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ION VAPOUR DEPOSITED (IVD) ALUMINIUM COATINGS FOR THE CORROSION PROTECTION OF HIGH STRENGTH STEEL

by

B.R.W. HINTON AND W.J. POLLOCK

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SUMMARY

Extensive metallographic, corrosion and hydrogen embrittlement tests have been carried out with high strength steel sheet coated with ion vapour deposited (IVD) aluminium. Metallography showed that the thickness of the as-deposited IVD coatings was not uniform and that the coatings contained extensive microporosity. The application of glass bead blasting after coating significantly reduced the porosity but did not remove it completely. The IVD coatings were found to be effective in preventing corrosion of the steel substrate provided the coating was sufficiently thick and its porosity low. Exposure of IVD coated steel to aqueous enviroments was found to produce hydrogen embrittlement of the high strength steel substrate under certain conditions.

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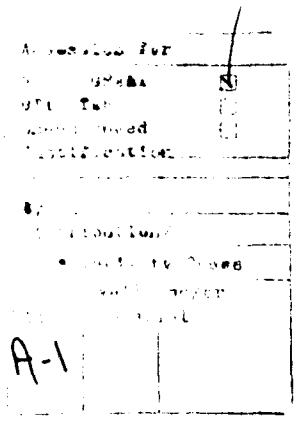

1. INTRODUCTION

Many high strength steel aircraft components are protected from corrosion by cadmium coatings. These coatings may be deposited either electrolytically or by vapour deposition in a vacuum.

Cadmium possesses a low rate of corrosion and is therefore a very durable coating. It also provides sacrificial corrosion protection to the underlying exposed steel in the event that the cadmium is damaged or removed. The RAAF periodically repairs and overhauls vacuum cadmium plated high strength steel components. As no vacuum plating facilities exist in Australia the components must be sent overseas. This is both expensive and time-consuming. An alternative approach is to use low hydrogen embrittlement electroplated cadmium (LHE). However, there is concern as to whether or not the LHE processing is of a suitable and reproducible standard. Cadmium-titanium plating is another possible alternative. However, there is some doubt as to whether cadmium-titanium plating facilities in Australia can meet specification requirements.

An alternative to cadmium plating is an aluminium coating produced by Ion Vapour Deposition (IVD). Hawker de Havilland (Australia) are currently operating an IVD process, "Ivadising", developed by McDonnell Douglas. Various McDonnell Douglas reports (1, 2) suggest that IVD Aluminium is an effective coating for the corrosion protection of high strength steels in aircraft service environments. The availability of this IVD aluminium coating facility offers the RAAF (at least in principle) a method for recoating high strength steel components without excessive time delays and without any hydrogen embrittlement concerns common to electroplating processing. Therefore, the RAAF (4) requested that ARL:

1. Assess and compare the quality of the coatings produced by Hawker de Havilland (Aust.) and those supplied by McDonnell Douglas, particularly in relation to microstructural features and substrate preparation.

2. Test the adhesion of the coatings.

3. Assess the effects of an aggressive environment on the coating in both the undamaged and damaged conditions.

4. Examine the possibility of embrittlement of the steel substrate, as a result of exposure of the coating to aggressive environments, including paint strippers and chemical strippers.

2. EXPERIMENTAL DETAILS

2.1 Materials and Treatments

2.1.1 Coated sheet samples

Samples of 4130 sheet steel (100 x 25 x 1.1 mm) were processed by Hawker de Havilland (HD) in accordance with Military Specification MIL-C-83488A. They each received four layers of ion vapour deposited (IVD) aluminium while held stationary in the IVD chamber. Other samples (150 x 25 x 1.1 mm) were coated by McDonnell Douglas (MD), but were not subjected to glass-bead blasting. The HD samples were received as three separate batches. The samples in the first batch numbered HD 1 to 9 had been coated at the same time, at nine different positions in the IVD chamber as shown on the grid in Fig. 1. A second batch of samples had been glass-bead blasted (B) after various stages of a four-pass deposition process, (I = 1 IVD pass). The coatings on these samples are designated as 4I + B (HD 10), I + B + 3I (HD 11), and I + B + 3I + B (HD 12). The third batch (HD2/---) contained samples which had been treated as follows: 4I, 4I + C, 4I + B, 4I + B + C, where C is treatment after IVD coating with a chromate conversion-coating process, to Military Specification MIL-C-5541.

2.1.2 Bolts

Aircraft quality steel bolts 6.5 mm diameter and 19 mm long were also evaluated. These were of the "Hi-Torque" type and had been coated by McDonnell Douglas using the IVD "Barrel Coating Process".

2.1.3 Tensile specimens

Notched tensile specimens (Fig. 2) made from 9.5 mm round 4340 steel bar, were used for the embrittlement tests. These were austenitised at 815°C for 1 hour, quenched in oil at 40-60°C and then double-tempered at 260°C for 1 plus 1 hours with air-cooling to 20°C between tempers. The notch was prepared by low stress grinding after heat treatment. Four passes of IVD coating followed by glass-bead blasting were applied by Hawker de Havilland.

2.2 Corrosion Tests

Specimens, 25 x 15 x 1.1 mm, cut from the sheet samples were used for corrosion tests. The two cut edges were masked with a proprietary lacquer. A line 15 x 0.5 mm and 0.5 mm deep was scribed onto one surface of some of these specimens before testing, to simulate IVD coating damage.

The aircraft bolts were tested: (i) in the as-received condition, or (ii) after having been torqued in a simulated 7075 alloy structure and removed. The torquing effectively damaged parts of the coating on the bolts.

Most of the corrosion tests were conducted under constant immersion conditions, by suspending specimens on nylon thread in 100 ml of 0.1 NaCl solution (pH 6 to 7) open to the air. (Experience at ARL and elsewhere over many years has shown that this concentration of NaCl produces one of the most corrosive environments for aluminium). Further corrosion tests were carried out under alternate immersion conditions according to ASTM G44, with 10 minutes immersion followed by 50 minutes drying. This environment approximates to the wet/dry conditions experienced by many aircraft components in service.

2.3 Adhesion Tests

The adhesion of the IVD coating to the steel substrate was assessed using two methods. With the first method, a scratch 15 mm long and approximately 0.5 mm wide was scribed into the coated steel sheet with a reproducible force to expose the steel substrate. The edges of the scratch were examined, at a minimum of 8 diameters magnification, for evidence of poor adhesion. A second method involved applying Scotch Adhesive Tape No. 250 to the coating surface over the scratch, then removing the tape with a quick pull and examining for coating removal.

2.4 Tensile Tests

The slow strain rate tensile test was used to determine susceptibility to embrittlement. Details of this technique and the test equipment have been described elsewhere (3). Briefly, it involves testing a tensile specimen at a strain rate of 2×10^{-4} mm s^{-1} in an embrittling environment and recording the fracture stress. The degree of embrittlement is quantified by reductions in the fracture stress when compared with unembrittled specimens. Slow strain rate tests in paint strippers Turco 5351 and Ardrox 2106 were conducted with the central portion of the IVD coated notched specimen immersed in the paint stripper contained in a 40 ml Teflon chamber. Slow strain rate tests were also conducted in air after exposure to 2.5 M sodium hydroxide to remove the IVD coating and also after exposure to 0.1 M NaCl and tap water for 200 hours.

3. RESULTS

3.1 Evaluation of Coating and Substrate

3.1.1 Sheet samples

All Hawker de Havilland (HD) and McDonnell Douglas (MD) as deposited samples had a dull grey appearance (Figure 3) whereas those samples with a bead-blasted finish were shining and bright (Figure 4). Very small but visually detectable depressions were present on the surfaces of almost all of the IVD samples (Figures 3 and 4). Table 1 lists the average diameters and depths of 10 of these depressions. Scanning electron micrographs of HD and MD unbeaded surfaces are shown in Figures 5, 6, and 7. Both samples displayed a well-defined grain structure with a nodular or "cauliflower"-like appearance typical of vapour-deposited metal.

Although both HD and MD coatings were produced by the same process, the grain structures exibited different structural characteristics (c.f. Figures 5 and 7). The grain size of the HD coating is finer than for the MD coating and the MD grains had a feathery appearance. Both structures exhibited significant microporosity. On surfaces blasted with glass-beads, laps and folds were clearly visible as evidence of the deformation resulting from this operation (Figure 8). However, there were many areas which had not been deformed in the beading operation, eg. at B in Figure 8.

3.1.1.1 Coating thickness and microstructure

Coating thickness was measured on metallographic sections taken from three areas of each sample. The coating thickness was generally uniform across any one side of each of the glass-beaded or as deposited HD samples, but often differred in thickness from one side to the other, e.g. HD 3, HD 6, in Table 2. With the as-deposited HD specimens 1 to 9, there was a variation in coating thickness relating to their position in the chamber, as shown in Table 2. The thickness of the glass-beaded IVD coating on sample HD 10 was significantly less than the thickness of the coatings on all other HD samples. Based on the limited number of metallographic sections, it would appear that samples from positions 4 and 5 in the IVD chamber are certainly thinner than samples from other positions. As exhaustive statistical analysis to substantiate this observation was not carried out. IVD coatings on MD specimens were not uniform in thickness. In some areas the coating was non-existent. There was also a large variation from side to side for any one sample, e.g. MD 1 (Table 2).

