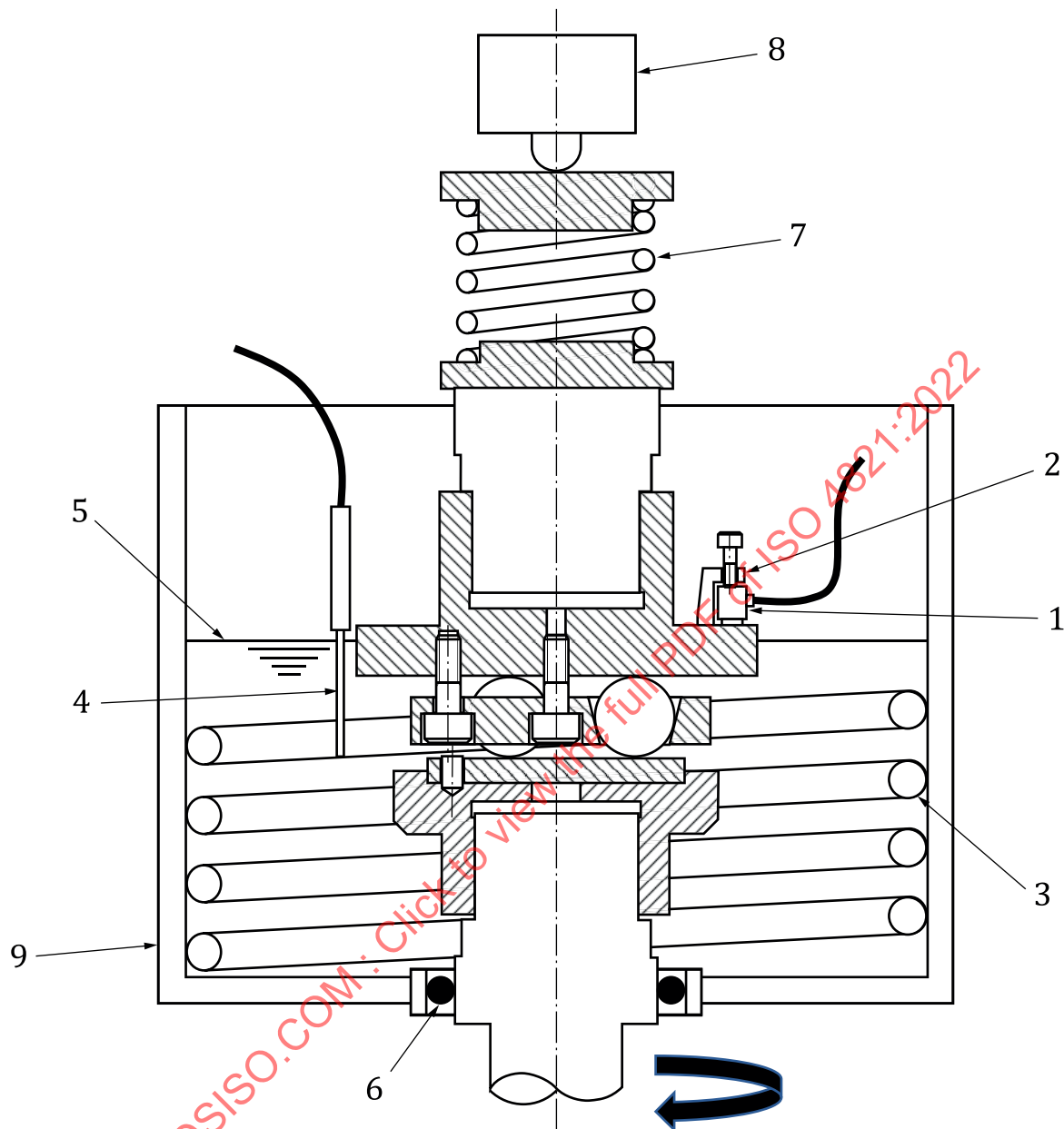
Figure 2 — Sample of ball holder

5.5 Oil bath and equipment for controlling oil temperature

The lubrication is maintained by either oil bath or circulation type. [Figure 3](#) shows an example of the oil bath system. It is permitted to have an arrangement “upside down”, i.e. the rotating part on top.

