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**Comparison of Coating
Adhesion values obtained
from ASTM D4541 and
ASTM C633 Pull-off Tests**

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Comparison of Coating Adhesion values obtained from ASTM D4541 and ASTM C633 Pull-off Tests

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ABSTRACT

Good coating adhesion between coating and substrate is fundamental to the successful application of coatings. In many cases lack of confidence in coating adhesion is the key factor preventing their wider application. Accurate measurement of adhesion is therefore essential for greater exploitation of coatings technology. This report summarises the results obtained from two widely available pull-off adhesion tests. These are the ASTM D4541-02 portable pull-off test and the ASTM C633-01 adhesion test for thermally sprayed coatings. The two techniques were used to measure the adhesion strength of a range of different thermally sprayed and electroplated coatings. Consistent results were obtained from both test methods, however, the techniques were both found to be limited by the strength of the epoxy adhesive used to bond pull-off stubs to the coating.

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1 INTRODUCTION

Surface coatings offer engineers the opportunity to modify the surface properties of a component, independently of the bulk material. The availability of cost effective coating techniques such as thermal spraying and electroplating enables coatings to be used for a diverse range of applications. However, the over-riding criterion in all applications is the same, the coating must adhere to the substrate. Reliable adhesion test methods are therefore essential to guarantee the quality of a coating. This report summarises the results obtained from two widely available pull-off adhesion tests. These are the ASTM D4541-02¹ pull-off test and the ASTM C633-01² adhesion test method. These techniques have been used to measure the adhesion strength of a range of seven different thermally sprayed and electroplated coatings.

2 EXPERIMENTAL PROCEDURE

2.1 ASTM D4541 ADHESION TEST

The ASTM D4541 adhesion tests¹ are conducted by attaching aluminium stubs (13 mm diameter) to the coating using a high strength epoxy adhesive. The force required to pull the stubs from the substrate is measured and can be used to calculate the adhesive strength of the coating to the substrate.

The adhesive used in these tests was a modified polyamide-epoxy adhesive film FM1000 produced by Cyanamid and supplied by Metcut, Cincinnati, U.S.A. Elcometer supplied the stubs for the pull-off test ready grit-blasted for better mechanical bonding. The stubs were cleaned prior to use by washing the surface in isopropanol. A sheet of the adhesive film was cut to the dimensions of the stub and placed between the stub and the coating. The specimens were placed into an oven and a mass sufficient to produce a pressure of 0.3 MPa placed on top of the stubs. The adhesive was cured in the oven at 175°C for 60 minutes.

The pull-off tests were conducted using a commercially available Elcometer Patti 110 test rig with a F20 piston (Figure 1). The pull-off stub was attached to the top platen of the rig with a screw thread, so that it was free to move relative to the lower platen. The stub was then pulled from the substrate by inflating a pneumatic bladder that pushes the two platens apart. The peak pneumatic pressure at failure was recorded and converted to a failure stress using look-up tables supplied with the equipment. The fractured surfaces were then visually examined to determine the location of the failure in the specimen.

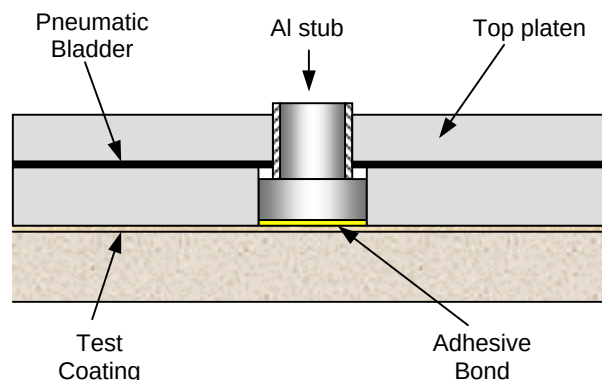


Figure 1 Schematic Representation of the ASTM D4541-02 Pull-Off Test¹

Equipment

2.2 ASTM C633 ADHESION TEST

The ASTM C633 adhesion test² involves coating one end of a cylindrical block with the coating under investigation, bonding the coated face to another block that acts as a pull-off stub and applying a tensile force to the whole assembly using a tensile testing machine (Figure 2).

