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Thermal spraying — Evaluation of adhesion/cohesion of thermal sprayed ceramic coatings by transverse scratch testing

Projection thermique — Évaluation des propriétés d'adhérence/cohésion des revêtements céramiques appliqués par projection thermique, par essai de rayure transversale

Reference number
ISO 27307:2015(E)

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Foreword

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The committee responsible for this document is ISO/TC 107, *Metallic and other inorganic coatings*.

Introduction

Thermal sprayed ceramic coatings have been widely applied in modifying surface properties of metal components. It is useful to prevent various types of wear, corrosion, and thermal damage, thereby extending components service life and reducing the need for expensive and repetitive maintenance. Droplet morphology, size and density of pores, inter/intra-lamellar thermal cracks, and trapped inclusions are used to characterize the microstructure of thermal sprayed ceramic coatings. Although cracks are purposely introduced to the coating structures, for example, in thermal barrier coating for improvement of its thermal performance, the poor bonding between splats and the imperfections in the form of pores or thermal cracks cause the mechanical properties of thermal sprayed ceramic coatings to be considerably less than those of the corresponding monolithic materials. Evidently, the durability and functionality of coatings is critically dependent on the adhesion between the coating and the underlying substrate as well as the cohesion among splats. So, the adhesion/cohesion strength is one of the most important properties for thermal sprayed coatings and needs to be evaluated systematically.

To assist and promote the development of thermal sprayed ceramic coatings, it is important that good test methods are available for evaluation of their adhesive and cohesive strength. This International Standard gives guidance conducting of transverse scratch testing which can provide this information.

Thermal spraying — Evaluation of adhesion/cohesion of thermal sprayed ceramic coatings by transverse scratch testing

1 Scope

This International Standard specifies a method for adhesion and cohesion evaluation of thermal sprayed ceramic coating-substrate systems by means of a transverse scratch test. This International Standard is suitable for thermal sprayed ceramic coatings with thickness from 50 µm to over 1 000 µm. It is also suitable for testing ceramic coatings deposited by other spray coating processes, such as cold spray, as well as aerosol deposition.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 20502, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of adhesion of ceramic coatings by scratch testing*

3 Terms and definitions

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

3.1
adhesion
state in which coatings and the underlying substrate are held together by interfacial forces which may consist of valence forces or interlocking forces or both

3.2
cohesion
strength of bonding between structural elements in coatings

4 Principle

Scratch testing involves drawing an indenter of known geometry across a coating-substrate system. In this test, the indenter is of a known material, size, and geometry such as a Rockwell conical diamond indenter. Testing is performed over a given length under a constant load. If the coating is removed from the substrate, then the adhesion of the coating-substrate system can be evaluated and the adhesion level may be ranked against similar coating-substrate system combinations. If the failure occurs only in the thermal sprayed ceramic coating, it reveals the cohesion of coatings itself. In the case of no observed failure, the test load may be increased until the coating failure occurs. The various coating failure modes during scratch testing are shown in Annex A.

During scratch testing, cracks can form as a result of the tensile stress induced behind indenter and these stresses could balance the compressive frictional stresses ahead. Crack formation can be detected by means of an acoustic emission (AE) sensor attached to the indenter holder of scratch testing machine. In addition, the tangential force between the indenter and test surface can be monitored to provide more information on the failure conditions. This tangential force, often incorrectly referred to as a frictional force, is the result of both friction between the indenter and the test specimen and the ploughing force required to deform the coating-substrate system.

5 Equipment and materials

A scratch testing machine is an instrument used to rigidly hold the indenter and to apply both the normal load and the driving force to produce scratches. In general, the displacement of a spring is used to achieve the chosen loading program. Magnetically driven assemblies are equally appropriate.

The scratch testing machine consists of a rigidly mounted diamond indenter, normally having Rockwell conical diamond geometry. In the present case, a diamond indenter with a 20 µm tip radius is recommended. The scratch tester used shall have been calibrated in accordance with the procedures given in ISO 20502.

6 Procedures of transverse scratch testing

6.1 General requirements for the testing specimen

The specimen size for scratch testing varies as the sample holders of scratch testing machines differ. Generally, a specimen size of 20 × 20 mm^2 is recommended.

After spraying, the coatings are sectioned perpendicular to the coating surface and embedded in a suitable material and then polished.

Using standard metallographic procedures, the cross-section of the coatings shall be polished to Ra < 0,5 µm before the scratch testing (see ISO 4287).

NOTE Embedding in a cold mounting medium, such as acrylic resin, is recommended as specimens might be sensitive to heat or pressure. Vacuum impregnation is not typically required.

Prior to scratch testing, the test specimen should be freed from surface contaminants, such as oil, grease and moisture, by cleaning it with alcohol or acetone and allowing it to dry. Finger prints on the test area should be avoided.

The following information is required and shall be obtained from the coating fabricator:

a) substrate material and thickness;

b) coating material and thickness.

6.2 Environmental conditions

Scratch testing requires interaction between the indenter and the specimen, which might be sensitive to environmental conditions. Therefore temperature and relative humidity should be monitored and controlled to ensure reproducibility.

NOTE The recommended environmental conditions are

— temperature: 22 °C ± 2 °C, and

— relative humidity: 50 % ± 5 %.

6.3 Scratch testing on cross-section

Scratch testing procedures generally include the following steps.

Ensure the diamond indenter is clean and free from debris from previous scratches. If necessary the indenter may be cleaned by wiping it with a soft tissue soaked in petroleum ether. If adhering debris is still observed under an optical microscope (recommended magnification: × 200), #1200 and #2400 SiC paper can be used to remove it, followed by wiping with a soft tissue soaked in petroleum ether. Ultrasonic cleaning of the indenter should not be used as cavitation damage can occur.

Following cleaning, the indenter shall be allowed to reach room temperature before proceeding with the test.

Set the scratch length to 2 mm to 3 mm and the indenter traverse speed to 0,1 mm min^{-1} to 10 mm min^{-1}. Mount one of the previously prepared test samples rigidly in the sample holder and bring the diamond indenter, recommended tip radius of 20 µm, into contact with the substrate portion of the sample such that the scratch, of selected length, will lie perpendicular to the coating-substrate interface and will traverse the coating and end in the mounting medium. Scratch the sample using a constant normal load of 3 N. Lift the indenter, reposition the sample such that the next scratch will be made a minimum of 4 mm away from the previous one in order to avoid interaction of the stress fields from adjacent scratches, reposition the indenter as for the first scratch, and produce a second scratch. Repeat this procedures until the required number of scratches under the initial load have been produced, normally at least 5, changing test samples as necessary. Inspect the diamond to ensure that it is clean and free from debris and clean as necessary. Repeat the above procedure for constant normal loads of 5 N, 10 N, 13 N, 15 N and 20 N (or more), inspecting the diamond and recleaning as necessary before each change of load, until all scratches under the highest load produce adhesive failure, as observed under an optical microscope with a magnification of 100:1.

NOTE The need for at least 5 scratches to be made at each load is related to the heterogeneity of thermal sprayed ceramic coatings, which results in large variations in the response of the coating to scratch testing.

Using an optical microscope, take images of each of the scratches, using a magnification of >100:1, ensuring that the substrate, coating and mounting medium are all in the field of view.

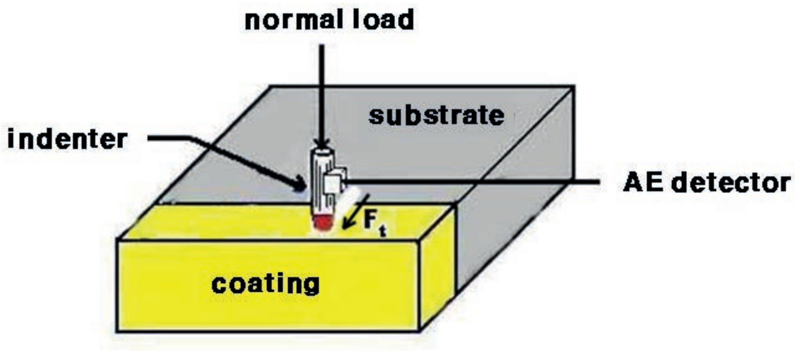


Figure 1 — Schematic of scratch testing on cross-section of thermal sprayed ceramic coating/substrate system

6.4 Evaluation of failure mode

Using the images taken after scratch testing, determine the percentage of each failure mode present. Typical failure modes of no crack, cohesive crack, and adhesive crack are illustrated in Annex A.

Adhesive failure is indicated by the presence of cracks generated at an interface between the coating and another part of the coating-substrate system (bond coat/substrate or top coat/bond coat), whereas cracks generated within the coating are indicative of cohesive failure. In the case of both types of failure being present in a sample group, a failure mode is regarded as valid if it constitutes more than 30 % of the observed failures.

Example of failure mode analysis.

Table 1 — Example of evaluation of the failure mode

Coating[a]	Load	Number of scratches	No crack	Cohesive crack	Adhesive crack
A	3	12	80	20	0
A	5	12	75	25	0
A	10	12	60	35	5
A	13	12	45	40	15
A	15	12	0	65	35
A	20	12	0	20	80
B	3	12	80	20	0
B	5	12	55	33	12
B	10	12	20	45	35
B	13	12	10	40	50
B	15	12	0	20	80
B	20	12	0	0	100

[a] For coating A: coating strength = 10 N, adhesive strength = 15 N. For coating B: coating strength = 5 N, adhesive strength = 10 N.