The level of lubricant is that at which the test piece is completely immersed.

The temperature of oil in the oil bath is controlled by a heater to within 2 °C of the set temperature at 80 °C.

**Key**

- | | | | |
|---|------------------|---|-------------|
| 1 | AE sensor | 6 | oil seal |
| 2 | AE sensor holder | 7 | load spring |
| 3 | oil heater | 8 | load cell |
| 4 | thermocouple | 9 | oil bath |
| 5 | lubricant | | |

Figure 3 — Oil bath and equipment for controlling oil temperature

5.6 AE sensor and its holder

The range of the frequency covered by the AE sensor should be 10 kHz to 1 000 kHz (see ISO 12713). The sensor surface contacts on the upper frame. An example of the AE sensor holder and its assembly are also shown in [Figure 3](#). The level of the lubricant is lower than the location of the AE sensor for the protection.

5.7 AE sensor amplifier

The amplifier for enhancing the AE signal is used. It is recommended to use the type of amplifier with a built-in filter that cuts off the frequency band of unnecessary signals generated from the equipment.

5.8 Universal counter for AE events

Since it is necessary to adjust the threshold value of the AE signal generated at the time when DLC coatings are delaminated, a specification having a function capable of adjusting the count trigger level is desirable. It is also recommended to have an external output to measure the cumulative value of the number of AE events in time series.

6 Testing procedure

6.1 Pre-treatment of specimen before test

Wash specimens ultrasonically in acetone, ethanol or hexane to remove the anti-oxidation lubricant.

NOTE It is presupposed that the user is aware of the Environmental Health and Safety (EHS) regulations regarding the use of chemicals, for example the Global Harmonized System (GHS) classifications.

6.2 Set up of adhesion test

Clamp balls and the disk specimen to contact and fix firmly in position. Before loading, lubricant oil shall be warmed up and kept at $80\text{ °C} \pm 2\text{ °C}$ for at least 10 min with 30 min^{-1} disk rotation to agitate lubricant oil. Then apply the set load on the disk.

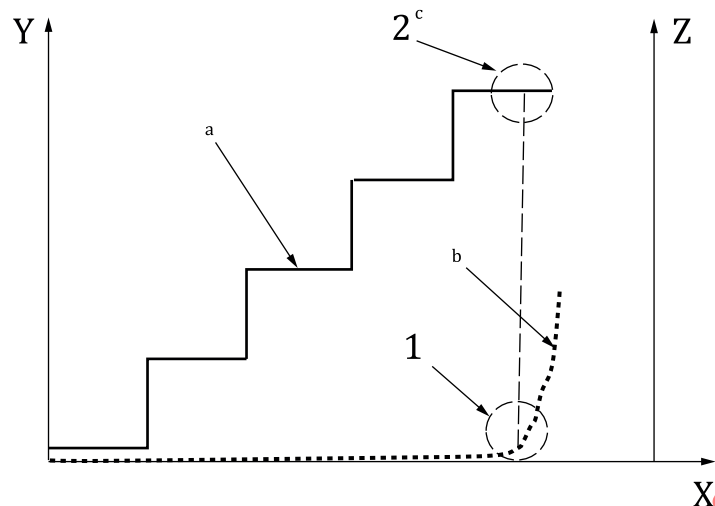
6.3 Lubricant

For the test lubricant, SM or SN specified by engine oil API 1509 is recommended, and 5W30 or 0W20 specified by SAE J300 is recommended as viscosity grade.