Differences in the microstructure of the IVD coating occurred according to the origin of the coating (HD or MD) and in relation to the sequence of deposition and glass-beading. The as-deposited IVD coatings HD 1-9 possessed similar microstructure, with no discernible differences relative to their position in the chamber. Samples of both HD and MD IVD as-deposited coatings exhibited columnar grain structures with extensive microporosity. In the HD coatings this porosity was quite fine (approximately 0.1 m) but much wider pores (> 1 m) were noted in many areas. These pores often extended to within a few microns of the steel substrate (Figure 9a). By comparison, (Figure 9b) the porosity in the MD coatings appeared to be coarser, and in some areas, the steel substrate was not coated.

Glass-bead-blasting is used to consolidate the coating and reduce porosity. In general, this did appear to produce the desired effect. However, as mentioned earlier, the bead-blasting process did not eliminate all porosity. Where beading had been carried out after four completed IVD passes (4I + B), significant porosity remained. This resulted because incomplete closure occurred during impact by the beads or due to a very low density of impacts by the beads in isolated areas (Figure 10a). With specimens beaded after the first IVD pass (I + B + 3I), this first layer of Al was almost free from porosity. However, the remaining layers had a columnar grain structure and were very porous (Figure 10b). Beading after the first and final IVD passes appeared to have the greatest effect on reducing the porosity. Nevertheless, there still remained regions where complete closure of the pores had not been achieved (Figure 10c).

3.1.1.2 Steel substrate

The steel substrates below the IVD layer for all HD and MD samples were heavily contaminated. This contamination included sharp angular glassy particles, and what appeared to be remnants of rust (Figure 11). Energy dispersive X-Ray analysis identified silicon, aluminium, iron, manganese and calcium in this contamination. Metallographic sectioning revealed that these contaminated areas of the steel substrate were often associated with local depressions in the IVD coating (Figure 12). The coating in these depressions was frequently much thinner, and sometimes more porous, than neighbouring areas of coating.

3.1.2 Bolts

The bolts had been coated by McDonnell Douglas using an IVD-Barrel Coating Process. In this process the bolts are loaded into a rotating mesh cylinder within the IVD chamber and are coated as they randomly rotate.

Scanning electron microscopy indicated that the coated bolt surfaces contained fine laps, folds and depressions which resulted from the tumbling process. The thickness of the coating, as determined metallographically, varied over the surface of the bolt. Table 3 lists the thicknesses at four positions. The data are from two bolts and represent a spread of five measurements. In many cases, the thickness of the coating on the thread surface was significantly less than the thickness at the crest or root (Figure 13).

The bolt coatings had a very fine grained structure free from any of the microporosity observed with the sheet samples. The steel substrate had also been very well prepared and no surface contamination was detected.

3.1.3 Tensile specimens

Table 4 shows the results of ten measurements of coating thickness along the gauge length of one of the tensile specimens and along the flank of the notch. These data indicate that the coating thickness along the gauge length and within the notch is variable, and that the coating within the notch is significantly thinner than the coating on the gauge length. These data are slightly magnified because these measurements were taken from a longitudinal chord section of the tensile gauge length.

The IVD coating microstructure on the tensile specimen was fine-grained but with some microporosity, particularly in the specimen notch, where the glass-beading process had not been effective in totally consolidating the microstructure. The steel substrate adjacent to the coating was heavily contaminated with glassy angular particles.

3.2 Corrosion Tests

With each of the test specimens, the breakdown of the IVD-Al coating proceeded through a similar sequence of stages. The time to reach each stage after the commencement of testing and the duration of each stage, differed between the constant and alternatively immersed specimens, and also according to whether or not the IVD-Al had been blasted with glass beads.

3.2.1 Sheet specimens

3.2.1.1 Constant Immersion Tests

Within 1 or 2 days of immersion, a white corrosion product formed in the test solution and isolated pits developed in the coating (Figure 14). At this initial stage, pits did not contain corrosion product and were frequently surrounded by a bright halo. In many cases, the steel surface was visible at the base of each pit. Pitting was often associated with pre-existing depressions in the coating.

Pitting is believed to result from rapid dissolution of aluminium at pre-existing porosity or defects, such as depressions in the coating, soon after immersion. Pores behave as crevices with narrow and restricted geometry, and such conditions quickly lead to the development of low pH and high dissolution rates. Once perforation through the coating to the substrate occurs, aluminium will dissolve more rapidly while providing a level of galvanic protection for the exposed steel.

The diameter of the pits and the surrounding halo increased with increasing exposure time. The gradual galvanic consumption of the aluminium occurs not only from around the pit wall but also from the coating surface adjacent to the pit. The next stage in the coating breakdown involved the accumulation of white corrosion product within and around the pits (Figure 15). Precipitation of this corrosion product occurs because the solution in the vicinity of the pits gradually becomes saturated in aluminium hydroxide.

After a few weeks of exposure, pitting was more extensive on the non-glass-beaded IVD surfaces (Figure 16) than on beaded surfaces. With time, a crater of corrosion product forms around the pits and, using a low power stereomicroscope, iron rust is detected within many of the pits (Figure 17). This rust forms as the aluminium loses its effectiveness to act as a sacrificial anode. Precipitation of the aluminium hydroxide polarizes the aluminium coating thus reducing the available galvanic current. This current is then insufficient to provide cathodic protection for the steel substrate. Metallographic sections through pitted specimens showed that aluminium dissolved radially at pores and also along the substrate/aluminium interface where the galvanic driving force is high (Figure 18). On coating surfaces where pitting was not widespread, e.g. those which had underdone two separate periods of glass-bead blasting, only general corrosion of the aluminium was observed as the tests progressed (Figure 19).

After long exposure times, blisters were detected on the specimen surfaces, particularly on those which had been blasted with glass beads (Figure 20). Some blisters contained a brownish liquid, very likely coloured by rust. Some contained only white corrosion product; others a combination of the two. Other blisters contained only clear liquid. The surfaces of the blisters were very thin and not all of them exposed the underlying steel substrate. It appears that, as corrosion proceeds within pores, either along the substrate/coating interface or between IVD layers, the formation of corrosion product and/or the evolution of hydrogen is sufficient to blister the IVD layers. The general dissolution of the outer IVD surface and its further thinning by stretching would also assist the blistering process.

Eventually, with increasing exposure times, red rust stains apparent to the unaided eye developed in the deposits of white corrosion product over pits and within areas of general corrosive attack and blistering (Figure 21). This stage is termed rust bleed-out. The appearance of bleed-out was usually non-uniform on the IVD surface. However, with time, the rust stain became widespread (Figure 22). Despite appearances, some aluminium was still present on the surface at this stage. However, it was very thin, fragmented and only locally adherent.

3.2.1.2 Alternate immersion tests

The process of deterioration of the IVD coating under alternate immersion conditions was generally similar to that described above. However, there were some differences. In particular, white corrosion product accumulated more rapidly on specimen surfaces, especially those which had been blasted with glass-beads. This made it difficult to detect the first signs of rust developing within pits. This accumulation of corrosion product eventually became a hard surface incrustation (Figure 23). On all specimens, coating breakdown first occurred along coated edges probably because the solution remained on the edges longer, when specimens were removed from the test solution, and because the IVD-Al coating was thinner on the edge corners.

3.2.2 Damaged specimens

Sheet specimens damaged by scribing were subjected to both continuous and alternate immersion tests. The coating surfaces away from the scribe marks deteriorated with increasing exposure time as described in the above sections. The coating adjacent to the scribe and the exposed steel behaved as a scaled up version of a pit in the coating penetrating to the steel substrate. Initially, preferential dissolution of the aluminium occurs adjacent to the scribe, as the induced galvanic current protects the steel. With time, white aluminium corrosion product builds up in, and along, the scribe next to the aluminium walls. Subsequently, rust appears in the scribe. Precipitation of white corrosion product did not always occur before the rust was detected. In the few samples tested, it was noticed that aluminium had completely dissolved away from the scribe to a distance of 0.5 to 1 mm (Figure 24). It appears that rusting occurs when the aluminium can no longer provide the cathodic current needed to protect the increased area of exposed steel.

3.2.3 Aircraft Bolts

The corrosion behaviour of the IVD coating on the steel bolts, tested under constant immersion conditions, was quite different from that occurring in the IVD coated sheet steel. Pitting did not occur and breakdown was a result of general corrosion of the IVD layer. This occurred firstly within the threads and along the edges of the countersink head, Figure 25. Corrosion then spread along the remainder of the bolt Figure 26. Finally, with the use of low power microscopy, the grey colour of bare steel could be seen. Rusting of the bare steel quickly followed after the IVD layer on the bolt shank and head also thinned and perforated. General rusting of the bolts occurred rapidly once this stage had been reached (Figure 27).