7 Scratch testing report

The scratch testing report shall include the following information:

a) environmental conditions (temperature and relative humidity);

b) coating and substrate thickness and materials;

c) coating process used;

d) indenter tip radius;

e) indenter traverses speed;

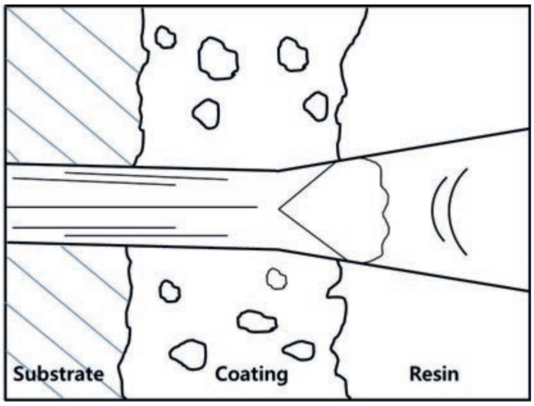
f) test load;

g) percentage of each failure mode.

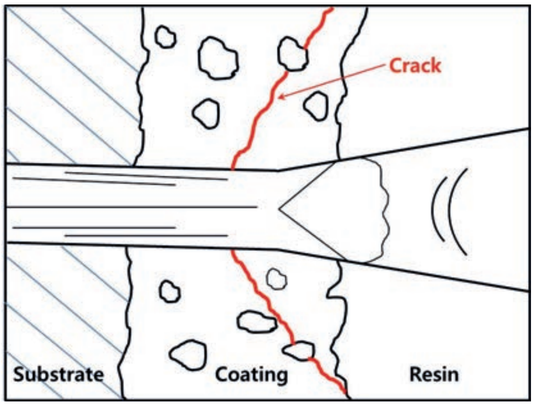
NOTE A model form for scratch testing record is presented in Annex B.

Annex A
(informative)

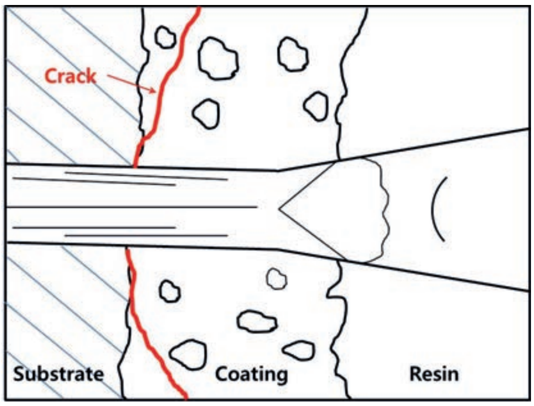
Typical coating failure modes after scratch testing



a) No crack



b) Cohesive crack



c) Adhesive crack

Figure A.1 — Typical coating failure modes after scratch testing

Annex B
(normative)

Transverse scratch testing record

Document No.					
Temperature	°C		Relative humidity		%
Substrate material			Coating material		
Test condition	Indenter traverse speed				mm/min
	Scratch length				mm
Test results					
Test load	No.	Results	Test load	No.	Results
N	1	No crack/Cohesive/Adhesive	N		No crack/Cohesive/Adhesive
N	2		N		
N	3		N		
N	4		N		
N	5		N		
N			N		
N			N		
N			N		

Percentage of each failure mode							
Load (N)	No crack (%)	Cohesive crack (%)	Adhesive crack (%)	Load (N)	No crack (%)	Cohesive crack (%)	Adhesive crack (%)
Date Institute				Operator			

Annex C
(informative)

Round robin test results of transverse scratch testing for plasma sprayed ceramic coatings

The round robin test has been done for a reference guide to evaluate adhesion/cohesion strength of plasma sprayed ceramic coatings among Korea, Japan, and Finland on the basis of ISO/TC 107 N 939 Resolution 17.

Commercially available TiO_2 and yttria-stabilized zirconia (YSZ) powders were used to prepare thermal sprayed coatings. TiO_2 and YSZ coatings with 50 µm of NiCr bond coat and 150 µm of ceramic top coating were deposited on stainless steel substrates by commercial plasma spray system. Argon was used as a primary plasma gas and hydrogen was used as an auxiliary one.

After spraying, test samples were prepared in a standard way of grinding and polishing, where the coatings were sectioned perpendicular to the coated surface. The scratch test was conducted on the cross-section of the spray coatings using different scratch test machine in three countries, Korea, Japan, and Finland, separately. The test conditions are listed in Table C.1.

Table C.1 — Test condition used in the round robin test

	Korea	**Japan**	**Finland**
Indenter	Conical diamond with 120° angle and 20 µm ± 2,0 µm tip radius	Conical diamond with 120° angle and 20 µm tip radius	Conical diamond with 120° angle and 20 µm ± 1,0 µm tip radius
Scratch mode	Constant load	Constant load	Constant load
Load tested	3 N, 5 N, 10 N, 13 N, 15 N, and 20 N	3 N, 5 N, 10 N, 13 N, 15 N, and 20 N	3 N, 5 N, 10 N, 13 N, 15 N, and 20 N
Scratch length	2 mm	2 mm	2 mm
Scratch speed	0,1 mm/s	0,1 mm/s	6,6 mm/s
No. of measurement	4	4	4 per specimen
No. of specimen	3	3	3 per applied load
Relative humidity	—	—	50 %
Temperature	—	—	22 °C

Figures C.1 and C.2 show optical micrographs on the cross-section of plasma sprayed TiO_2 coatings scratched at a constant load of 3 N and 20 N, which was tested in Finland. From the results, 4 of no cracks and 6 of cohesive cracks were founded on the TiO_2 coatings tested at 3 N. In a case of 20 N, 1 of cohesive cracks and 4 of adhesive cracks could be founded. At a low scratch load, no crack or cohesive crack were major failure modes in both coatings. As increasing scratch load to 20 N, most of the failure mode changed to adhesive crack manner.

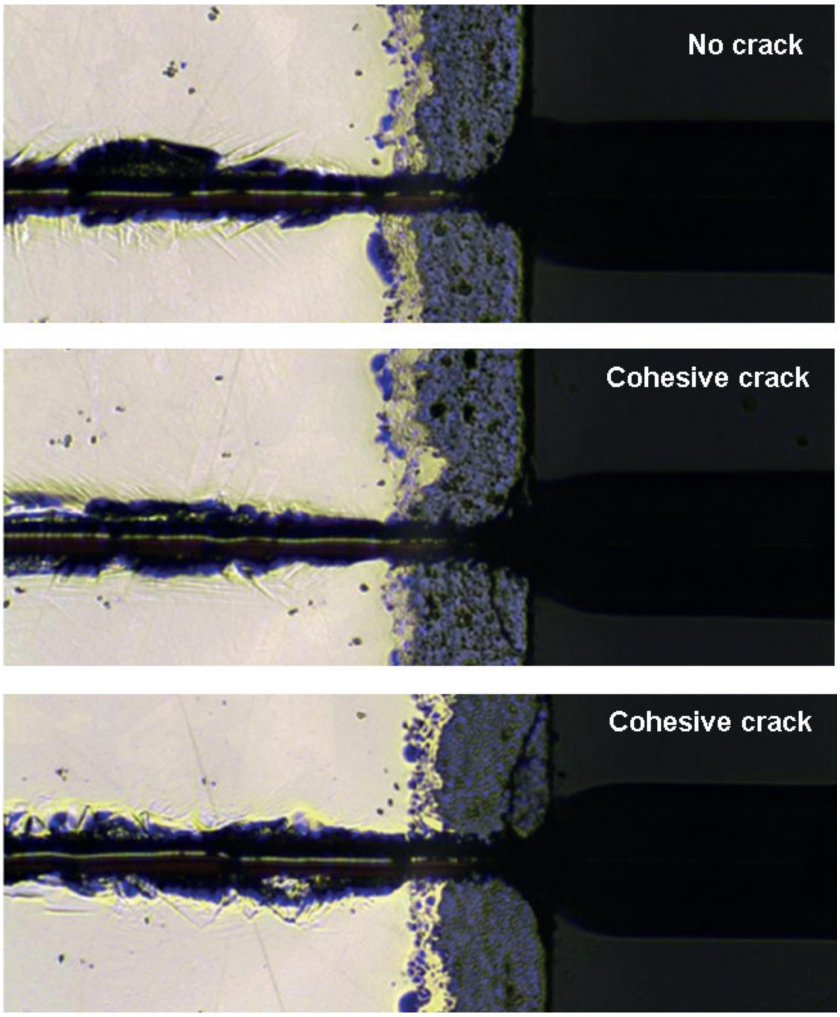


Figure C.1 — Optical micrographs on the cross-section of plasma sprayed TiO_2 coatings scratched at a constant load of 3 N, which tested in Finland

