

Measurement Note

Measurement of Coating Adhesion Using the Rockwell Indentation Technique

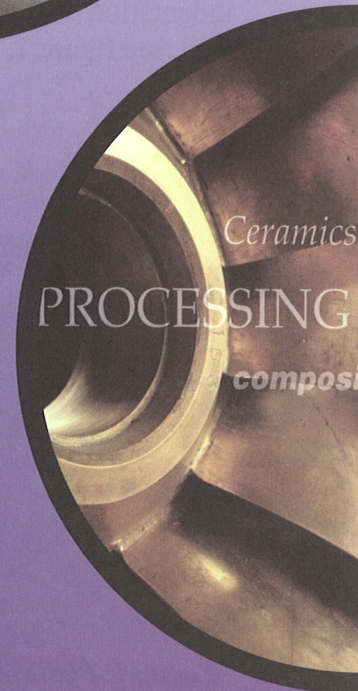
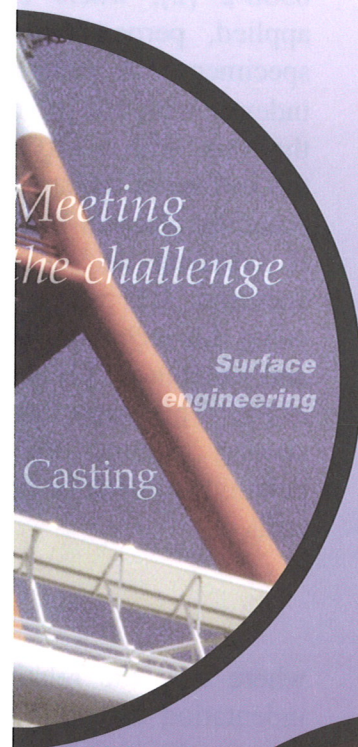
The use of coatings is increasingly of interest to industry. Application of a coating to an industrial component can help provide protection from corrosion, wear, abrasion, erosion and chemical attack etc., providing an increased lifetime to the component. There are potential problems that can limit the life of a coating, for example adhesion and ductility, therefore, a quick and easy, reliable test to investigate these mechanical properties is desirable.

Current adhesion tests, which include pull out, push in, four-point bend and butt tension tests, are generally destructive. One simple test is the Rockwell indentation technique, which can be used to assess the performance of coating adhesion to the substrate, causing minimal damage. This can also provide additional information such as whether the coating will fail in a brittle or ductile manner when indented. It is a quick and easy technique and only destructive to a very small area that may not affect the function of the component. It can usually be used on real components and does not require the preparation of special test coupons. This form of test is ideal as a method of ranking the integrity of coatings.

This note describes the Rockwell indentation technique, illustrating how a classification can be applied to the indent. Results from a recent DTI funded project are presented, showing how the adhesion of different coatings can be ranked. The effect of substrate pre-treatment is also examined.

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Description of Rockwell Adhesion Test

Experimental Method

A Rockwell hardness testing machine conforming to EN ISO 6508-1 [1] was used. An indentation was made, conforming to EN ISO 6508-2 [2], where a point load was slowly applied, perpendicular to the surface of the specimen. A spherical Rockwell C diamond indenter with 200 μm radius was used for each of the tests, with a 150 kg load. After indentation the surface of the specimen was examined in the scanning electron microscope (SEM) to identify if cracking or delamination of the coating occurred. It is possible to fully quantify adhesion using this technique, originally derived by Drory and Hutchinson [3] and simplified by Osgerby *et al* [4]. This states that if partial spallation of the coating occurs, the size of the adhesive delamination can be described by the ratio of the radii,

$$r/R \quad (1)$$

where r is the relative size of the radius of indentation and R is the radius of delaminated area, see figure 1.