3.2.4 Tensile specimens

Corrosion, including pitting of the aluminium coating, on the tensile specimens occurred during 200 hours of constant immersion in aerated and dearated sodium chloride solution and in tap water, prior to tensile testing. More pits were present on those specimens exposed to aerated solution. Rust appeared in the pits produced in the aerated solutions but not in those which had developed in deaerated solutions. Metallographic sections revealed that many of these pits were associated with corrosion of the coating immediately adjacent to the steel substrate.

3.2.5 Comparative Assessment of the Breakdown of Various Coatings

An attempt was been made to compare qualitatively the behaviour of the various coating types under both continuous and alternate immersion conditions (Tables 5 and 6). Assessment of the performance of the IVD coatings on sheet specimens was obtained by comparing the times for the first microscopic detection of rust and the first visual evidence of bleed-out. These data are shown in Tables 7 and 8. Tables 9 and 10 list the exposure times at which bleed-out was detected in scribe-damaged sheet specimens and high strength aircraft bolts.

For purposes of comparison, corrosion tests were also conducted with coupons taken from unpassivated porous cadmium plated high strength steel tensile specimens. The bare steel ends were masked before testing. Coating life results are listed in Table 11.

3.3 Tensile Tests

Tests on bare steel specimens in air, at a cross-head displacement rate of 2×10^{-4} mm/s produced a mean fracture stress of 2456 MN/m^2 with a standard deviation of 52 MN/m^2. One IVD-Al coated specimen tested in air gave a fracture stress of 2449 MN/m^2.

Tests on three IVD coated specimens in Ardrox 2106R paint stripper produced a mean fracture stress of 2409 MN/m^2 with little scatter, whereas significant embrittlement and substantial scatter were observed when similar tests were conducted using Turco 5351 paint stripper (Figure 28).

Tests in which the whole of the IVD coating was removed in 2.5 N sodium hydroxide, and the specimens subsequently loaded to failure in air showed little evidence of hydrogen embrittlement (Table 12). However, when the IVD coating was first removed from the notched region only and the remainder dissolved in sodium hydroxide, a measurable level of embrittlement was noted (Table 12, Fig. 2C). In this latter test, a galvanic couple persists during the test period.

The results of testing IVD plated specimens in air after immersion in aerated 0.1N sodium chloride for 200 hours indicated a small degree of embrittlement. Similar tests in de-aerated 0.1N sodium chloride resulted in very severe embrittlement. These results are very consistent, exhibiting a low standard deviation (54 MN/m^2). Specimens tested after 200 hours exposure in de-aerated tap water also resulted in an unacceptable level of embrittlement (Figure 28).

3.4 Adhesion Tests

Low power microscopic examination of as-deposited HD and MD specimens which had been scribed revealed that part of the IVD coating adjacent to the scribe had lifted from the steel substrate (Figure 29). Cracks also developed in the coating adjacent to the scribe marks. No signs of poor adhesion were detected with tests on the glass-beaded coatings.

The as-deposited HD and MD AIV-Al coatings also failed the Scotch Tape tests. Figure 30 and 31 show respectively, (a) part of the coating removed by the tape, and (b) the remaining substrate surface. However, in Fig. 31, the exposed metal was still aluminium, indicating that only part of the IVD coating had been removed. All glass-beaded coatings passed this adhesion test.

4. DISCUSSION

IVD coatings on steel are used mainly for the corrosion protection of steel substrate in aggressive aqueous environments. In such environments aluminium corrodes at a much slower rate than unprotected steel in the same environment. This protection philosophy is satisfactory, as long as the aluminium coating is free from defects and is strongly adherent to the substrate. Once damage or penetration of the coating occurs, the aluminium should still provide galvanic protection for the steel substrate. However, a high level of galvanic protection over a long period can not be expected, except in very mild operating environments. The results of this study support this protection philosophy.

4.1 Quality of Coatings Submitted for Examination

A high quality coating is one which has at least a sound microstructure, a uniform thickness and strongley adheres to an adequately prepared substrate. In this present study, only the barrel IVD coated steel bolts satisfy these requirements. The coatings on the sheet steel substrates were considered to be less than adequate.

However, the as-deposited HD IVD coatings were somewhat better than the as-deposited MD IVD coatings.

The as-deposited HD and MD coatings both contained considerable microporosity. These pores behave as inbuilt active corrosion crevices and contribute to the early failure of the coating, the subsequent corrosion of the steel substrate, and probably leads to poor adhesion. Specifications MIL-C-83488 and MACAIR P.S. 13143 both cover ion vapour deposited aluminium coatings and require coatings to be free from porosity. Glass-beading is used to consolidate the coating and reduce porosity. However, as our investigations have shown, this consolidation fails to remove all porosity. Glass beading is most effective in reducing porosity to a minimum if it is applied after the first IVD pass followed by further glass-beading after the final IVD pass. However, such a procedure would not be commercially attractive

The thicknesses of the Al coating between different HD and MD sheet samples varied considerably. This may be associated with their relative position in the IVD chamber. There was also a significant variation in coating thickness on opposite sides of individual specimens. This could result when the specimens are placed horizontally in the chamber in such a way that one side of the sheet sample is partially in shadow from the molten aluminium passing beneath the samples. A comparison of coatings applied to specimens hung vertically with those applied to the specimens placed horizontally is currently in progress. MIL-C-83488 specifies a minimum thickness for Class 1 coating of 0.001 inches (25.4 μm). All samples met this requirement, except HD 10, HD 2/1, HD 2/3 and MD 5.

The substrate preparations on HD and MD sheet samples and tensile specimens were in our opinion inadequate. Remnants of mill scale, rust and mechanical cleaning contaminants produced local variations in coating thickness and porosity. MIL-C-83488 requires that the base metal be free from such defects. Poor substrate preparation would clearly result in: (a) lower corrosion resistance of the IVD coating, (b) reduced adhesion to the substrate and (c) lower protective qualities. The tests carried out in this study also indicate poor adhesion of the as-deposited HD and MD IVD coatings. These samples would fail the adhesion tests laid down in MIL-C-83488.

4.2 Corrosion Resistance of coatings

The corrosion resistance of the IVD coated sheet samples and steel bolts were assessed using both alternate immersion tests and constant immersion tests. In our opinion these test conditions closely resemble the operating environments likely to be experienced by IVD components in service, where periods of wet and dry and prolonged periods of wetness are encountered.

The protection provided by the coatings to the steel substrate has been assessed by measuring the times to detect rust, both microscopically and visually. Tables 7 and 8 show that under both constant immersion and alternate immersion conditions, the protection afforded to the steel by the IVD coating is increased when

the coating is subjected to glass-beading, particularly if the first and final IVD layers are glass-bead-blasted. These results are consistent with the beading process reducing microporosity and hence, the tendency for the coating to corrode.

Under alternate immersion conditions the first signs of rust take longer to appear compared with constant immersion conditions. The reasons for this are not clear at this stage. However, it was noticed that, during the alternate immersion tests, heavy deposits of white aluminium corrosion product formed on specimen surfaces. These deposits could stifle corrosion in the pores of the coating by excluding oxygen and restricting ionic diffusion.

There is a considerable scatter in the corrosion rate data (Tables 7 and 8). This is partly due to the subjective nature of the test, but is more likely the result of specimen to specimen variations in: (a) the degree of microporosity, and (b) coating thickness. In Figure 32, the times for visual detection of rust are plotted against coating thickness. The data can be divided into two groups. One group consisting of data from specimens which had been glass-bead-blasted, and another group consisting of data from specimens which had not been bead blasted. All the data show a clear correlation of increasing corrosion resistance of the coating with increasing coating thickness. It is also clear (Fig. 32) that corrosion resistance is increased if porosity is reduced by glass-bead blasting. At low coating thicknesses, porosity appears to have a reduced influence on corrosion resistance, although the data in this region are incomplete. Figure 32 shows that glass-beading, at some stage in the coating process, significantly improves the corrosion protection properties of the coating at thicknesses greater than 20-25 μm. The time to rust data obtained with the barrel-coated bolts are also plotted in Fig. 32. These results are of interest because of the low average coating thickness in the thread where rusting first occurs. The improved corrosion resistance of this coating compared with the coating on the sheet samples of comparable thickness is due to the almost complete absence of microporosity, and the fine grain equiaxed structure of the barrel coating.

If a defect-free or non-porous coating can be produced, the minimum coating thickness required for protection will be reduced, and such an aluminium coating would corrode very slowly in most aqueous aircraft environments. To consistently produce a defect-free or totally non-porous coating is probably very difficult. Hence, the best coating for corrosion resistance will result from a processing compromise (influenced by economics) between thickness, acceptable or manageable porosity, and appropriate use of glass-bead-blasting.