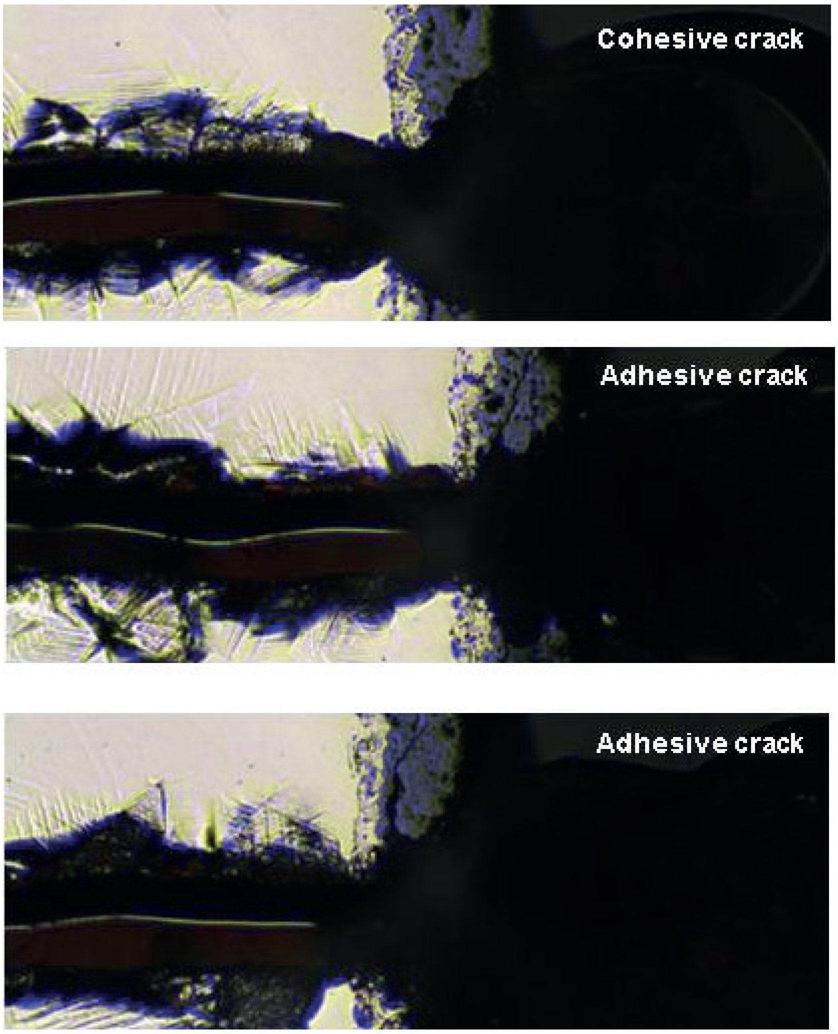


Figure C.2 — Optical micrographs on the cross-section of plasma sprayed TiO_2 coatings scratched at a constant load of 20 N, which tested in Finland

Optical micrographs on the cross-section of plasma sprayed YSZ coatings scratched at a constant load of 3 N and 20 N, which also tested in Finland, are shown in Figures C.3 and C.4. With similar to TiO_2 coating, 7 of no cracks and 4 of cohesive cracks were founded on the YSZ coatings tested at 3 N. It shows 2 of cohesive cracks and 10 of adhesive cracks at a 20 N of scratch load.

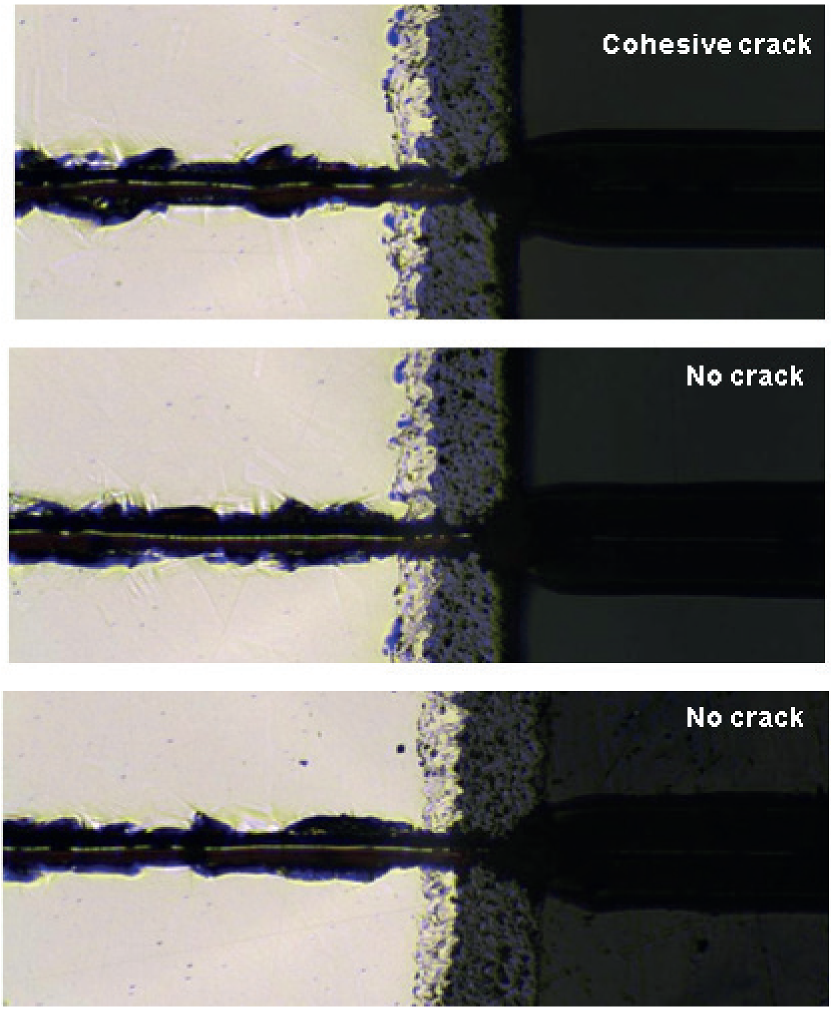


Figure C.3 — Optical micrographs on the cross-section of plasma sprayed YSZ coatings scratched at a constant load of 3 N, which tested in Finland

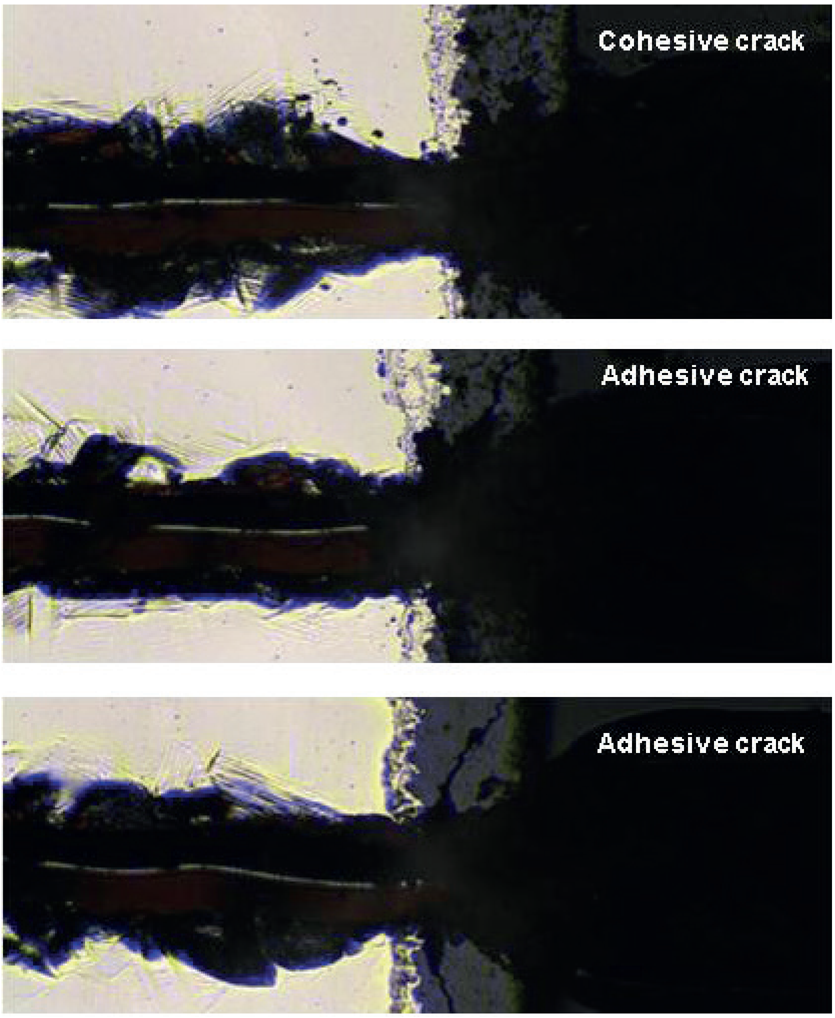


Figure C.4 — Optical micrographs on the cross-section of plasma sprayed YSZ coatings scratched at a constant load of 20 N, which tested in Finland

Test results obtained in Finland are summarized in Table C.2.