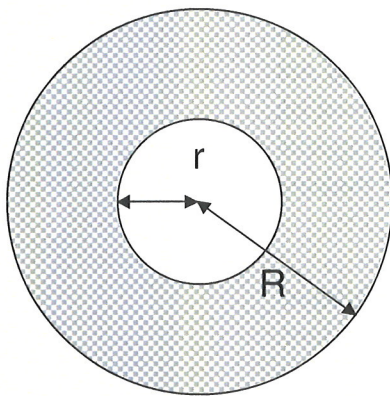


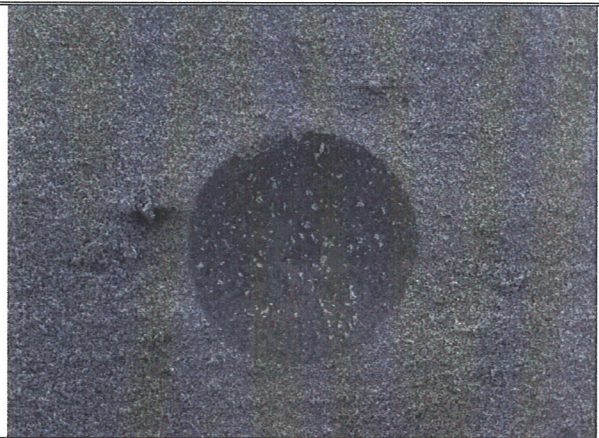
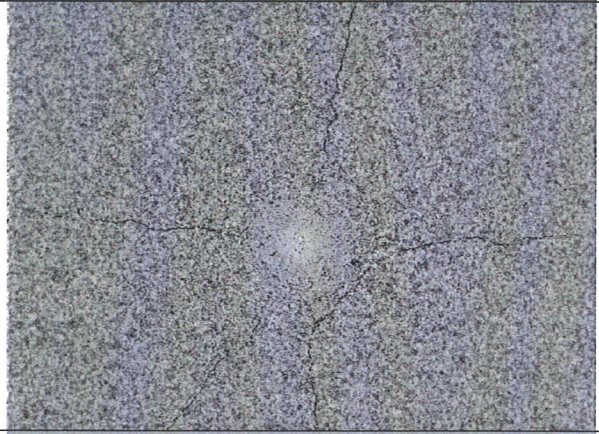
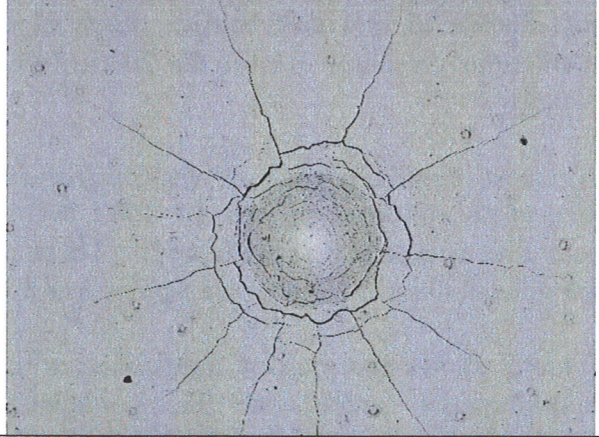
Figure 1: Plan view of indentation showing central indentation and surrounding delamination

Classification of Results

The results can be classified in four main ways, see table 1. This classification gives a relative scale where coatings progress from class 0 where all deformation is ductile, through classes 1 and 2 to class 3 where considerable amounts of delamination occur.

Class 0 indicates ductility – cracking and spallation are not observed and the adhesion is good. Cracking occurs around the indentation in class 1 with radial cracks observed but without spallation. Class 2 has extensive radial and/or circumferential cracking around the periphery of the indentation, partial spallation may also occur. When adhesion is poor, complete delamination of the coating occurs, this is class 3.

Table 1: Classification of Indentation

Class	Observation	Example
0	No cracking or spallation. In this case the adhesion is good and the coating is ductile.	
1	Cracking around indentation without spallation. Here radial cracks are observed spreading into the coating away from the indentation.	
2	Cracking around the periphery of the indentation with radial and/or circumferential cracking observed. Partial spallation may also occur.	
3	Complete delamination of the coating around the indentation – this shows poor adhesion.	