Specification MIL-C-83488 requires that Class I, Type I coatings (as applied to sheet samples) subjected to the salt spray test shall show no evidence of corrosion of the substrate metal in 21 days of exposure. Unfortunately, this specification does not indicate by what technique that evidence should be sought. Clearly, this area needs clarification. Alternate immersion corrosion tests (rather than salt spray tests) were used in the present study. Although the salt spray test is probably a more severe test, it is interesting to compare the performance of the coating against the specification requirements. If visual detection of rust (i.e. bleed out) is used as the criteria for failure, most of the sheet samples would probably meet specification requirements. However, HD 10 (4I + B) under constant immersion and

sample MD 5 under alternate immersion would most likely fail. It is significant that these samples do not meet the specified coating thickness. If microscopic detection of rust is used to indicate failure, then all of the as-deposited coatings tested under constant immersion conditions would fail, glass-bead-blasted coatings could be considered borderline, and the coatings subjected to two bead blasting passes would satisfy the specification. Under alternate immersion conditions, the corresponding data are incomplete. However, they do show that coatings which have had two bead-blasting passes are the most resistant to corrosion.

On an aircraft component in service, the IVD coating would normally be chromated and painted, thus the lifetime of the coating would be extended. However, it is highly likely that during service the paint and chromate film would be damaged and the coating exposed. In such a situation, the appearance of rust through the coating, even on a microscopic scale, suggests that the steel substrate is at risk. Corrosion of the steel (i.e. rust) could, if pitting conditions develop, lead to sites capable of initiating fatigue in highly stressed components. Furthermore, as the tensile tests indicate, perforation of the aluminium, without the formation of rust, even in what may be considered a rather benign environment, viz. deaerated tap water, may be associated with hydrogen embrittlement of the steel substrate. To minimise the risk of early fatigue crack initiation and/or hydrogen embrittlement, a high quality coating is required, i.e. one without significant porosity (e.g. barrel coatings) or one where the porosity is reduced to a minimum.

It is interesting to compare the corrosion results obtained, with results from cadmium-coated specimens (c.f.. Tables 7 and 8 with Table 11). These data indicate that only IVD coatings containing more than one glass-bead-blasted layer match the performance of the cadmium coatings tested under identical conditions.

Tables 9 and 10 provide information on the effect a damaged coating has on the corrosion of the steel substrate. Data from constant immersion corrosion tests with scribed samples indicate that, while differences exist between one sample and another, rust was visible on the bare steel substrate after 45 to 87 days of exposure. With alternate immersion tests these times were even less. These differences are to be expected since, under such conditions, galvanic coupling will not always be complete or effective. However, the results do indicate that a damaged IVD coating does offer significant sacrificial protection to steel substrate. Uncoated 4130 steel rusts within hours of exposure to NaCl solutions. These results imply that, if a painted IVD-coated component should sustain damage, and thus expose the underlying steel, corrosive attack of the steel would still be delayed.

Damage produced on coated aircraft bolts by repeated torquing does not appear to have affected the corrosion resistance of the coating. The application and removal of the nuts did not completely remove the coating on the bolt thread, but would certainly have reduced the coating thickness. The fact that similar corrosion rates were involved for damaged and as-received bolts, reflects the high quality of the barrel coatings.

4.3 Hydrogen Embrittlement Tests

Slow strain rate testing was used in the present work to assess the degree of hydrogen embrittlement induced in IVD-coated, notched tensile, 4340 steel specimens during exposure to various environments. It has been demonstrated previously (Ref. 3) that a minimum mean fracture stress of 1700-1850 MN/m^2, exhibited in three low embrittlement cadmium plated and baked tensile specimens when tested in paint strippers, at a cross-head displacement rate of 2×10^{-4} mm/s, corresponds to the pass/fail condition in standard notched C-ring tests. This result was used as a guide for determining the conditions in which IVD-coated steel components could be safely used.

The present results confirm that the IVD process itself does not cause hydrogen embrittlement of the steel. Tests conducted with Ardrox 2106 R paint stripper, also showed that this product does not embrittle the steel. In contrast, multiple tests with Turco 5351 paint stripper produced a low mean stress and high standard deviation. This product would come close to causing failure in a standard C-ring test. Slow strain rate tests of notched tensile specimens tested in air, after removal of the IVD coating in 2.5 N sodium hydroxide, indicate a small amount of hydrogen embrittlement. This could be removed by baking at 190°C for 23h. A similar degree of embrittlement was also experienced, when IVD-plated 4340 steel specimens were exposed to aerated 0.1 M sodium chloride for 200h. However, much greater embrittlement occurred when IVD-coated specimens were exposed, either to de-aerated 0.1 M sodium chloride or to de-aerated tap water. The low fracture stress values indicate that exposure to these environments for only 8 days causes sufficient embrittlement to produce failure in standard C-ring tests. These conditions could readily arise during service, particularly in situations where condensation occurs in occluded areas of structure and ingress of air is limited (e.g. crevices, fastener holes, etc). IVD-Al coating, in its present form, should not be used on ultra-high strength steel components in situations where the above condition can develop during service.

CONCLUSIONS

1. As-deposited IVD coatings exhibit extensive microporosity. Glass-bead blasting after the first IVD pass and after the final IVD pass, significantly reduced the porosity. Little if any porosity could be detected in coatings applied to steel bolts using the barrel process.

2. The thickness of IVD coatings from sample to sample was not uniform.

3. The coating thickness on any one sheet differed from one side to the other.

4. The as-deposited coatings were found not to adhere strongly to the substrate. Glass-bead-blasting appeared to improve the adhesion.

5. The substrate preparation for all sheet and tensile specimens was inadequate. Residual non-metallic surface impurities contributed to microporosity, localised thickness variations and poor adhesion.

6. Corrosion tests showed that IVD coatings can be very resistant to corrosion in aqueous chloride environments and, thus, protect steel substrates. That is provided the coating is sufficiently thick and its porosity is low, which may be achieved either by glass-bead blasting or by deposition under barrel-coating procedures. Coatings of this quality were found to have corrosion resistance and protection qualities for steel similar to electrodeposited cadmium.

7. Corrosion tests with damaged coatings showed that the IVD-Al coating is capable of galvanically protecting the exposed steel substrate.

8. The IVD process itself does not cause hydrogen embrittlement of the high strength steel subtrates.

9. The use of paint strippers with IVD-Al coating and the use of sodium hydroxide solution to strip the IVD coating can, under certain conditions, lead to hydrogen embrittlement of the high strength steel substrate.

10. Exposure of IVD-Al coated steel to aqueous environments can, under certain environmental conditions, produce embrittlement of high strength steel substrate if the IVD coating itself is totally unprotected, e.g. by either chromating and/or painting.

REFERENCES

1. E.R. Fannin and D.E. Muehlberger, MCAIR Report 78-006, 1978.

2. E.R. Fannin, MCAIR Report 78-007, 1978.

3. W.J. Pollock and C. Grey, ARL Aircraft Materials Report 118, December 1985.

4. RAAF Letter, "Corrosion Protection for High Strength Steel Alloys", AIR 4/0135-01 (50).

TABLE 1

SIZE OF SURFACE DEPRESSIONS - μm

SAMPLE	COATING	DIAMETER	DEPTH
HD 9	4I	58	36
MD 3	4I	77	35
HD 11	I + B + 3I	35	19
HD 12	I + B +3I + B	62	27
HD2/6	4I + B + C	84	42

TABLE 2

SHEET SAMPLE COATING - THICKNESS μm

SAMPLE	COATING	SIDE A	SIDE B
HD 1	4I	42	38
HD 2	4I	38	38
HD 3	4I	44	34
HD 4	4I	33	34
HD 5	4I	30	34
HD 6	4I	45	34
HD 7	4I	39	38
HD 8	4I	38	42
HD 9	4I	44	40
HD 10	4I + B	15	14
HD 11	I + B +3I	36	40
HD 12	I + B + 3I + B	23	33
HD2/1	4I	24	19
HD2/3	4I + B	18	22
HD2/5	4I + B + C	17	20
HD2/7	4I + B + C	18	20
MD 1	4I	29	47
MD 3	4I	23	36
MD 5	4I	17	21

TABLE 3

BOLT COATING THICKNESS

POSITION	THICKNESS μm
Shank	11 - 20
Thread Root	10 - 14
Thread Surface	9 - 17
Thread Crest	9 - 17

TABLE 4

TENSILE SPECIMEN COATING THICKNESS

	THICKNESS μm	
POSITION	RANGE	AVERAGE
Gauge Length	46 - 60	55.2
Notch Flank	24 - 36	28.0