Table C.2 — Summary of the test results obtained in Finland

Coating	Load (N)	No. of scratches	No crack (%)	Cohesive crack (%)	Adhesive crack (%)
TiO_2	3	10	40	60	0
TiO_2	5	12	33,3	66,7	0
TiO_2	10	12	8,3	75	16,7
TiO_2	13	11	18,2	81,4	0
TiO_2	15	12	0	36,4	63,6
TiO_2	20	9	0	11,1	88,9
YSZ	3	11	63,6	36,4	0
YSZ	5	12	58,3	41,7	0
YSZ	10	12	58,3	41,7	0
YSZ	13	12	0	83,3	16,7

Table C.2 *(continued)*

Coating	Load (N)	No. of scratches	No crack (%)	Cohesive crack (%)	Adhesive crack (%)
YSZ	15	8	0	37,5	62,5
YSZ	20	12	0	11,1	88,9

Figure C.5 shows optical micrographs on the cross-section of plasma sprayed TiO_2 coatings scratched at a constant load of 3 N, which obtained in Japan. It shows 7 of no cracks and 5 of cohesive cracks. Figures C.6 to C.10 show scratch grooves scratched at 5 N, 10 N, 13 N, 15 N, and 20 N. All test results obtained in Japan are summarized in Table C.3.

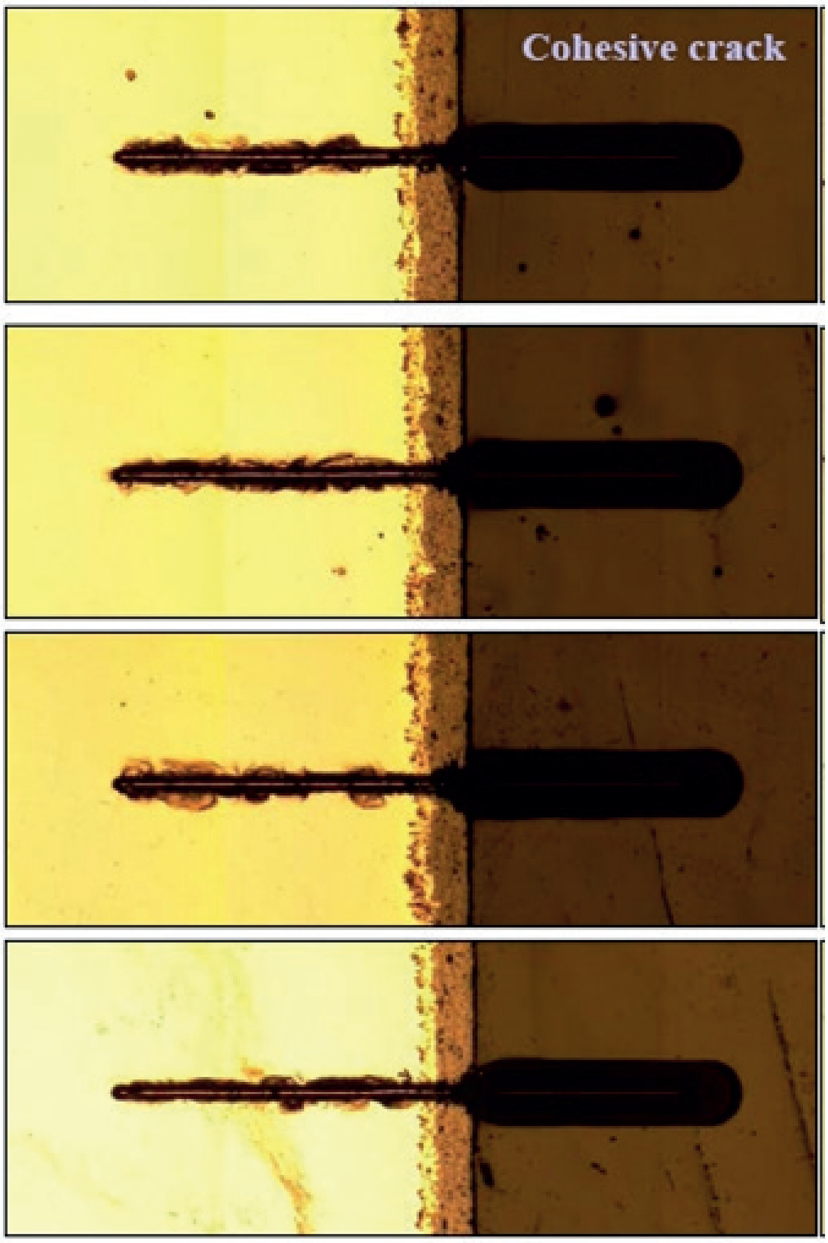


Figure C.5 — Optical micrographs on the cross-section of plasma sprayed TiO_2 coatings scratched at a constant load of 3 N, which tested in Japan

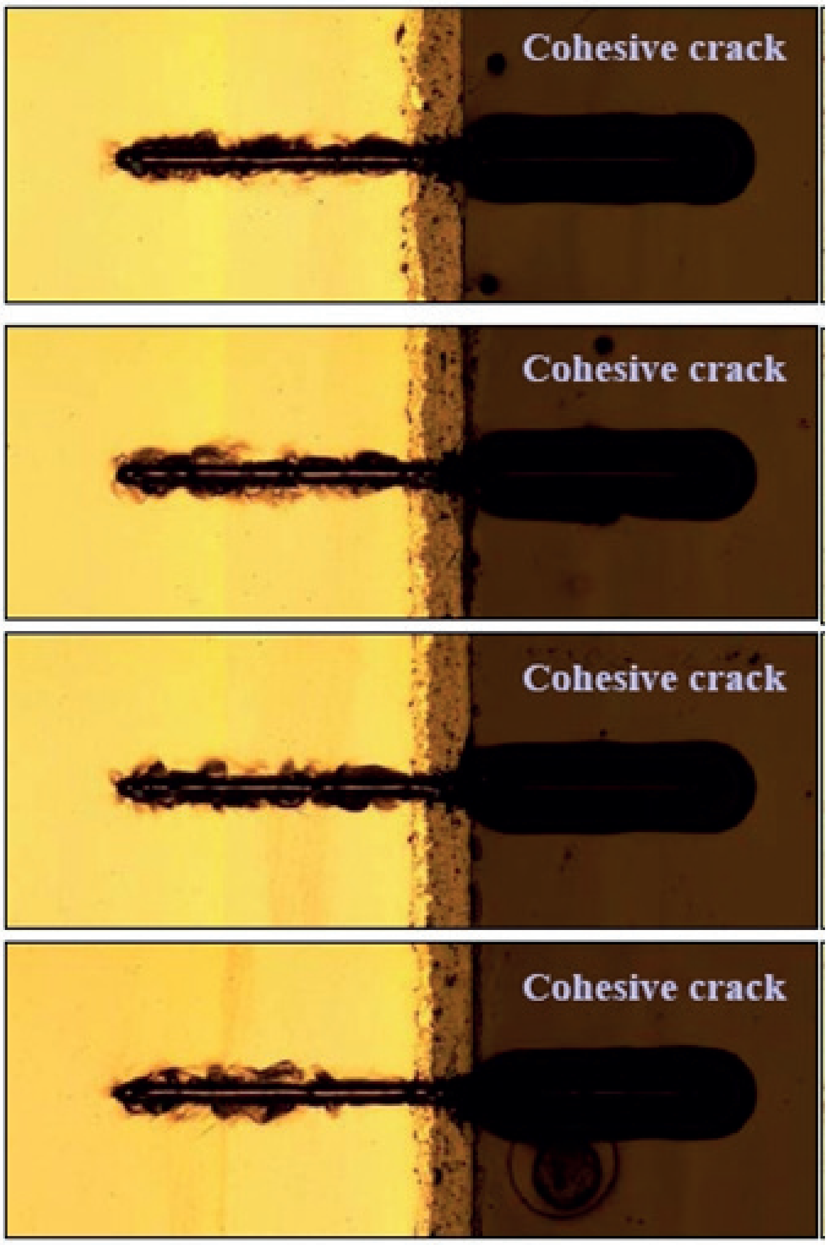


Figure C.6 — Optical micrographs on the cross-section of plasma sprayed TiO_2 coatings scratched at a constant load of 5 N, which tested in Japan