Case Study

Six materials were tested:

- Plasma sprayed hydroxy-apatite
- HVOF diamond jet chromium carbide
- HVOF JP 5000 WC/Co
- Top gun alumina
- Chromium plate
- Co-deposited electroplate.

For each material, the coating was applied at three nominal thicknesses; 100, 200 and 300 μm . Additionally, the effect of surface pre-treatments on adhesion was investigated by comparing the adhesion of diamond jet chromium carbide and JP 5000 WC/Co on substrates which had been shot blasted using a fine and coarse shot. The Rockwell indent was made on each sample and examined using the scanning electron microscope.

Results

Rockwell indentations were made on each of the coatings at room temperature. The results are presented in order of class, ranking from the most ductile to the most brittle. Each of the indentations presented are for the 200 μm thick coatings.

The most ductile coating is the co-deposited electroplate followed by the JP 5000 WC/Co (both fine and coarse shot blasted). These are examples of class 1 damage, see figures 2 and 3.

Figure 2 shows the area of indentation with a circumferential crack on the co-deposited electroplate coating.

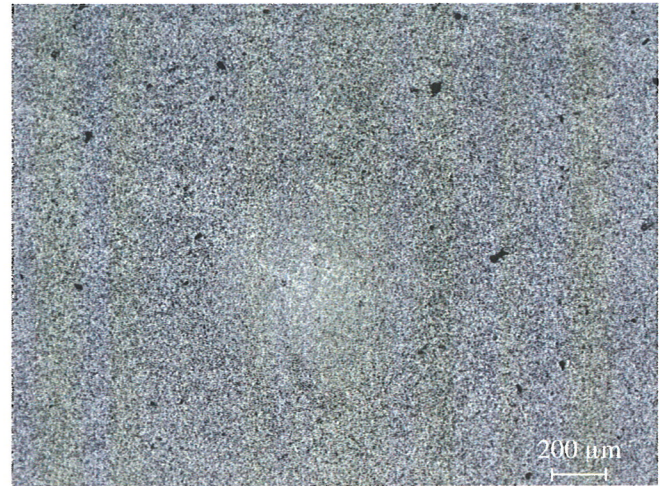
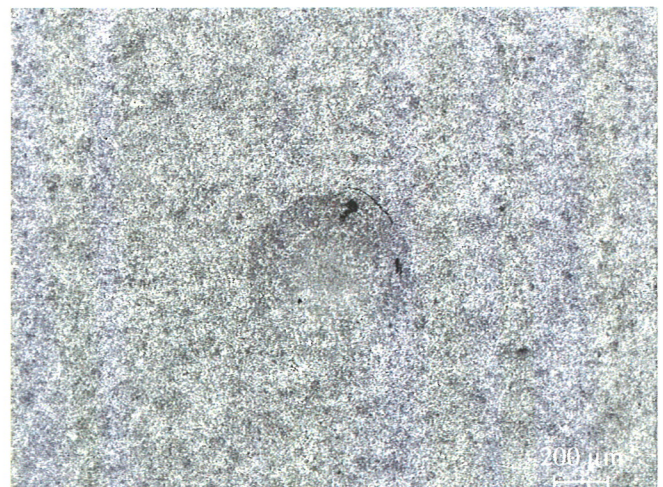
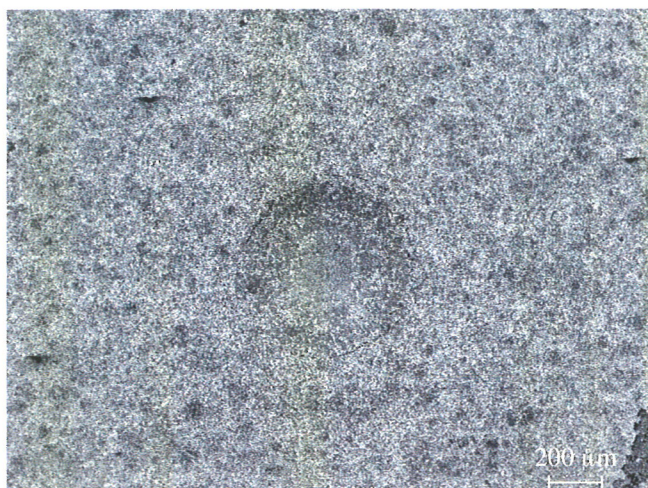


Figure 2: Co-deposited electroplate

The JP 5000 WC/Co also showed class 1 damage for both the fine and coarse shot blast treatment, see figure 3 (a) and (b).