The specimens used consist of two circular blocks 25 mm in diameter and 38 mm long. One end of each block was adapted so that it could be fixed into a self-aligning tensile testing machine. The other end of each block was machined so that the end faces were parallel to each other and normal to the loading axis. The flat end of the lower block was then coated and bonded to the upper block. The adhesive used to bond the blocks together was the same adhesive as that used in the ASTM D4541 tests (FM1000). Prior to bonding the block used as the pull-off stub was grit blasted using fine silica grit and the surface cleaned using isopropanol. The adhesive was cured at 175°C for 60 minutes whilst applying a pressure of 0.3 MPa.

The whole assembly was fitted into a tensile test machine with the coated block attached to the lower grips and the pull-off stub attached to the upper grips (Figure 2). A tensile load was applied to the test piece by increasing the displacement at a constant rate of 1.0 mm/min until rupture occurred. The maximum load that was applied to the specimen was recorded. The failure stress was determined by dividing the maximum load by the cross-sectional area of the specimen. The fractured surfaces were then visually examined to determine the location of the failure in the specimen.

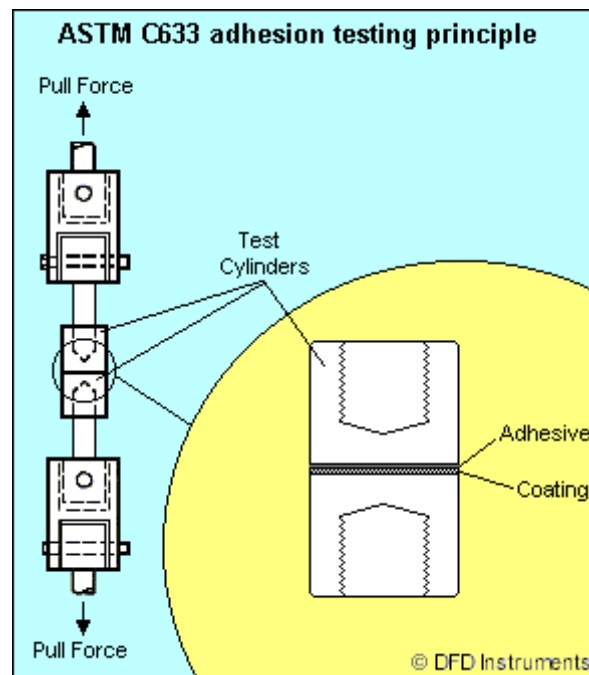


Figure 2 Schematic representation of ASTM C633 Adhesion Test Equipment²

3 SPECIMENS

A range of seven thermally sprayed or electroplated coatings were examined in this study. Specimen preparation and coating conditions were the same for both test methods. However, the coatings for the ASTM D4541 specimens were applied to 50×50 mm stainless plates whereas the coatings for the ASTM C633 specimens were applied to the flat end of 25 mm diameter stainless blocks (as specified in the standard²). Details of the coating material, coating thickness, substrate preparation and the test methods used are summarised in Table 1.

Table 1 Specimen details, coating material, coating thickness and substrate preparation.

Coatings	Fine grit blast (F60 Alumina grit)			Coarse grit blast (NK20 Alumina grit)		
	100	200	300	100	200	300
Hydroxyapatite	-	-	-	-	-	B
Alumina	-	B	-	-	-	-
Cr ₃ C ₂ – NiCr	B	B	B	B	B	B
WC-Co	D	D	D	D	D	D
WC-Co-Cr	B	B	B	B	B	B
Apticote	-	-	-	-	-	B
Paxair	-	-	-	-	-	D