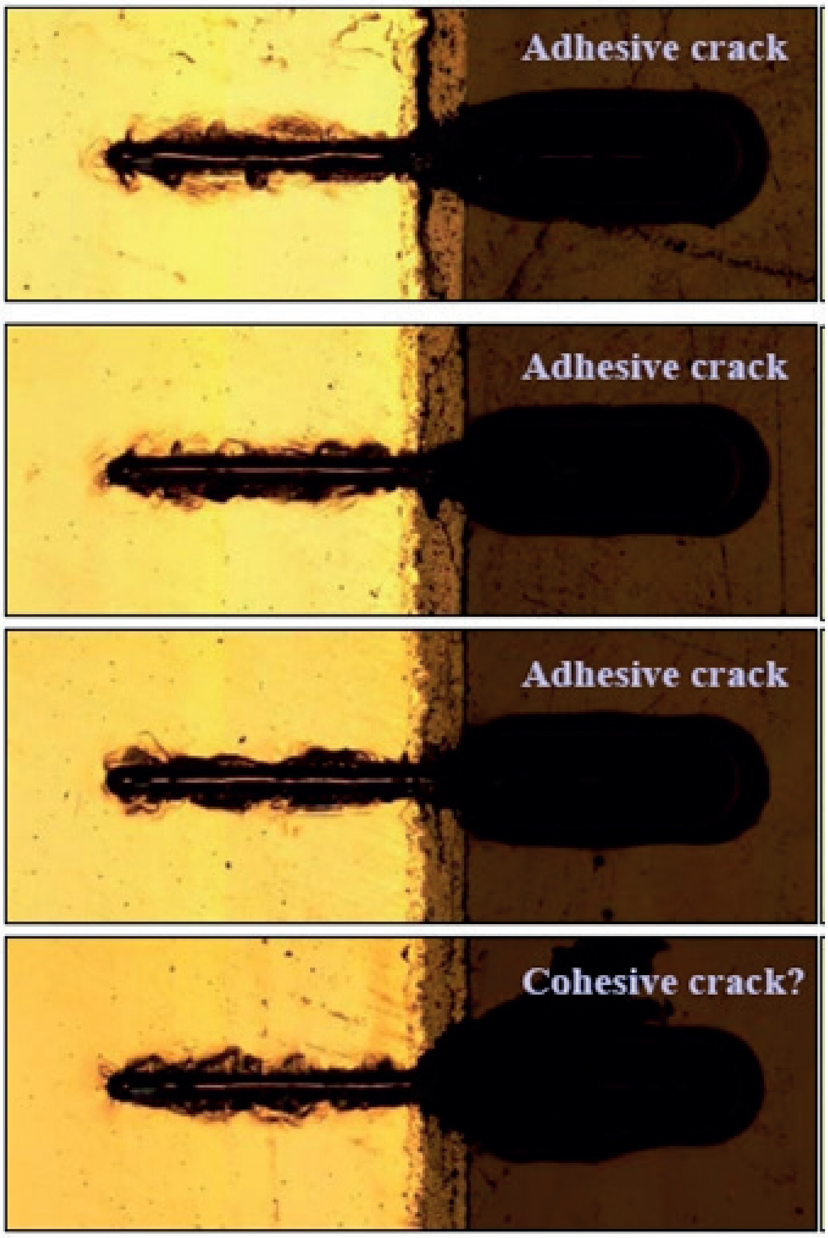


Figure C.7 — Optical micrographs on the cross-section of plasma sprayed TiO_2 coatings scratched at a constant load of 10 N, which tested in Japan

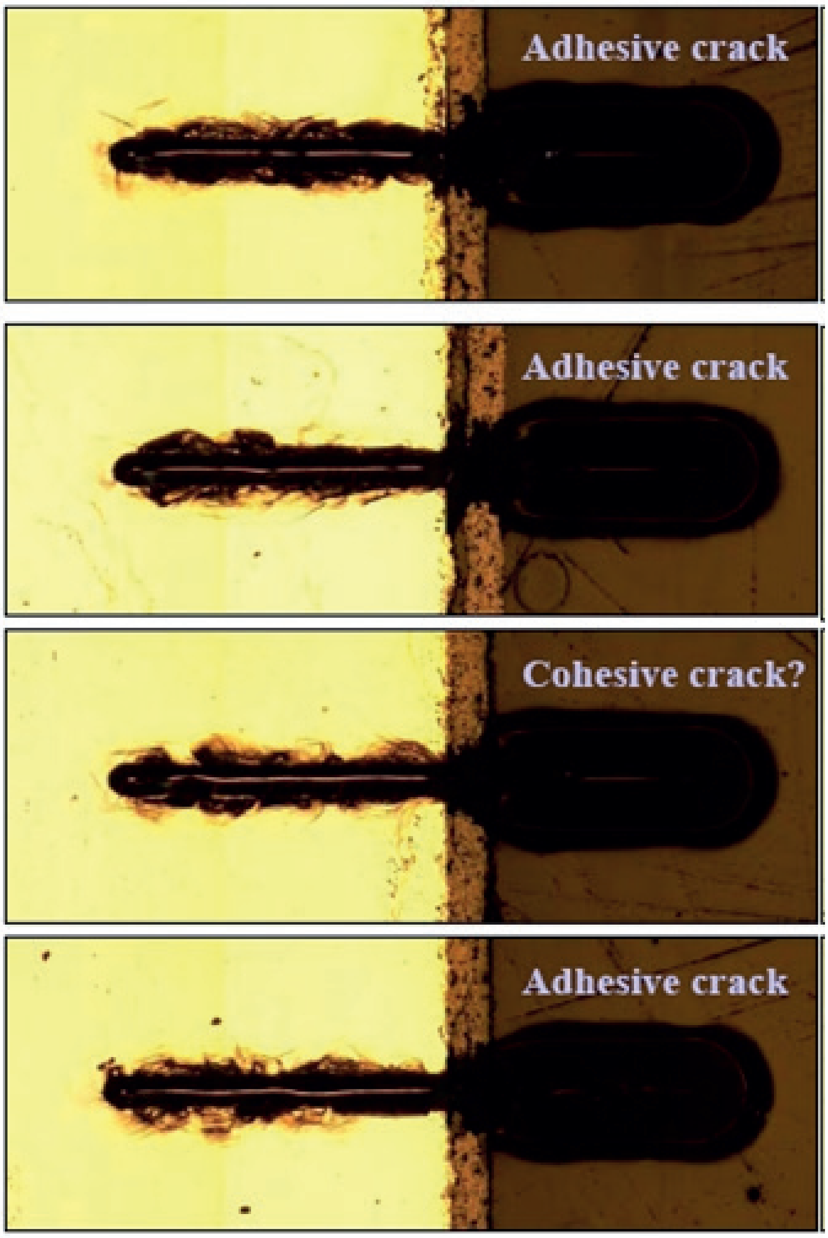


Figure C.8 — Optical micrographs on the cross-section of plasma sprayed TiO_2 coatings scratched at a constant load of 13 N, which tested in Japan

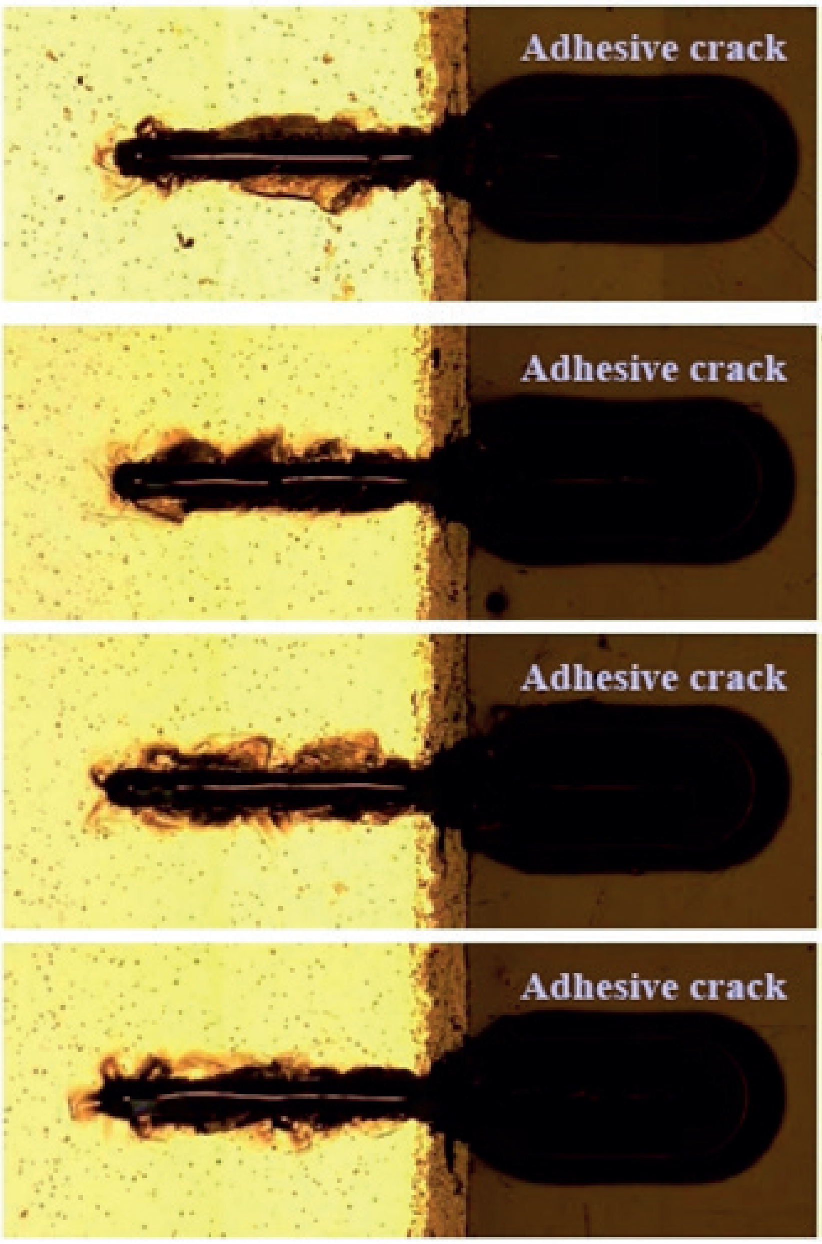


Figure C.9 — Optical micrographs on the cross-section of plasma sprayed TiO_2 coatings scratched at a constant load of 15 N, which tested in Japan