3a



3b

Figure 3: JP 5000 WC/Co – (a) fine and (b) coarse shot

No noticeable effects are observed from the different shot blasting processes.

Figures 4 to 6 are examples of class 2 damage. Extensive damage is observed around the area of each indentation but with no delamination.

Considerable cracking around the periphery of indentation is shown in figure 4 on the top gun alumina coating.

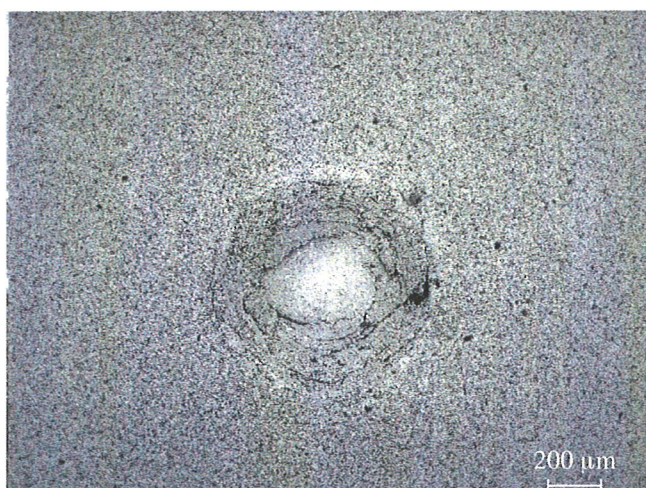
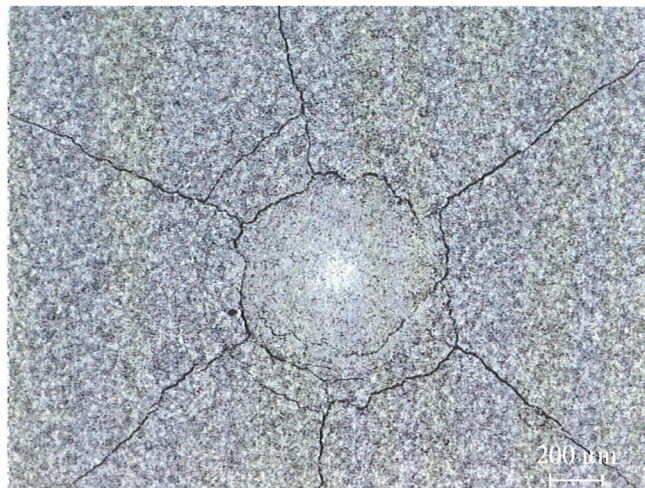
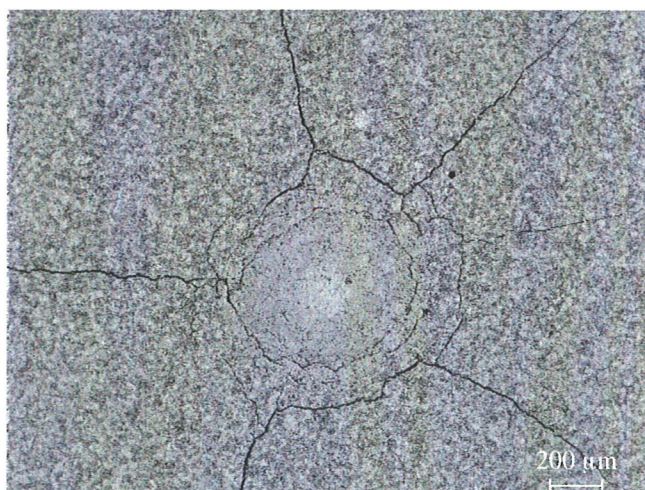


Figure 4: Top gun alumina

The diamond jet coatings both appeared to be brittle. Again there is no effect from the different shot blasting processes, see figures 5 (a) and (b). These coatings are brittle with both radial and circumferential cracking observed.