Note: D = ASTM D4541 adhesion test, B = Both ASTM D4541 and C633 adhesion tests

4 RESULTS AND DISCUSSION

Values for the failure stress of each coating are given in Tables 2-8, together with the location of the failure within the specimen. Failure location is important as it indicates whether the failure stress corresponds to the adhesive or cohesive strength of the coating. If fracture occurs along the coating-substrate interface the failure stress represents the adhesive strength of the coating. If failure occurs in the coating this indicates that the internal cohesive strength is less than the adhesive strength and that the failure stress represents the cohesive strength of the coating. If failure occurs in the epoxy adhesive it is not possible to determine an exact value for the strength of the coating. However, it can reasonably be assumed that both the adhesive and cohesive strength of the coating are both greater than the failure stress.

Repeatable values for the adhesive strength of the hydroxyapatite (3.4 ± 1.0 MPa) and alumina (58.5 ± 1.0 MPa) coatings were obtained using both ASTM adhesion test methods. Examination of the fractured specimen surfaces (Figures 3- 5) revealed that these coatings fractured along the coating/substrate interface completely removing the coating from the substrate. This confirms that the mode of failure was adhesive rupture and that the failure stress represents the adhesive strength of the coating.

Visual examination of the chromium carbide specimens revealed that with coarse grit-blasted substrates the coatings failed along the coating/substrate interface (Figure 6a)

whereas with fine grit-blasted substrates it was the epoxy adhesive that failed (Figure 6b). These different failure modes indicate that although the chromium carbide coatings all failed at similar stresses (Table 4) the adhesive strength of the coatings on the fine grit blast substrates may in fact be higher as it is the epoxy rather than the coating that has failed.

Inspection of the tungsten carbide specimens revealed that the epoxy adhesive failed before the coating in each of the tests (Table 5). This is clearly shown in Figure 7 where the white epoxy adhesive is visible on both the coated specimen and the pull-off stub. It can be concluded from these results that the adhesive strength of the coatings is greater than that of the epoxy.

Examining the values of failure stress obtained in tests where the epoxy adhesive has failed (Tables 4 and 5) it can be seen that the strength of the epoxy is lower in the ASTM D4541 test than it is in the ASTM C633 test. As the adhesive used in both tests was identical it would appear that it is the surface of the pull-off stubs that is responsible for this difference. In the ASTM D4541 test the stubs are supplied already grit-blasted and were simply cleaned before use, whereas in the ASTM C633 test the stubs were grit-blasted immediately before to use. It would therefore appear that grit-blasting the stubs prior to curing the adhesive may improve the strength of the adhesive, possibly by reducing contamination on the surface of the stubs.

Failure of the epoxy adhesive was also observed in the Apticote coatings (Table 7). Visual inspection of these specimens revealed that bubbles were present in the epoxy adhesive (Figure 8). Microscopic pores are known to exist in the surface of Apticote coatings. Therefore, a probable explanation for these bubbles is that moisture trapped in these pores has escaped while the epoxy is curing forming the bubbles. A method to prevent these bubbles occurring would be to preheat the specimens in an oven at over 100 °C to remove the moisture from the coating.

The final set of specimens to be examined were the Praxair coatings (Table 8). Inspection of the fracture surfaces revealed that the coatings had fractured between different layers within the coating. The failure stress in these tests therefore corresponds to the cohesive strength of the coating.

4.1 HYDROXYAPITITE COATINGS

Table 2 Adhesion strength of hydroxyapatite coatings measured using both ASTM adhesion test methods

	ASTM D4541 adhesion test		ASTM C633 adhesion test	
	Failure Stress (MPa)	Failure location	Failure Stress (MPa)	Failure location
Test 1	4.1	Coating/Substrate Interface	3.1	Coating/Substrate Interface
Test 2	3.8	Coating/Substrate Interface	2.7	Coating/Substrate Interface