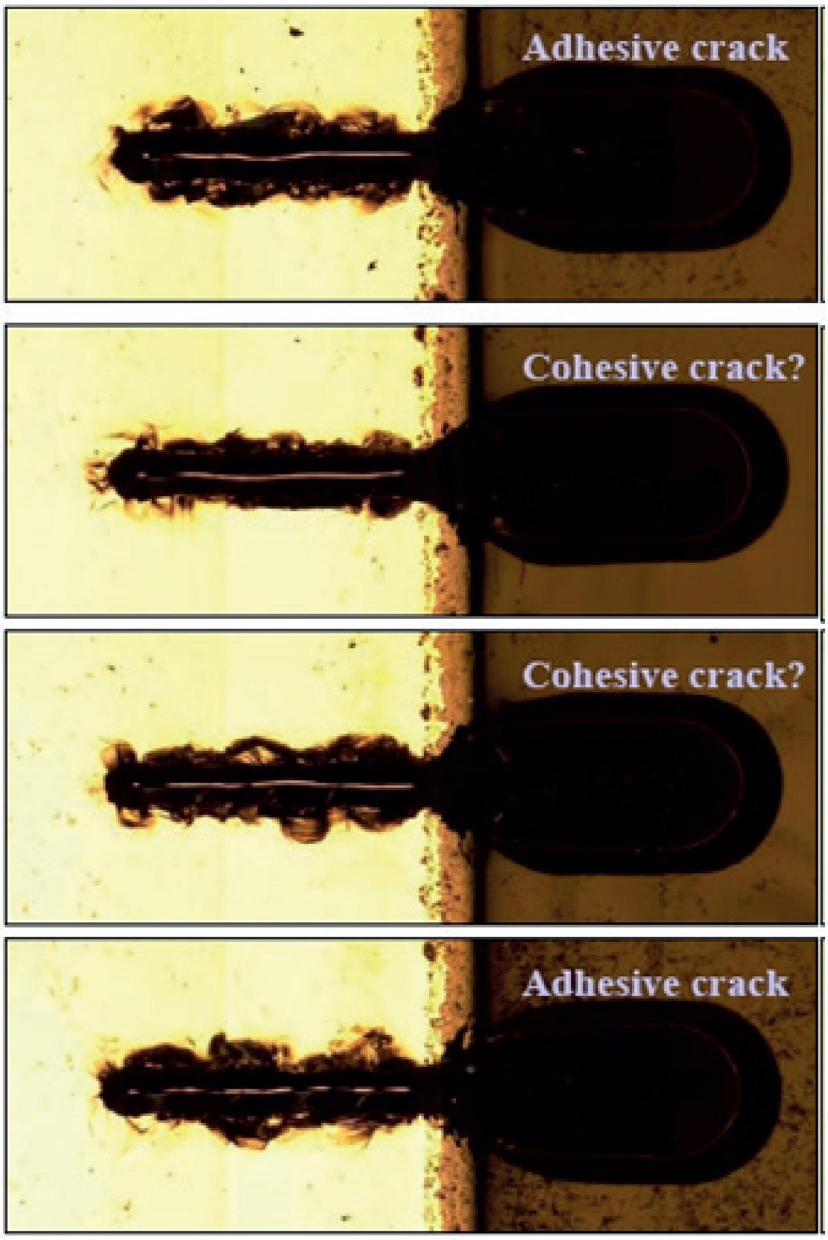


Figure C.10 — Optical micrographs on the cross-section of plasma sprayed TiO_2 coatings scratched at a constant load of 20 N, which tested in Japan

Table C.3 — Summary of the test results obtained in Japan

Coating	Load (N)	No. of scratches	No crack (%)	Cohesive crack (%)	Adhesive crack (%)
TiO_2	3	12	58,3	41,7	0
TiO_2	5	12	16,7	83,3	0
TiO_2	10	12	0	41,7	58,3
TiO_2	13	12	0	50	50
TiO_2	15	12	0	16,7	83,3
TiO_2	20	12	0	16,7	83,3

Table C.3 *(continued)*

Coating	Load (N)	No. of scratches	No crack (%)	Cohesive crack (%)	Adhesive crack (%)
YSZ	3	12	91,7	8,3	0
YSZ	5	12	75	25	0
YSZ	10	12	25	50	25
YSZ	13	12	8,3	50	41,7
YSZ	15	12	8,3	25	66,7
YSZ	20	12	0	12,5	87,5

Figures C.11 to C.16 show optical micrographs with AE signal, friction coefficient and friction force scratched at 5 N, 10 N, 13 N, 15 N, and 20 N, which obtained in Korea.

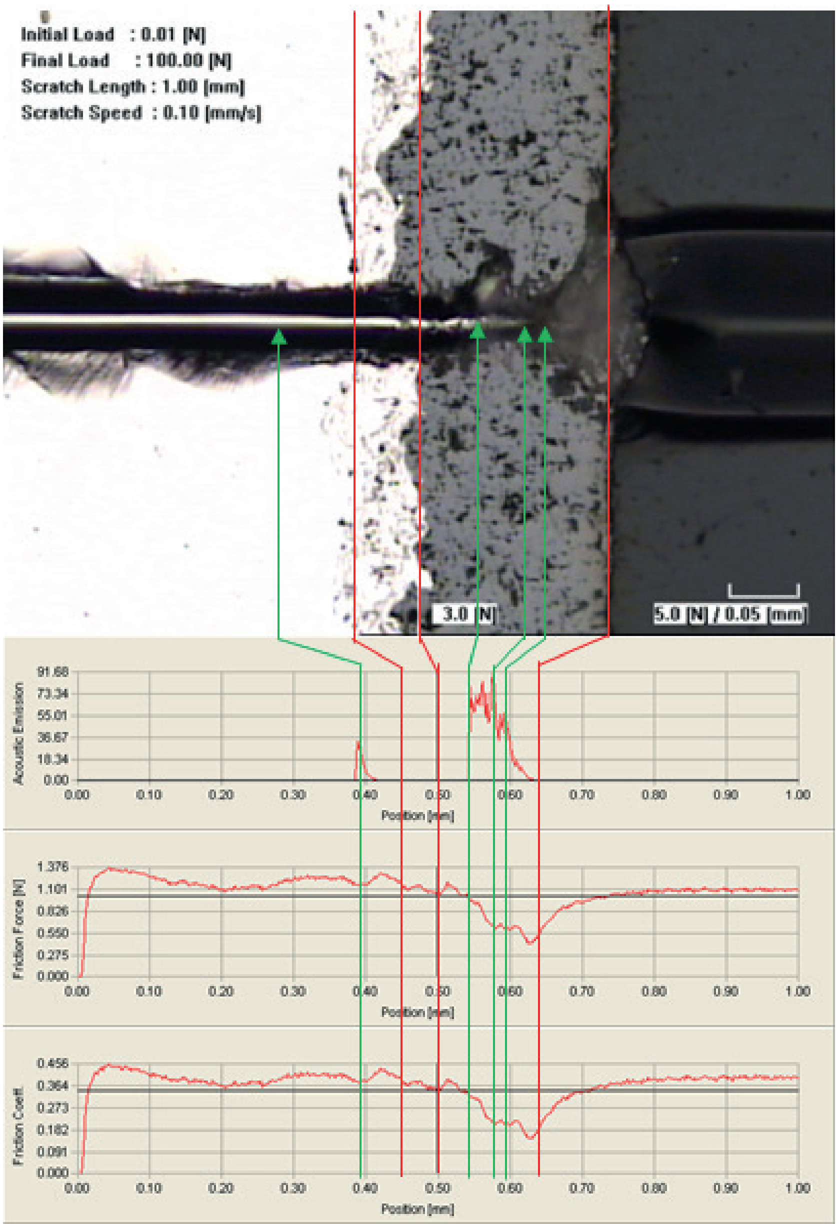


Figure C.11 — Scratch test results of plasma sprayed YSZ coatings scratched at a constant load of 3 N, which tested in Korea