6.4 Testing conditions for adhesion test

Recommended test conditions for measuring adhesion test are listed as follows:

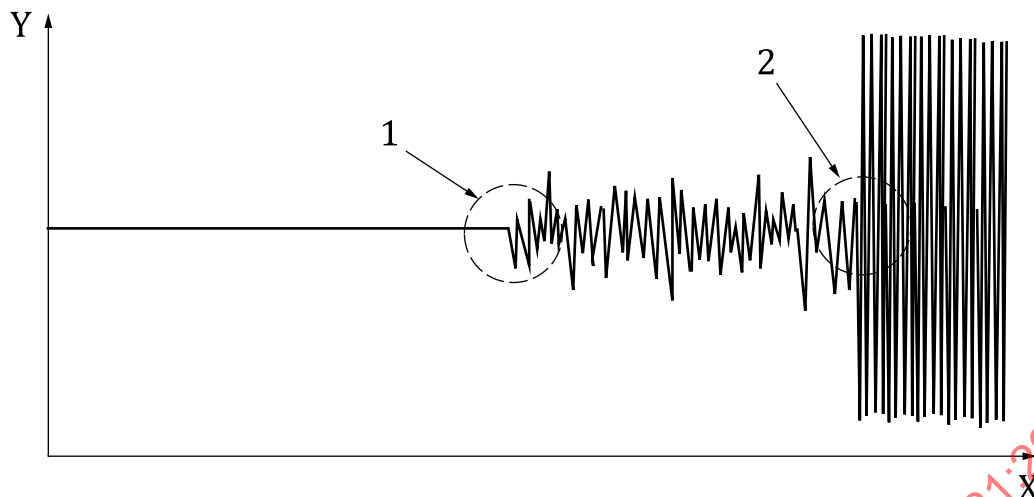
- applied load: step load is applied in 0,1 kN/10 s as shown in [Figure 4](#); initial load for the first step should be recorded;
- sliding speed: 0,028 m/s (30 min^{-1}).
- temperature of oil: $80\text{ °C} \pm 2\text{ °C}$.

**Key**

- X time
- Y load
- Z sum of AE events
- 1 start point of AE signal increment
- 2 point of the step load curve corresponding to point 1
- a The solid line represents step load, F .
- b The dotted line represents sum of AE events, N .
- c The load of point 2 is judged as the adhesion strength.

Figure 4 — Load pattern**6.5 Measurement of AE events**

Figure 4 also shows the example, that is, results of time series integration of the number of AE signals generated during the test. The load of point 2 of Figure 4 at which the number of AE signals increases rapidly is defined as the adhesion strength at which DLC coatings delaminated. Figure 5 shows an example of measuring the amplitude of the AE signal in the course of testing. In this example, it is shown that partial delamination of the coating occurred at the time of point 1 of Figure 5, and that the entire surface of the coating was delaminated after the time of point 2 of Figure 5. The time of point 1 of Figure 4 at which the AE signal increases sharply coincides with the time of point 1 of Figure 5 at which the amplitude fluctuates suddenly, so the load of point 2 of Figure 4 can be judged as the adhesion strength. As a preparation, pre-test is necessary to check the consistency between AE event increment at time of point 1 of Figure 4 and actual DLC delamination by scanning electron microscope (SEM) observation.

**Key**

X time

Y AE signal amplitude

1 start point of AE signal increment

2 start point of AE signal amplitude becoming quite big

Figure 5 — Transition of typical AE signal amplitude**6.6 Number of test repeats**

Repeat the adhesion strength test at least three times under the same testing conditions using new disk specimen.

7 Recommendation**7.1 Measurement of friction force**

Measure the friction force continuously during the test and record it by using a data logger or another recording device. An appropriate system for averaging the fluctuation with rotation period should be adopted. Before the test starts, the zero of the friction force by the measurement device should be checked with the specimens not in contact with one another.

Although the delamination of DLC coatings are judged at the point where the number of AE signals increases rapidly, an increase in the coefficient of friction may be recognized due to the exposure of the substrate by the delamination of DLC coatings and the friction between the exposed substrate and the balls. Therefore, it is desirable to grasp the change of the number of AE signals and the change of friction coefficient, and to determine the delamination of the coating complementarily.