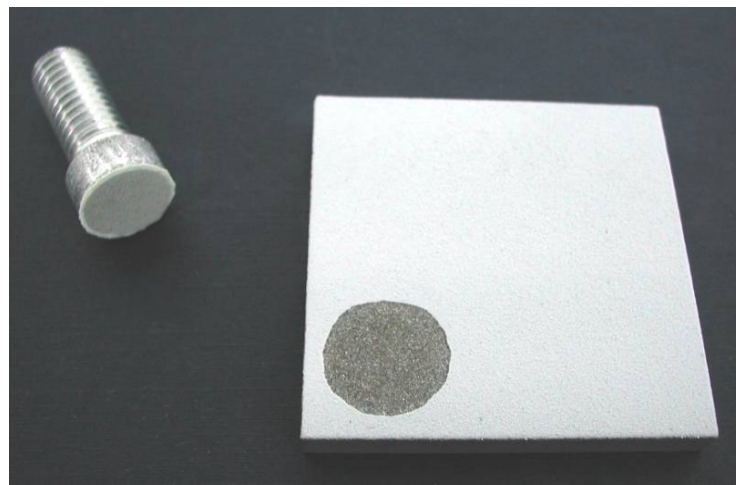


Figure 3 Hydroxyapatite coating was completely removed from the substrate in the ASTM D4541 adhesion test.

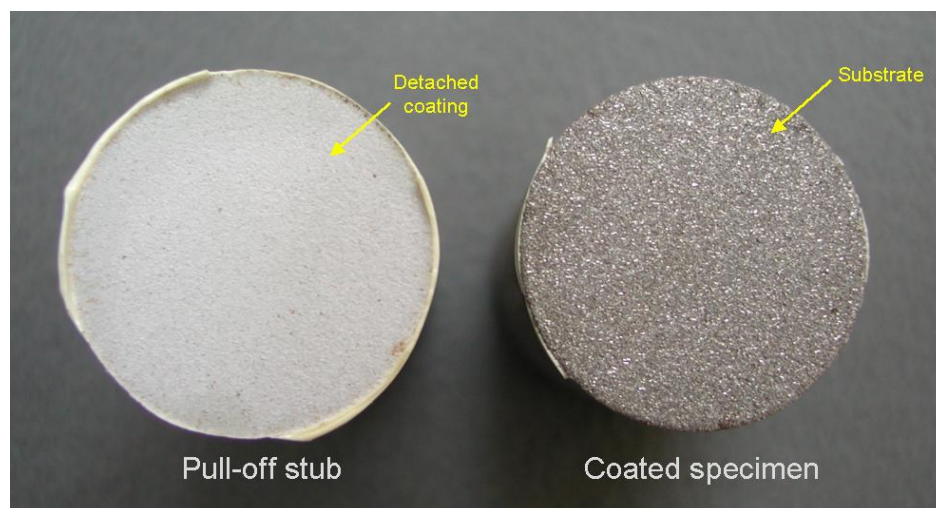


Figure 4 Hydroxyapatite coating completely removed from the substrate in the ASTM C633 adhesion test.

4.2 ALUMINA COATINGS

Table 3 Adhesion strength of alumina coating (Al_2O_3) on a fine grit blasted surface measured using both ASTM test methods

Thickness	ASTM D4541 adhesion test		ASTM C633 adhesion test	
(μm)	Failure Stress (MPa)	Failure location	Failure Stress (MPa)	Failure location
200	58	Coating/Substrate Interface	59	Coating/Substrate Interface