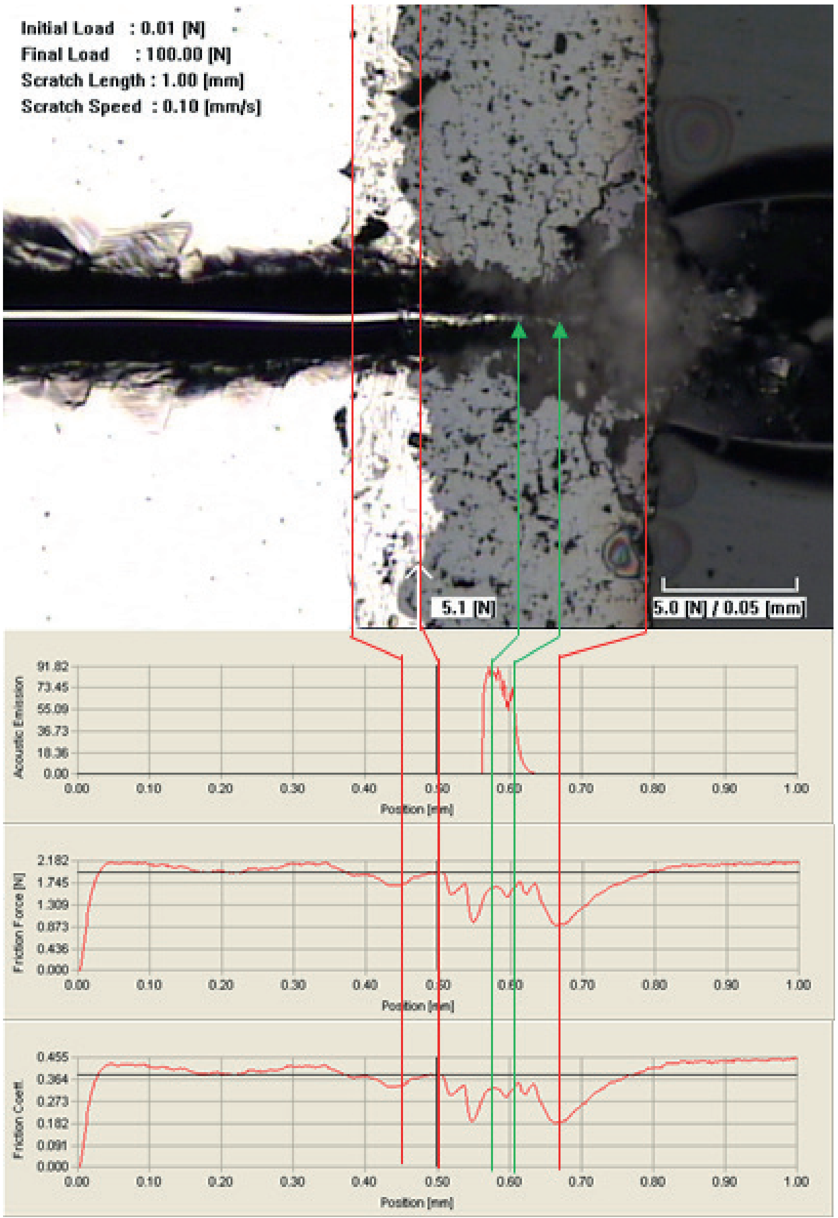


Figure C.12 — Scratch test results of plasma sprayed YSZ coatings scratched at a constant load of 5 N, which tested in Korea

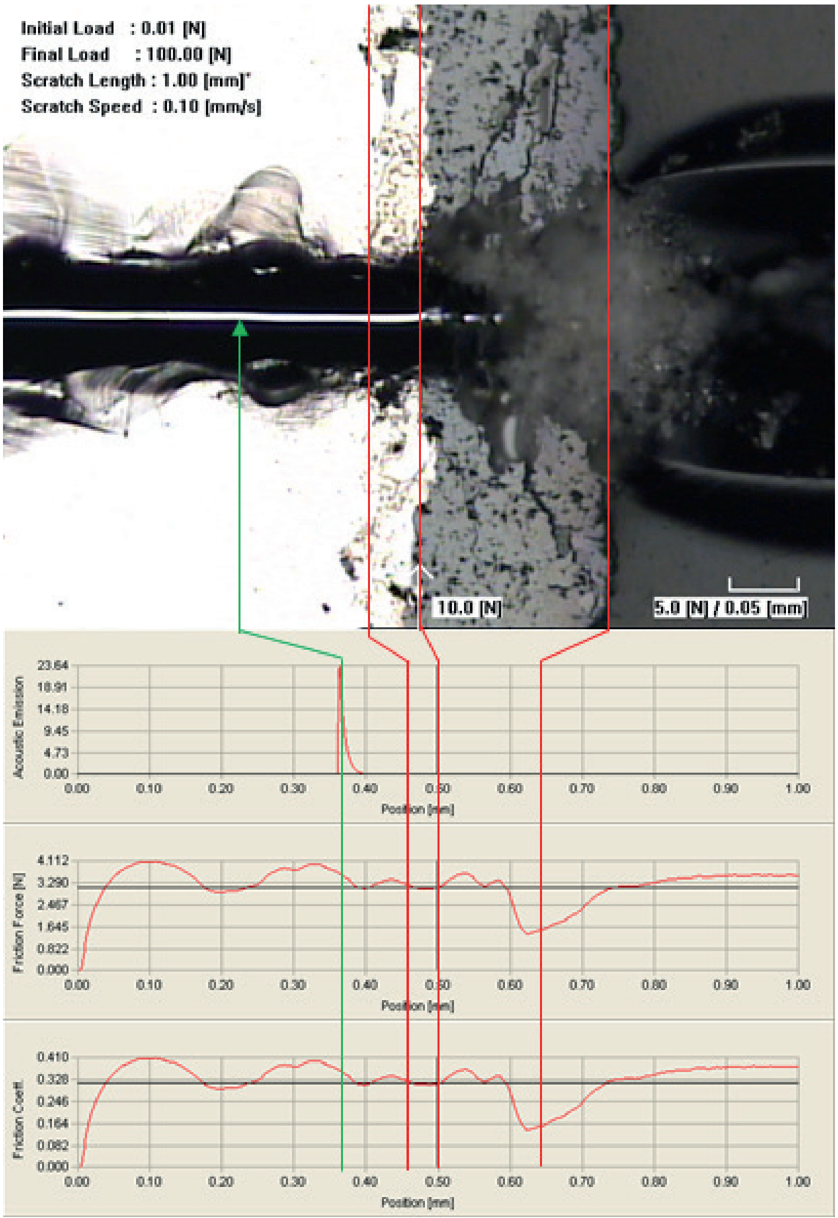


Figure C.13 — Scratch test results of plasma sprayed YSZ coatings scratched at a constant load of 10 N, which tested in Korea

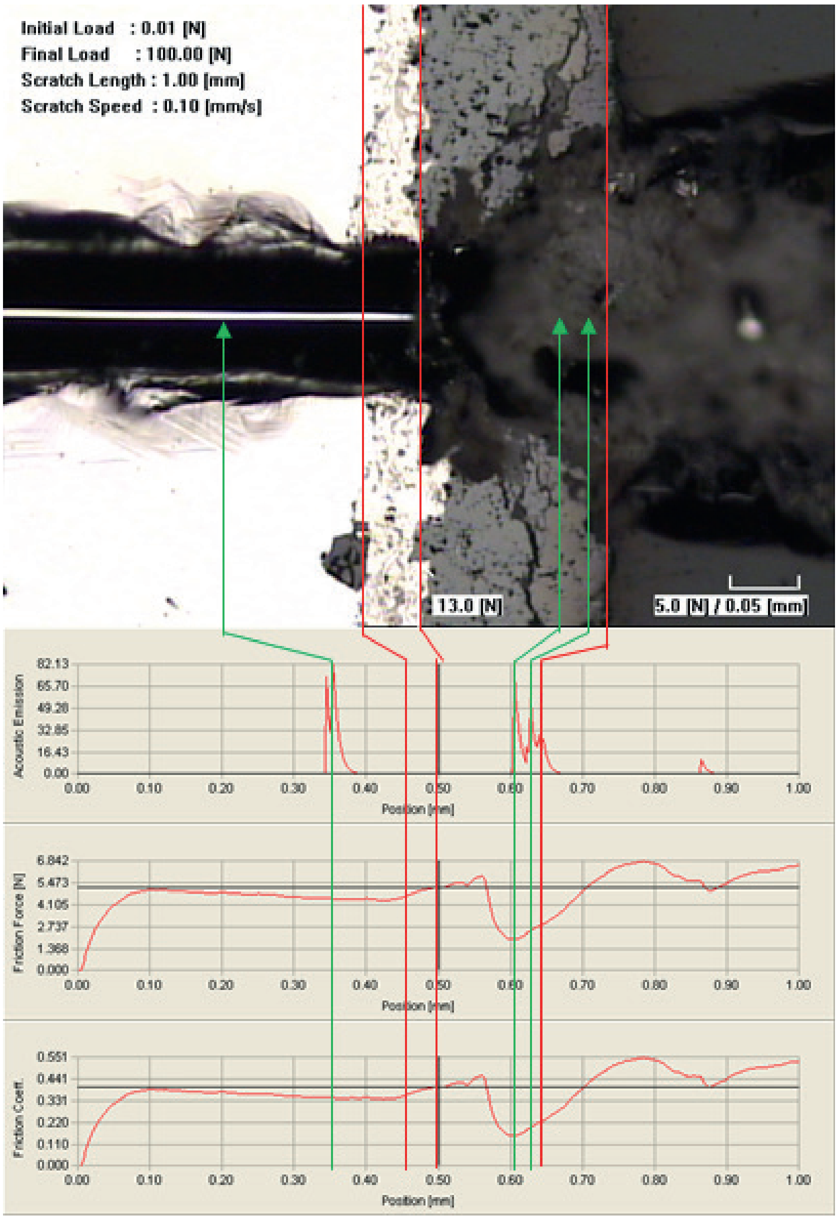


Figure C.14 — Scratch test results of plasma sprayed YSZ coatings scratched at a constant load of 13 N, which tested in Korea