TABLE 5

DEVELOPMENT OF CORROSION ON SHEET SAMPLES - CONSTANT IMMERSION IN NaCl

SAMPLE	TIME INTERVAL				
	After 1 Day	1 Week	1 Month	2 Months	4 Months
4I HD 5 HD 5 HD 4 HD 2/1	Much white corrosion product (WCP) in test cell	Many pits, rust in some pits	Widespread pitting, rust in many pits	Widespread pitting, rust in most pits HD2/1-Bleed Out	Bleed Out 50%
41 MD 1 MD 3	Much white corrosion product in test cell	Many pits, rust in some pits	Widespread pitting, rust in many pits	Widespread pitting, rust in most pits MD 3 - Bleed Out	Bleed Out 50%
4I + B HD 10 HD 2/3	A little white corrosion product in test cell	Many pits, some contain rust Few pits	Bleed Out Several pits	Bleed Out 80% Rust in a few pits	Bleed Out 20%
I+B+3I HD 11 HD 11	A little white corrosion product in test cell	Few pits	Few pits, some contain rust	Few pits some contain rust	Bleed Out 25% No bleed out
I+B+3I+B HD 12 HD 12	No white corrosion product in test cell	Few pits, a little white corrosion product in test cell	Few pits, a little WCP, Few blisters	Few pits, a little WCP, Few blisters	Bleed Out 5% No bleed Out

TABLE 6

DEVELOPMENT OF CORROSION ON SHEET SAMPLES - ALTERNATE IMMERSION IN NaCl

SAMPLE	TIME INTERVAL				
	After 1 Day	1 Week	1 Month	2 Months	4 Months
4I HD 8 HD 8 HD 7 HD 2/1	White corrosion product on surface and in test cell	Many pits, rust in some pits Few pits	Thick surface coating of white corrosion product, wide-spread pitting many contain rust	White corrosion product, pits rust HD2/1-Bleed Out	Bleed Out 50%
41 MD 5 MD 5 MD 5	White corrosion product on surface and in test cell	Many pits, rust in some pits	Bleed Out 100%		
4I + B HD 10 HD 2/3	Few pits	Many pits, a little white corrosion product	Bleed Out	Bleed Out 20%	
I+B+3I HD 11	Clear, No white corrosion product	Clear	A little white corrosion product on surface, few pits	Surface coated with white corrosion product, pits, a few contain rust	Bleed Out 1%
I+B+3I+B HD 12	Clear	Clear	Few pits, blisters	Few pits, blisters	Few pits,

TABLE 7

SHEET SAMPLE COATING LIFE - CONSTANT IMMERSION IN NaCl

SAMPLE	COATING TYPE	TIME TO RUST (DAYS)	
		MICROSCOPIC DETECTION	VISUAL DETECTION
HD 5	4I	1 - 5	74
HD 5	4I	1 - 5	84
HD 4	4I	3	101
HD 2/1	4I	6	34
MD 1	4I	1 - 3	84
MD 1	4I	1 - 3	84
MD 3	4I	1 - 3	40
HD 10	4I + B	1 - 5	29
HD 10	4I + B	9	17
HD 2/3	4I + B	47	77
HD 11	I + B + 3I	-	86
HD 11	I + B + 3I	27	152
HD 12	I + B + 3I + B	87	109
HD 12	I + B + 3I + B	105	159

TABLE 8

SHEET SAMPLE COATING LIFE - ALTERNATE IMMERSION IN NaCl

SAMPLE	COATING TYPE	TIME TO RUST (DAYS)	
		MICROSCOPIC DETECTION	VISUAL DETECTION
HD 8	4I	-	53
HD 8	4I	-	66
HD 7	4I	15	31
HD 2/1	4I	34	47
MD 5	4I	-	15
MD 5	4I	-	15
MD 5	4I	12	17
HD 10	4I + B	-	33
HD 2/3	4I + B	26	29
HD 11	I + B + 3I	45	86
HD 12	I + B + 3I + B	287	302

TABLE 9

DAMAGED SHEET SAMPLE COATING LIFE - TIME TO RUST (DAYS) - VISUAL DETECTION

SAMPLE	COATING TYPE	CONTINUOUS IMMERSION - NaCl	ALTERNATE IMMERSION NaCl
HD 2	4I	47	54
HD 2	4I	87	54
MD 3	4I	45	37
MD 3	4I	84	37
HD 11	I + B + 3I	59	
HD 12	I + B + 3I + B	66	

TABLE 10

AIRCRAFT BOLT COATING LIFE - CONSTANT IMMERSION IN NaCl

CONDITION	TIME TO RUST (VISUAL DETECTION) - DAYS
AS RECEIVED	83, 94
DAMAGED	107, 95

TABLE 11

CADMIUM COATING LIFE

SOURCE	TIME TO RUST	MICROSCOPIC DETECTION
	CONSTANT IMMERSION	ALTERNATE IMMERSION
Hawker de Havilland	91	192
	123	
Qantas	56	
	91	

TABLE 12

HYDROGEN EMBRITTLEMENT TEST: *Al IVD 4340 STEEL*

ENVIRONMENT	FRACTURE STRESS (MN/m^2)	MEAN (MN/m^2)	STANDARD DEVIATION (MN/m^2)
Al-IVD Coated: Test in Air	2449		
Test in Turco 5351 Paint Stripper	1682, 2077, 1446	1735	318
Test in Ardrox 2106R Paint Stripper	2397, 2480, 2349	2409	66
Dissolve Al in 2.5N NaOH, Test in Air	2449, 2545, 2530	2508	52
Dissolve Al from Notch Only in 2.5N NaOH, Test in Air	2256, 2434, 2485	2392	120
Dissolve Al from Notch Only, then dissolve remaining Al in 2.5N NaOH, Test in Air	2408, 2229	2319	127
Immerse in Aerated 0.1N NaCl for 200 h, Test in Air	2339, 2308, 2339	2329	18
Immerse in De-Aerated 0.1N NaCl for 200 h, Test in Air	1101, 995, 1069	1055	54
Immerse in De-Aerated Tap Water for 200 h, Test in Air	1368, 1703	1536	237

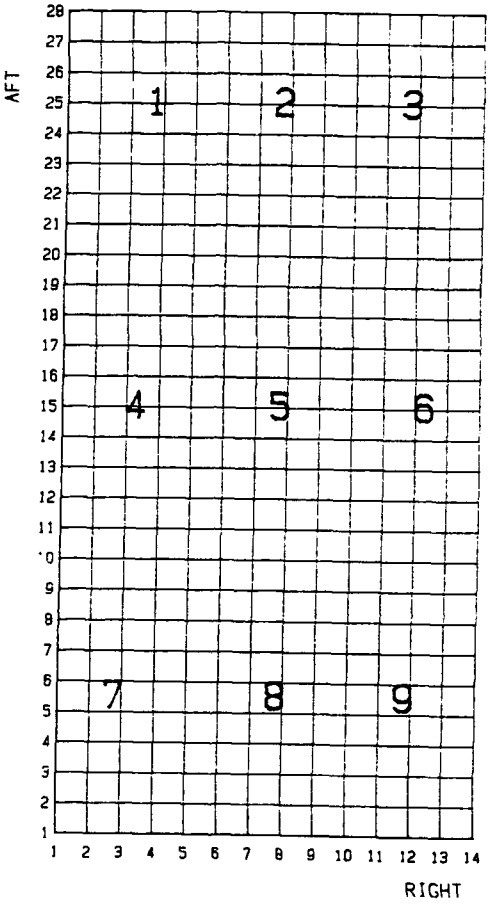

Figure 1: Substrate/Coupon Location Record

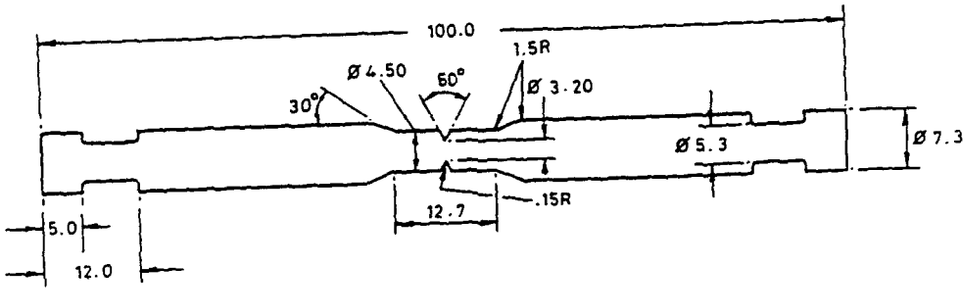

Figure 2: Notched Tensile Specimen

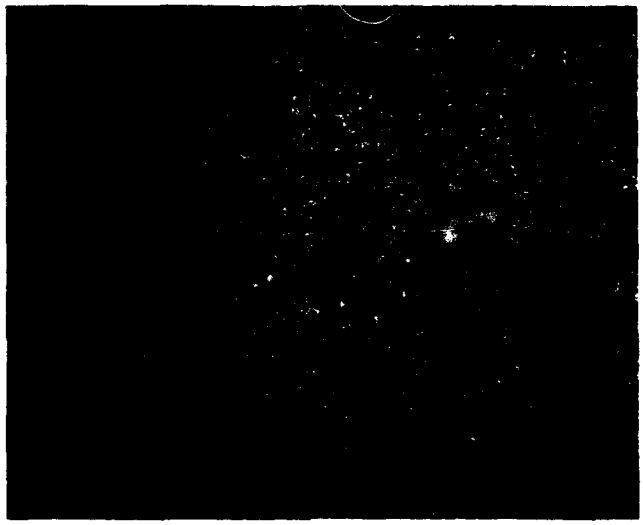

Figure 3: Optical micrograph of an as-deposited IVD HD sheet sample. Note the wide distribution of depressions (A) in the surface.
MAG: X128