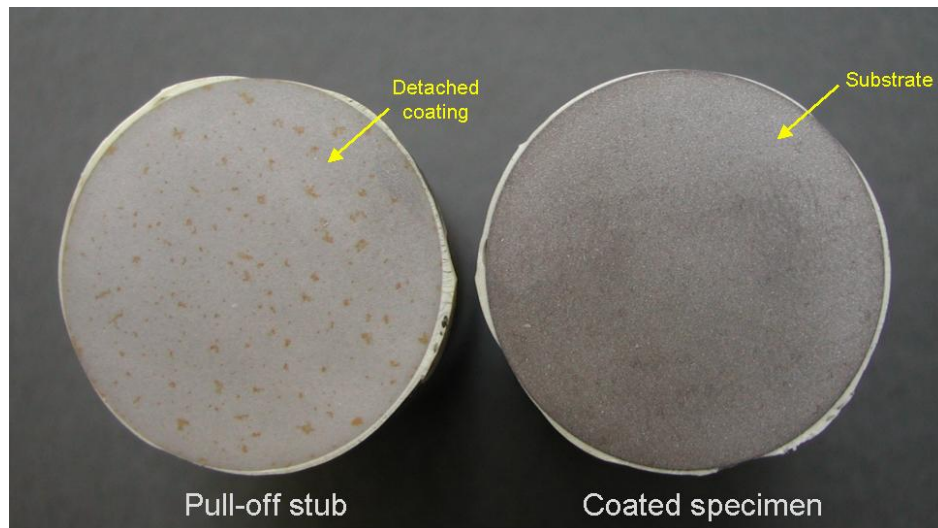


Figure 5 Alumina coating completely removed from the substrate in the ASTM C633 adhesion test.

4.3 CHROMIUM CARBIDE COATINGS

Table 4 Test results for chromium carbide (Cr_3C_2 - NiCr) on coarse and fine grit blasted surfaces measured using both ASTM adhesion test methods

Thickness (μm)	ASTM D4541 adhesion test		ASTM C633 adhesion test	
	Failure Stress (MPa)	Failure location	Failure Stress (MPa)	Failure location
<i>Coarse grit blasted substrate</i>				
100	72	Adhesive	92	Coating/substrate
200	67	Adhesive	91	Coating/substrate
300	73	Adhesive	91	Coating/substrate
<i>Fine grit blasted substrate</i>				
100	71	Adhesive	87	Adhesive
200	72	Adhesive	91	Adhesive
300	79	Adhesive	85	Adhesive