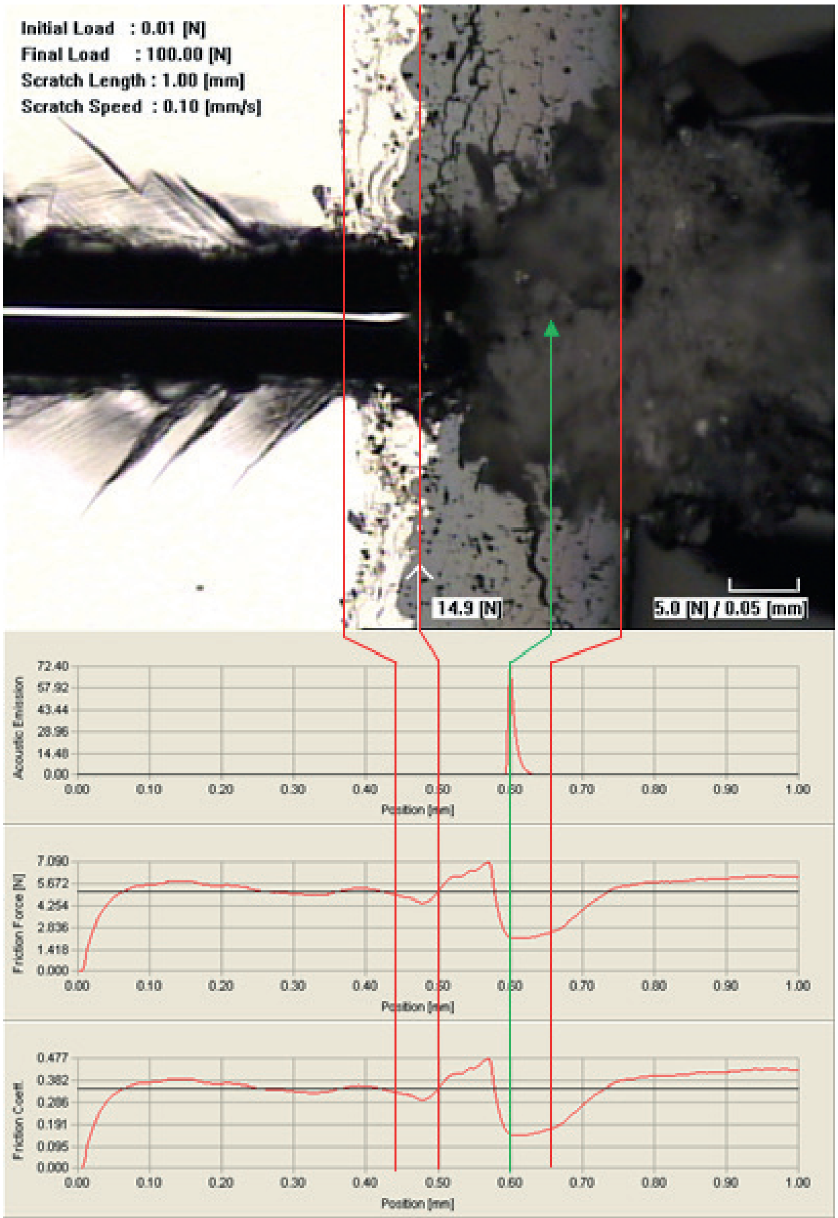


Figure C.15 — Scratch test results of plasma sprayed YSZ coatings scratched at a constant load of 15 N, which tested in Korea

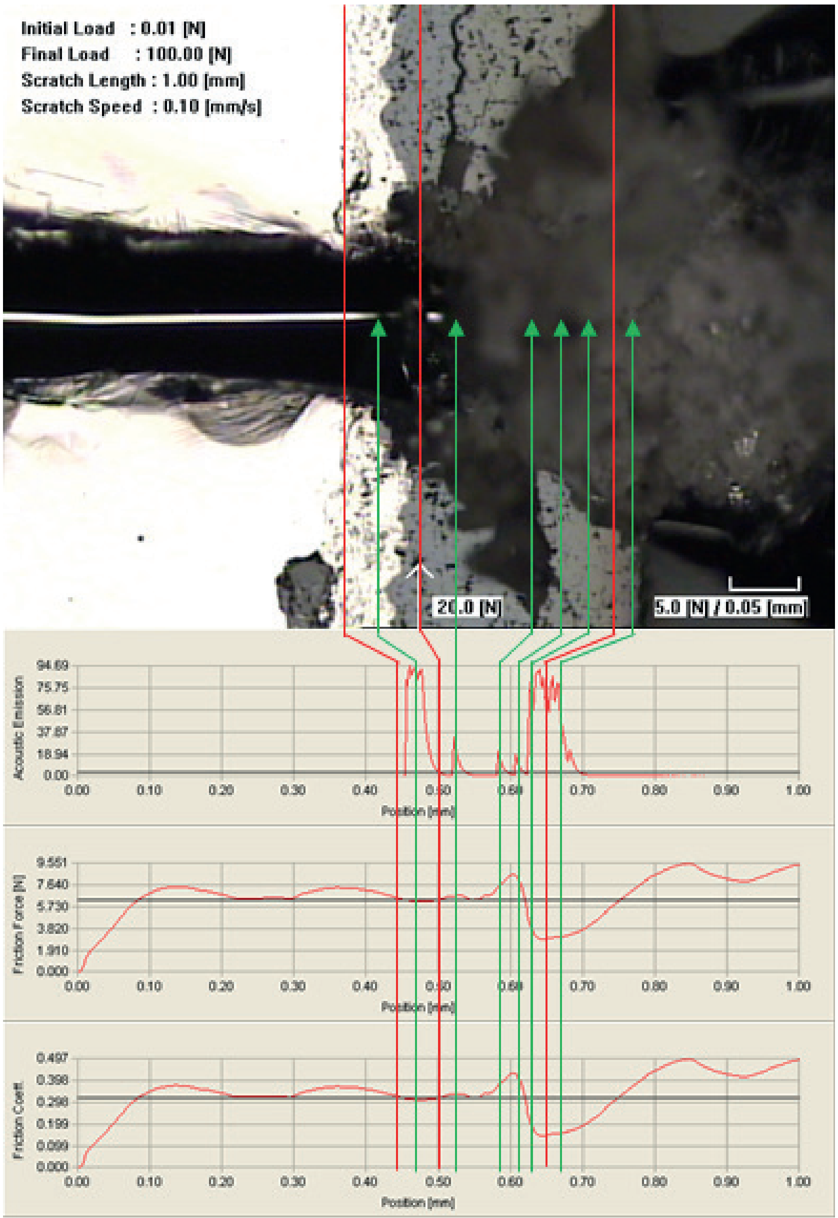


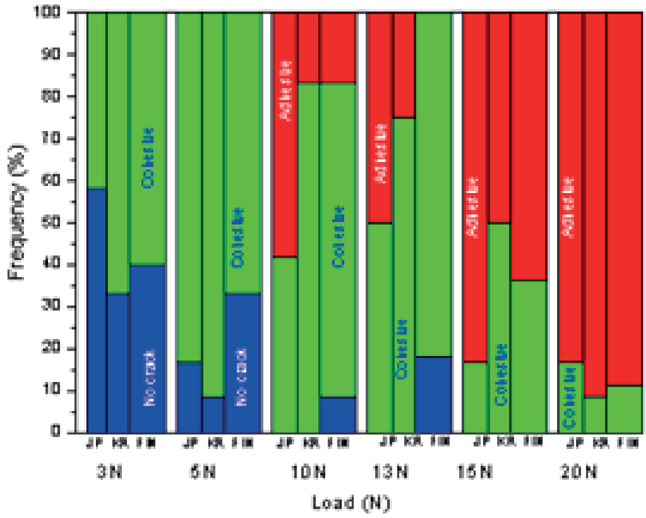
Figure C.16 — Scratch test results of plasma sprayed YSZ coatings scratched at a constant load of 20 N, which tested in Korea

Test results are summarized in Table C.4, which obtained in Korea.

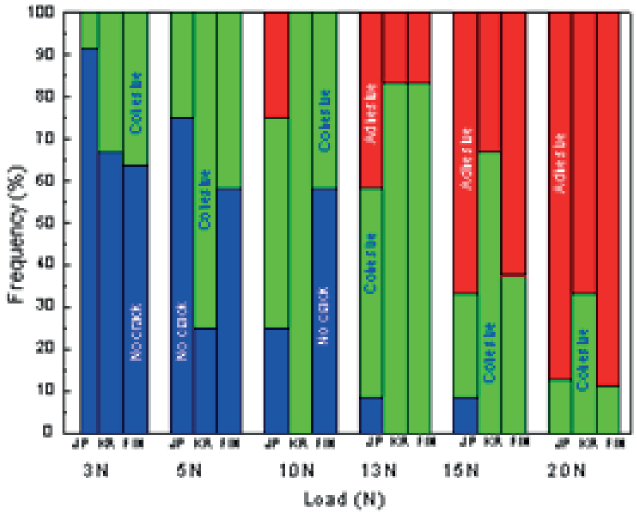
Table C.4 — Summary of the test results obtained in Korea

Coating	Load (N)	No. of scratches	No crack (%)	Cohesive crack (%)	Adhesive crack (%)
TiO_2	3	12	33,3	66,7	0
TiO_2	5	12	8,3	91,7	0
TiO_2	10	12	0	83,3	16,7
TiO_2	13	12	0	75	25
TiO_2	15	12	0	50	50
TiO_2	20	12	0	8,3	91,7
YSZ	3	12	66,7	33,3	0
YSZ	5	12	25	75	0
YSZ	10	12	0	100	0
YSZ	13	12	0	83,3	16,7
YSZ	15	12	0	66,7	33,3
YSZ	20	12	0	33,3	66,7

Figure C.17 shows comparison of the scratch test results which obtained in Finland, Japan, and Korea. From the figure, the data from three countries are fairly coinciding.



a) TiO_2 coating



b) YSZ coating

Figure C.17 — Comparison of the scratch test results which taken in Finland, Japan and Korea

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