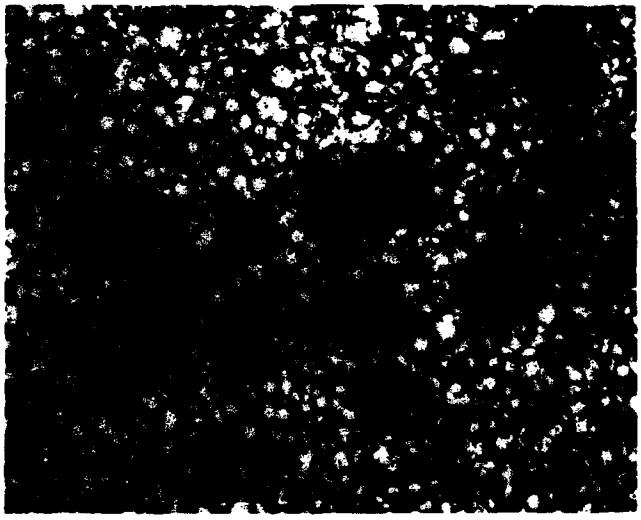

Figure 4: Optical micrograph of a glass-bead-blasted IVD HD sheet sample with depressions at A still retained. MAG: X128

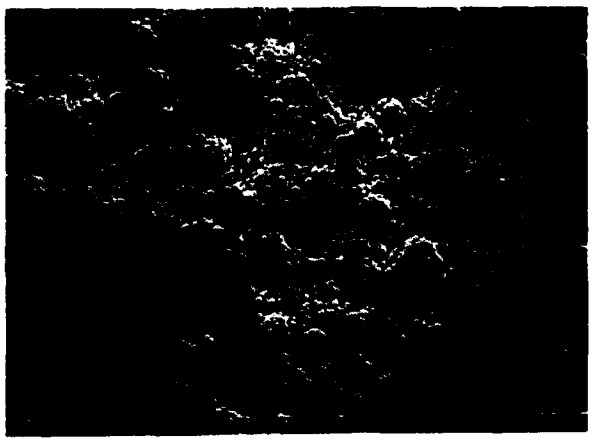

Figure 5: Scanning electron micrograph of an as-deposited IVD HD sheet sample. Note microporosity at A.

Figure 6: Higher magnification micrograph of surface in Figure 5.

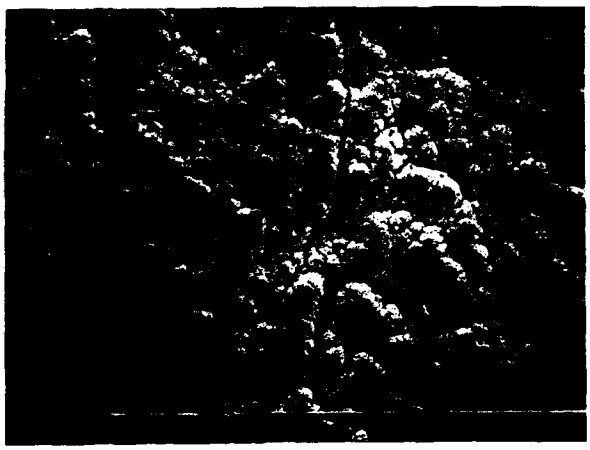

Figure 7: Scanning electron micrograph of an as-deposited IVD MD sheet sample. Note microporosity at A.

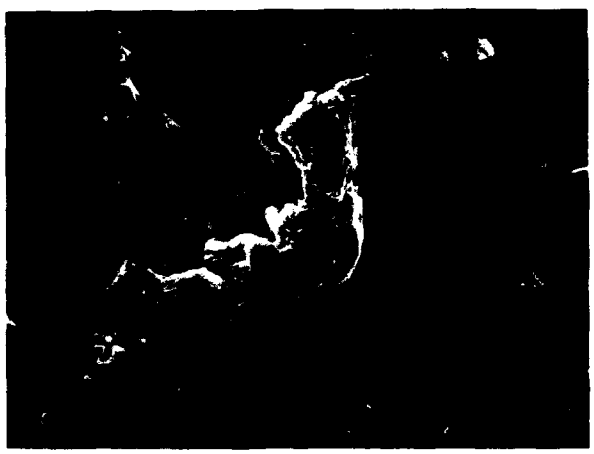

Figure 8: Scanning electron micrograph of a glass-bead-blasted IVD HD sheet sample showing areas of deformed surface (A) and unconolidated microporosity B.

Figure 9 (a): Metallographic section through the IVD coating on an as-deposited HD sheet sample. Note the columnar grain structure (A) and the coarse microporosity (B). MAG: X400

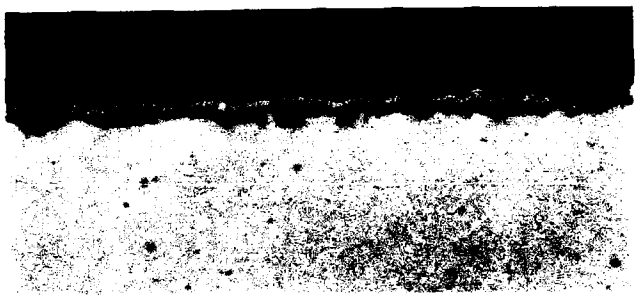

Figure 9 (b): Metallographic section through the IVD coating on an as-deposited MD sheet sample. MAG: X200

Figure 10 (a): Metallographic section through the glass-bead-blasted IVD coating on a HD sheet sample. Note the area A which had been unaffected by the glass-bead blasting adjacent to a bead-blasted area B. MAG: X400

Figure 10 (b): Metallographic section through an IVD coating (I + B + 3I) on a HD sheet sample. Note the porosity free layer adjacent to the steel substrate. MAG: X320

Figure 10 (c): Metallographic section through an IVD coating I + B + 3I + B on a HD sheet sample. Note the almost complete absence of microporosity. MAG: X320

Figure 11: Metallographic section through an as-deposited IVD coating on a MD sheet sample. Note the contaminated steel substrate (A). MAG: X500

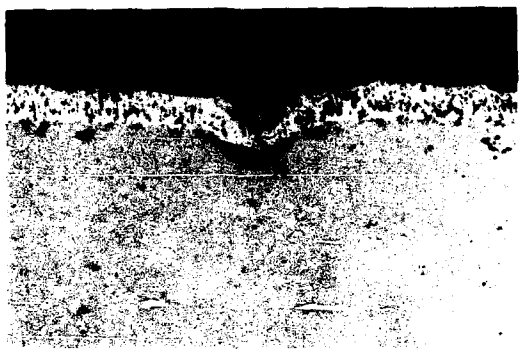

Figure 12: Metallographic section through a surface depression on a glass-bead-blasted HD sheet sample. Note the surface contamination immediately beneath the depression (A). MAG: X400

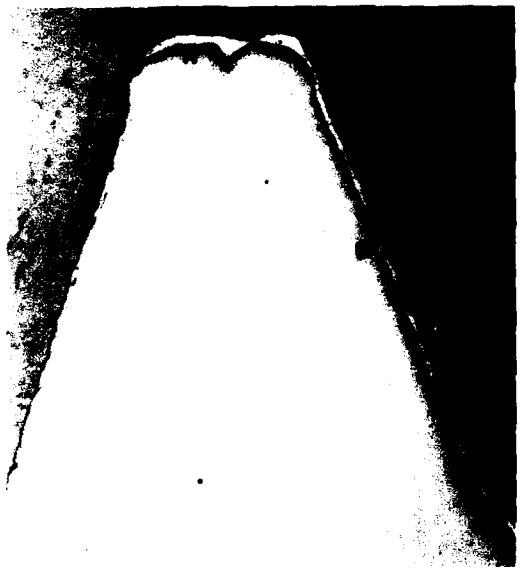

Figure 13: Metallographic section through a single thread of an aircraft bolt showing the variation in IVD coating thickness between the thread crest (A), surface (B) and root (C). MAG: X128

Figure 14: Optical micrograph of a pit formed on the coating surface. It is surrounded by a halo shown as a dark ring in this photo.
MAG: X64

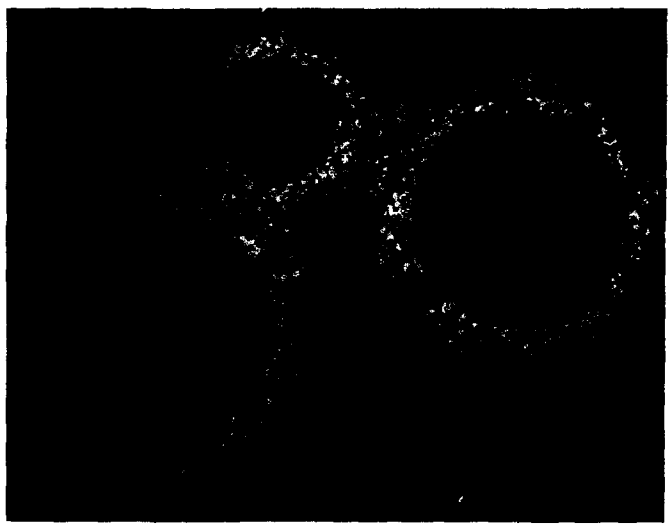

Figure 15: Optical micrograph of pits surrounded by deposits of white corrosion.
MAG: X128