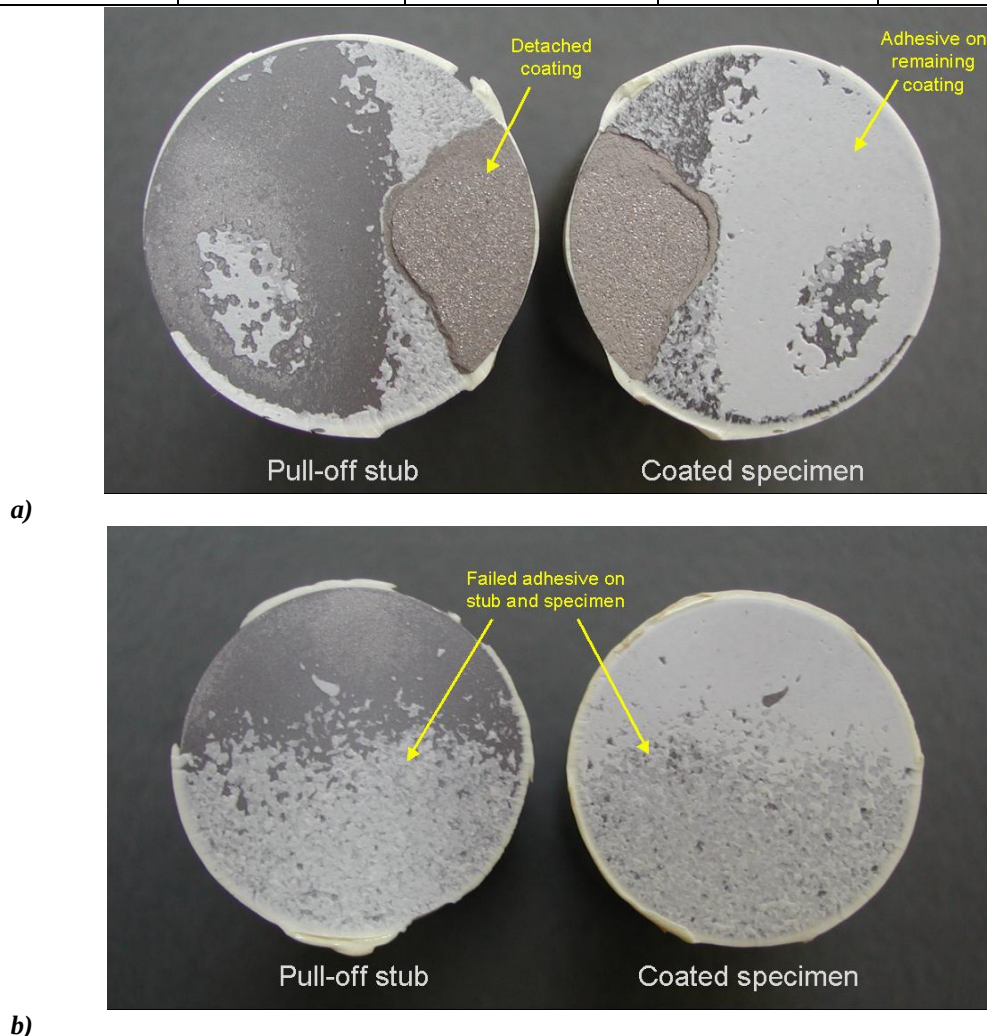


Figure 6 ASTM C633 test specimens with chromium carbide coatings:
a) coarse grit blasted substrate partly detach from the substrate
b) fine grit blasted substrates adhered to the substrate

4.4 TUNGSTEN CARBIDE COATINGS

Table 5 Adhesion test results for tungsten carbide (WC-Co-Cr) on coarse and fine grit blasted surfaces measured using the ASTM D4541 test method

Thickness (μm)	ASTM D4541 adhesion test	
	Failure Stress (MPa)	Failure location
<i>Fine grit blasted substrate</i>		
100	71	Adhesive
200	77	Adhesive
300	70	Adhesive
<i>Coarse grit blasted substrate</i>		
100	80	Adhesive
200	70	Adhesive
300	76	Adhesive

Table 6 Test results for Tungsten Carbide (WC-Co) on coarse grit blasted surfaces measured using both ASTM adhesion test methods

Thickness (μm)	ASTM D4541 adhesion test		ASTM C633 adhesion test	
	Failure Stress (MPa)	Failure location	Failure Stress (MPa)	Failure location
<i>Fine grit blasted substrate</i>				
100	75	Adhesive	81	Adhesive
200	79	Adhesive	89	Adhesive
300	82	Adhesive	92	Adhesive
<i>Coarse grit blasted substrate</i>				
100	76	Adhesive	86	Adhesive
200	75	Adhesive	86	Adhesive
300	82	Adhesive	95	Adhesive

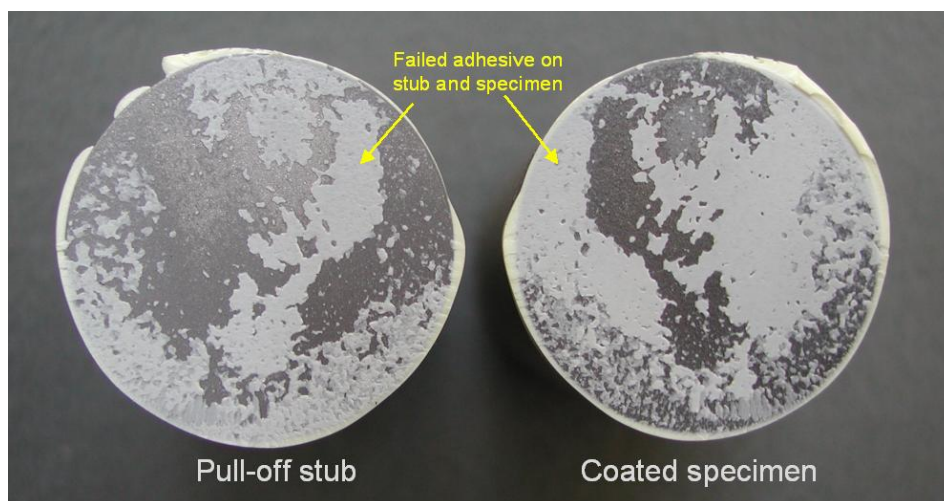


Figure 7 Coating adhesive strength of the tungsten carbide (WC-Co) coating is greater than that of the epoxy adhesive (ASTM C633 adhesion test).