5a



5b

Figure 5: Diamond jet chromium carbide – (a) fine and (b) coarse shot blast

The chrome plate coating was extremely brittle, with extensive radial and circumferential cracking observed, shown in figure 6. Radial and circumferential cracking extend well beyond the area of indentation.

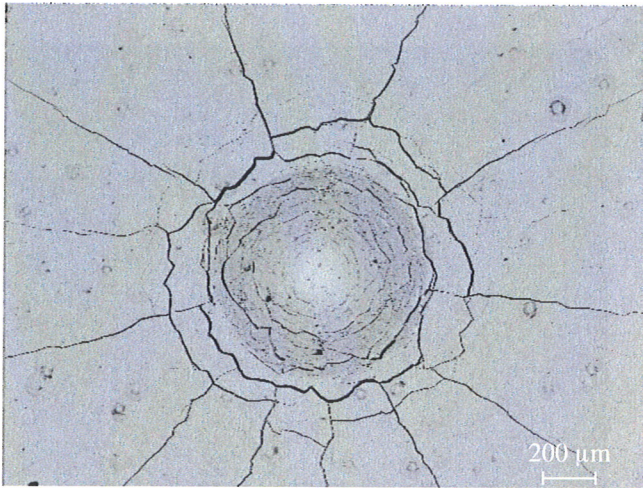


Figure 6: Chrome plate

The hydroxy apatite also showed extensive damage, see figure 7. There was an area around the coating where the damage was extremely severe, this is identified as class 2/3 damage as complete delamination has not occurred.

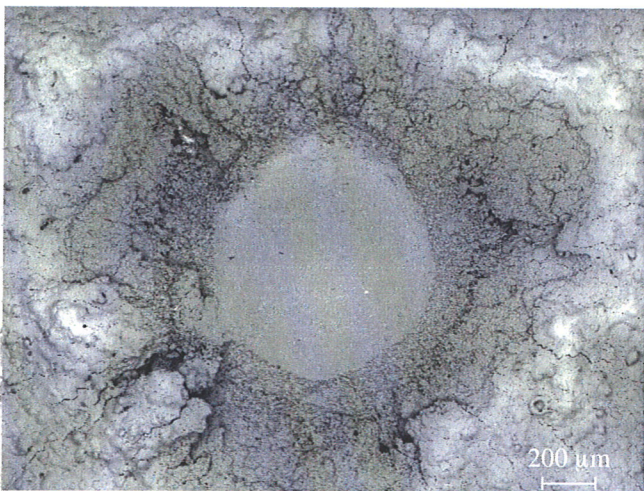


Figure 7: Hydroxy apatite

Conclusions

Despite cracking of the coating, no delamination occurred for any of the tests, i.e. no class 3 cracking was observed demonstrating good adhesion was maintained for each coating. Some coatings appeared to be much more brittle, with radial and circumferential cracking observed.

In terms of the adhesion properties, the coatings can be ranked in the following order:

- Co-deposited electroplate
- JP 5000 WC/Co
(both fine and coarse shot blast)
- Top gun alumina
- Diamond jet chromium
(both fine and coarse shot blast)
- Hydroxy apatite
- Chrome plate

Shot blasting did not affect coating ductility, and surprisingly did not affect adhesion either.

References

- [1] BS EN ISO 6508-1, 'Metallic materials – Rockwell hardness test', 1999
- [2] BS EN ISO 6508-2, 'Metallic materials – Rockwell hardness test', 1999
- [3] M. D. Drory and J. W. Hutchinson, Proc. R. Soc. Lond. A 1996 **452** 2319-2341
- [4] S. Osgerby, S. Saunders and J. Banks, NPL Measurement Note, CMMT(MN)26 'Scale and Coating Adhesion: A Procedure for Laboratory Tests', June 1998

Acknowledgements

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