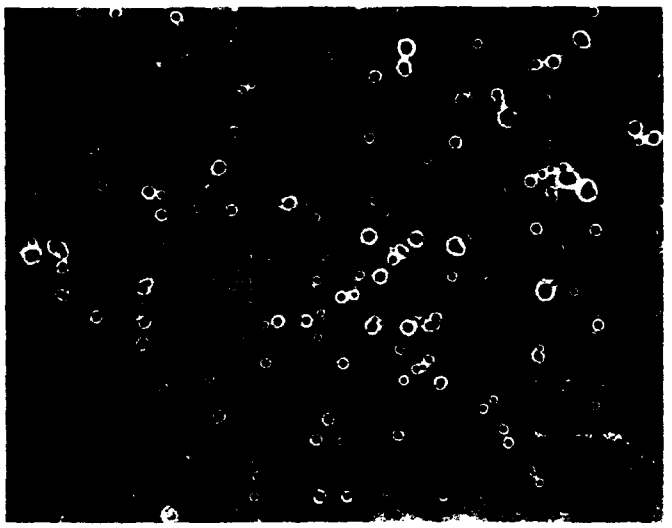

Figure 16: Optical micrograph showing extensive pitting on the surface of an as-deposited IVD-coated HD sheet sample. MAG: X4

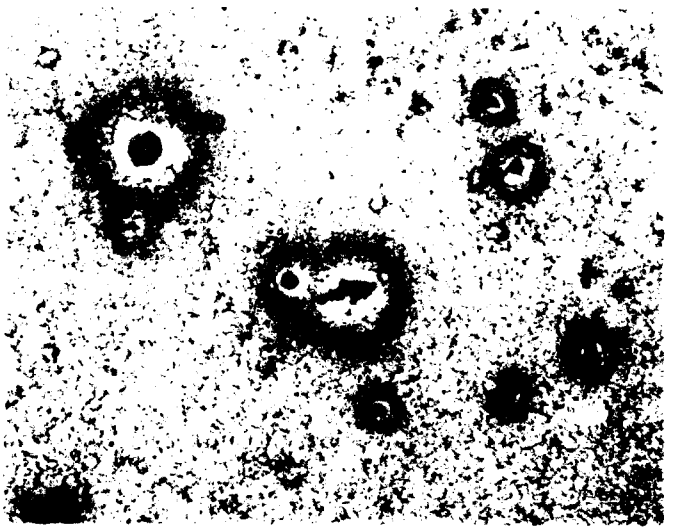

Figure 17: Optical micrograph showing rusting of the steel at the base of pits. MAG: X10

Figure 18: Metallographic section through a pitted coating. Note the corrosion of aluminium away from the pit along the aluminium-steel substrate. MAG: X500

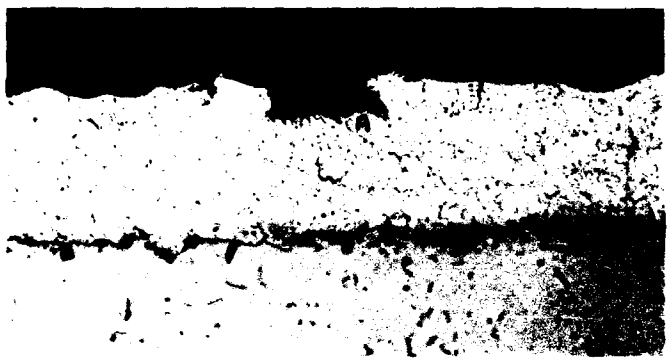

Figure 19: Metallographic section through general corrosion (A) on the IVD coating. MAG: X500

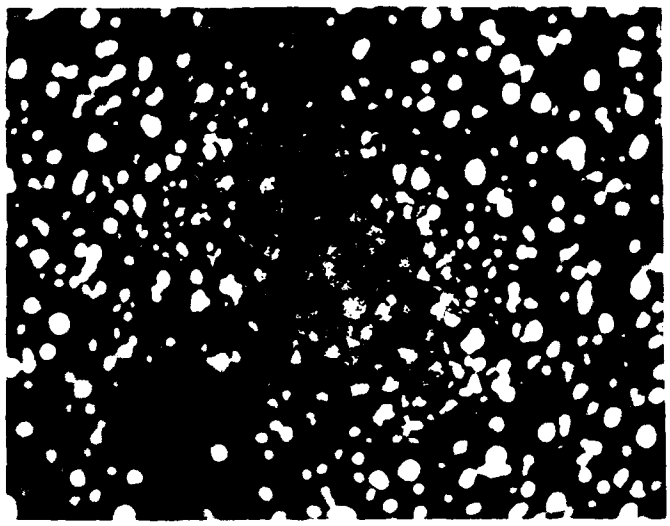

Figure 20: Optical micrograph showing a blister (in focus) on a beaded IVD surface (out of focus). MAG: X160

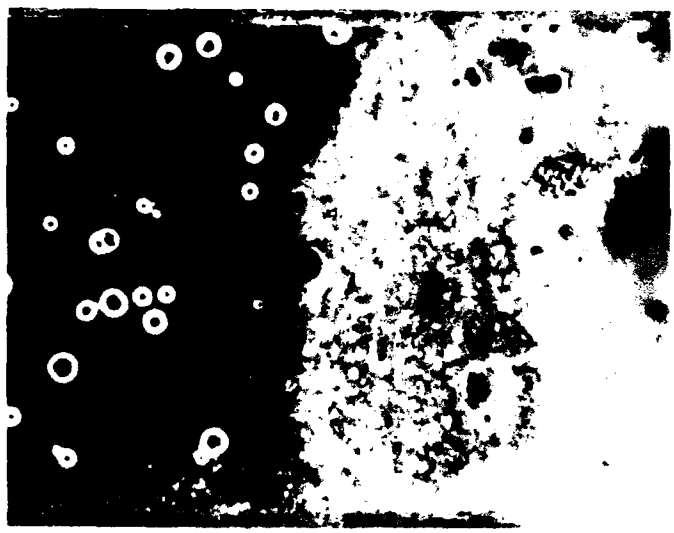

Figure 21: An as-deposited IVD surface showing "bleed out" of rust stain. MAG: x8.5

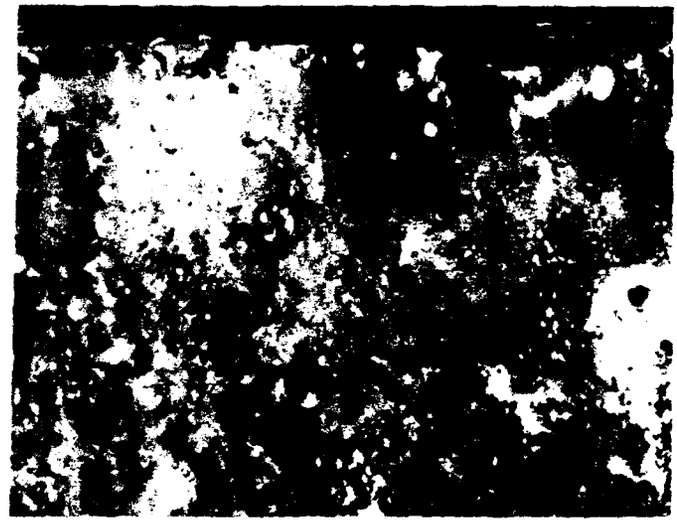

Figure 22: The advanced stages of the breakdown of an IVD coating.
MAG: X8.5

Figure 23: Optical micrograph of an IVD surface, after testing under alternate immersion, showing an almost complete covering of white corrosion product. MAG: X3

Figure 24: Corrosion associated with a scratched IVD coating. Note the extent of dissolution (D) of aluminium away from the scratch (S).
MAG: X8.5

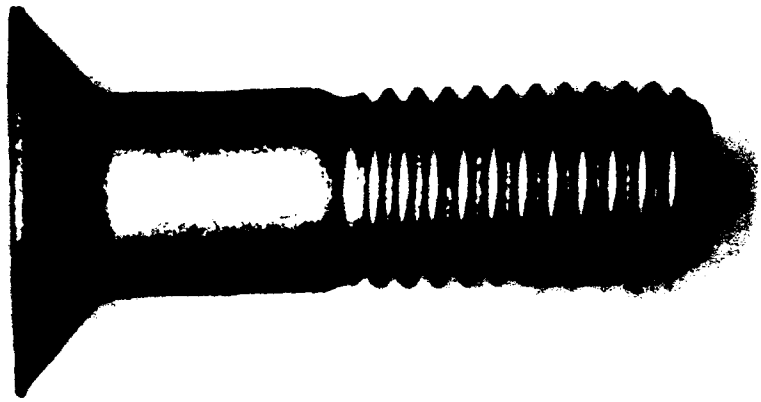

Figure 25: Aircraft bolt showing corrosion of IVD-Al coating in threads (A).
MAG: X7