4.5 APTICOTE COATINGS

Table 7 Adhesion test results for the Apticote coatings measured using both ASTM adhesion test methods

	ASTM D4541 adhesion test		ASTM C633 adhesion test	
	Failure Stress (MPa)	Failure location	Failure Stress (MPa)	Failure location
Test 1	69	Adhesive	64	Adhesive
Test 2	60	Adhesive	41	Adhesive

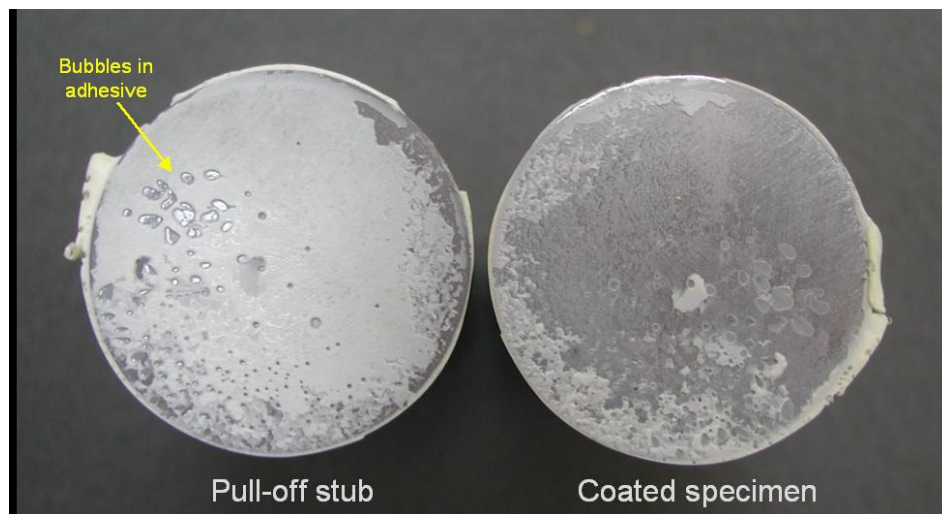


Figure 8 Moisture that escaped from pores in the coating is believed to have produced these bubbles in the adhesive while it was cured.

4.6 PRAXAIR COATINGS

Table 8 Cohesive strength of the Praxair coatings obtained using the ASTM D4541 adhesion test

	ASTM D4541 adhesion test	
	Failure Stress (MPa)	Failure location
Test 1	42	Cohesive within coating
Test 2	39	Cohesive within coating

5 CONCLUSIONS

Comparable values for the adhesion strength of the hydroxyapatite and alumina coatings were obtained from both ASTM adhesion test methods.

Variations in the adhesion strength of the chromium carbide coatings caused by grit blasting of the stubs were detected using the ASTM C633 adhesion test method.

The strength of the epoxy adhesive used in both ASTM test methods was insufficient to measure the adhesion strength of the tungsten carbide coatings. However, these test methods can still be used for proof testing the adhesion strength of the coatings up to 95 MPa.

Moisture trapped in the surface of the Apticote coatings appears to have escaped while curing forming small bubbles in the epoxy adhesive. It is suggested that these specimens should be preheated at over 100 °C prior to curing to remove the moisture.

Test results from the Praxair specimens indicate that these coatings fail cohesively rather than adhesively.

REFERENCE

- 1 ASTM D4541-02, "Standard Test method for Pull-off Strength of Coatings using Portable Adhesion Testers", ASTM 2002.
- 2 ASTM C633-01, "Standard Test Method for Adhesion or Cohesive Strength of Flame-Sprayed Coatings", ASTM 2001.