7.2 Observation of disk after the test

The sliding part of the DLC coating after the adhesion strength test should be subjected to an optical microscope or an electron microscope to confirm the delamination of the coating occurs. If the delamination is found, the judgment of the adhesion at the point where the AE signals increased sharply is correct. It is desirable to check indirectly using different methods.

7.3 Observation of balls after the test

It is desirable to check if there is any abnormal wear of the ball, any deviation in the trajectory, or any appearance of the ball rolling. While using new ball specimens for a new test is desirable, reusing ball

specimens is acceptable if users identify the ball sliding point with disk in the previous test and avoid contact with the same point again in the next test.

8 Test report

The test report shall contain at least the following information:

- a) descriptions of the type of DLC coatings, DLC film thickness and roughness, disk material and lubricant;
- b) a reference to this document, i.e. "Determined in accordance with ISO 4821:2022";
- c) the name and specification of adhesion testing apparatus (disk driving method, orientation of disk rotation, loading method, type of AE sensor, frequency of AE detecting measurement, set up condition of AE amplitude, heating method of lubricant, etc.);
- d) the testing conditions (step load diagram, sliding speed, rotational velocity of disk, sliding distance, temperature of lubricant, etc.), refer to [6.4](#);
- e) the following items which should be reported for each individual test, refer to [6.4](#):
 - 1) the adhesion strength of DLC coatings;
 - 2) the change in number of AE events during the test;
 - 3) the testing temperature and their ranges;
 - 4) the change of friction coefficient during the test;
 - 5) any other matters which should be noted with regard to the state of test and the specimen after the test;
 - 6) the results of estimated uncertainty of measurement;
 - 7) any deviations from the procedure.
- f) the date of the test.

Annex A (informative)

Example of stages for adhesion strength tests of coatings applied to engine parts

The positioning of this test method in the process of parts evaluation performed by automobile manufacturers or part suppliers is described in this annex. There are three stages of functional evaluation test methods of hard coatings for engines using various kinds of test rigs as shown in [Table A.1](#).

Table A.1 — Example of stages for adhesion strength tests of coatings applied to engine parts

Stage	Purpose	Test condition	Test method
1	Screening test of the adhesion strength	Dry condition using specific parts	Indentation scratch test
2	Precise evaluation test for the adhesion strength of coatings	Lubricated condition using actual engine parts	Rig test specialized for engine component
3	Validation of the actual engine	Actual engine test	Actual engine test Actual car test

Stage 1 is the initial screening test. Although the Rockwell indentation test defined in ISO 26423 is simple, it can be evaluated only in the five-step classification of the delamination morphology, and lacks quantitativeness. The scratch test defined in ISO 20502 is not suitable for the evaluation of a thin coating with many irregularities on the surface because the tip diameter of the probe is small. In addition, because it is evaluated without lubrication, it is susceptible to the coefficient of friction of the film. Therefore, the difference from the engine test using real parts is considered to be large, and the next stage 2 test is required.

At stage 2, by using a specially-designed test device capable of reproducing the same sliding conditions as the engine, consistency with the engine test is obtained by using a sample deposited on actual engine components. Because the special testing facility with actual engine components is expensive and requires know-how to control its behaviour, it is difficult to own and manage it by test users, except for automobile manufacturers or part suppliers.

Stage 3 consists of a bench evaluation using an actual engine or an engine evaluation on a car. Because it is a large evaluation, it can be evaluated only by automobile manufacturers or part suppliers.

The test method specified in this document is a test device equipped with a triple simple ball-on-disk contact test equipment that has correlation with conventional engine evaluation results and a relatively inexpensive acoustic emission measurement machine without using actual parts. A test that combines stage 1 and stage 2 is possible, and it is a method for quantitatively evaluating the adhesion strength of the coating in a short time.