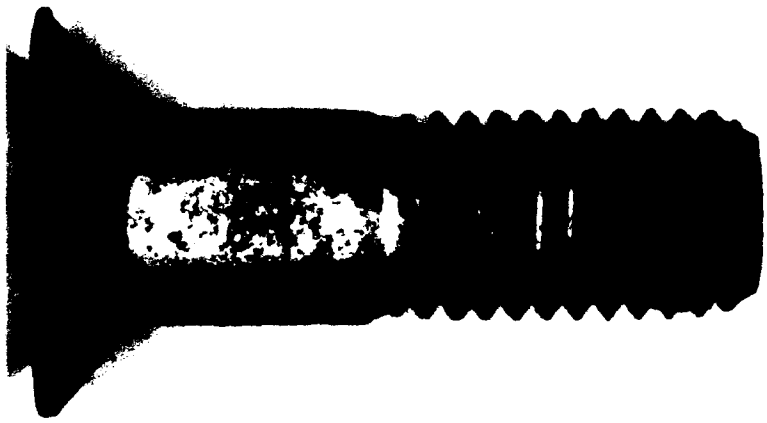

Figure 26: Advanced corrosion of the IVD-Al coating. MAG: X7

Figure 27: Rust "bleed out" through the showing IVD-Al coating and the almost complete breakdown of coating. MAG: X7

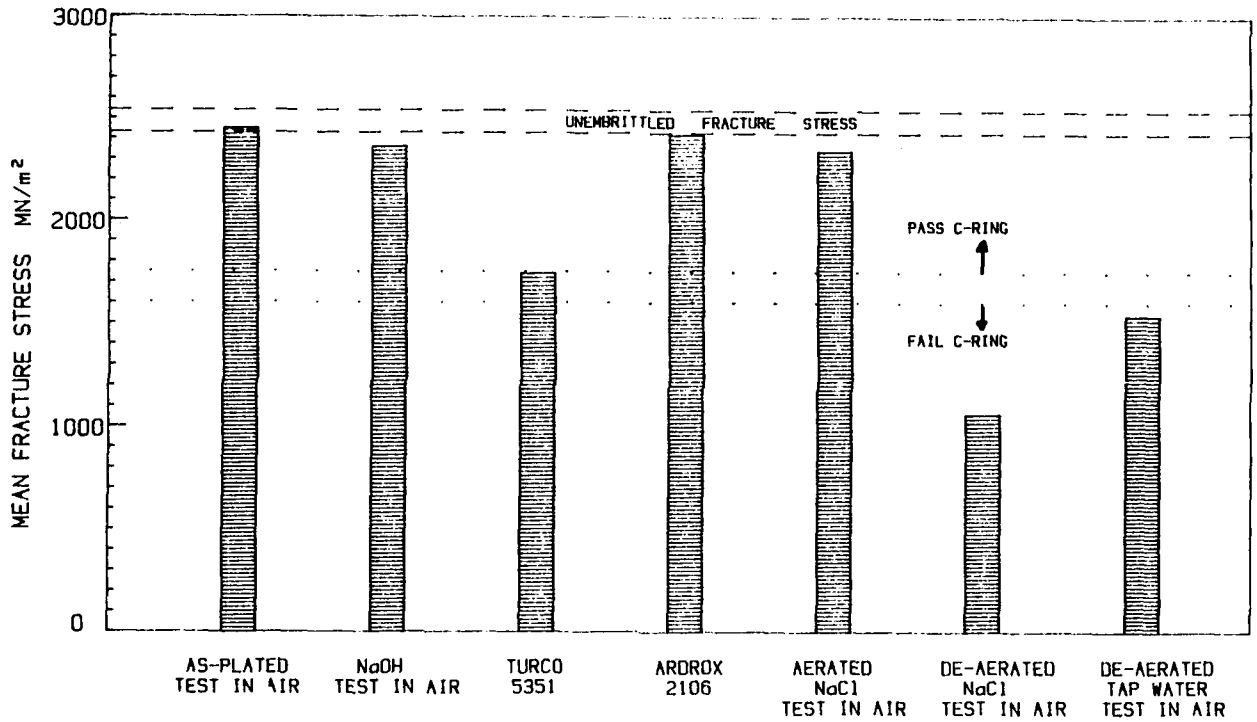

Figure 28: Mean fracture stress of AI-IVD plated 4340 steel notched tensile specimens subjected to various environments and tested at a crosshead displacement rate of $2*10^{-4}$mm/s

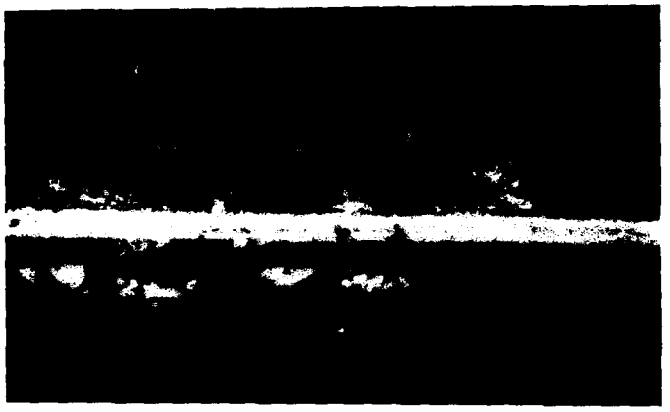

Figure 29: Optical micrograph showing cracking of the IVD coating (A) adjacent to the scribe mark on an as-deposited MD sheet sample. MAG: X64

Figure 30: Fragment of IVD coating removed with 250# Scotch Tape from an as-deposited HD sheet sample. MAG: X64

Figure 31: Surface of the sheet sample from which the fragment of IVD-Al coating shown in Figure 30 was removed. MAG: X64

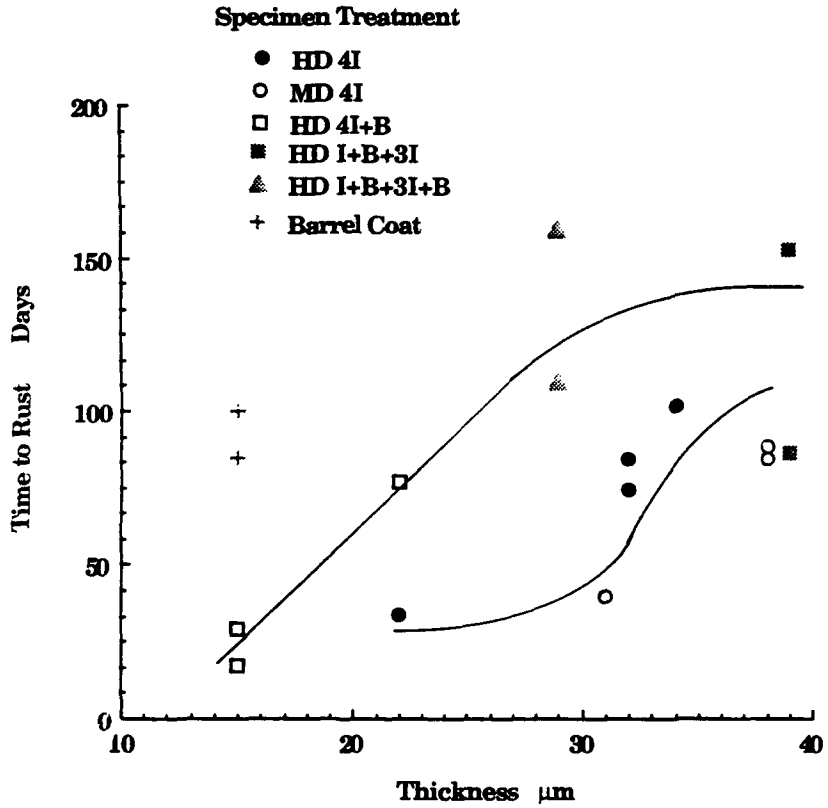

Figure 32: Time to rust, as determined by visual inspection, as a function of coating thickness, for specimens tested under constant immersion conditions.

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Extensive metallographic, corrosion and hydrogen embrittlement tests have been carried out with high strength steel sheet coated with ion vapour deposited (IVD) aluminium. Metallography showed that the thickness of the as-deposited IVD coatings was not uniform and that the coatings contained extensive microporsity. The application of glass bead blasting after coating, significantly reduced the porosity but did not remove it completely. The IVD coatings were found to be effective in preventing corrosion of the steel substrate provided the coating was sufficiently thick and its porosity low. Exposure of IVD coated steel to aqueous environments was found to produce hydrogen embrittlement of the high strength steel substrate under certain conditions.

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