Corrosion protection of smooth surfaces – coating adhesion

Catalina H.Musinoi Hagen, Alexander Kristoffersen, Norwegian University of Science and Technology, Trondheim/Norway; Ole Øystein Knudsen, SINTEF, Trondheim/Norway

Summary

Coatings on machined surfaces are generally found to degrade early, and this has been attributed to poor adhesion. Good adhesion normally requires blasting, but not all surfaces can be blast cleaned due to other functional requirements, such as seal (flange surface) or assembly (tightness of nuts). Our findings show that machined surfaces had poor resistance to cathodic disbonding and corrosion creep. Impact toughness was found on the average to be four times lower on machined surfaces than on blasted surfaces. Dry adhesion tests gave no discernable difference between machined and blasted surfaces. Wet adhesion testing, however, indicated that adhesion was strongly weakened on machined surfaces by permeation of water into the coating.

1 Introduction

In the Norwegian waters, protective coating systems for offshore installations and associated facilities are selected according to the standard NORSOK M-501 [1]. ISO 12944 is used in other industries [2]. Both standards relate the performance of coatings applied on steel to the state of the surface immediately prior to coating, and in most cases recommend blasting of the surface to increase coating adhesion. The purpose with the blasting is removal of unwanted surface layers and contaminants and the increase of surface roughness to increase adhesion. The blast cleaning method is however not suited for all surfaces. Surfaces that are machined for functional requirements, such as seal (flange surface) or assembly (tightness of nuts), cannot be blast cleaned before being coated. Smooth machined surfaces are therefore found on many components on ships and offshore installations, and are generally found to degrade early with subsequent corrosion of the steel substrate. The loss of adhesion to the steel surface is regarded as the main failure mechanism of protective coatings [3] as it results in the exposure of the steel to the corrosive environment. Surfaces on ships and offshore installations are often exposed to mechanical wear and damages, such as physical impacts from other objects. Corrosion is found to initiate at damages in the coating. A coating must hence not only possess adhesion to the substrate but also impact toughness in order to protect effectively against localized corrosion [4]. Typical corrosion processes are found to be blistering, underfilm corrosion creep and cathodic disbonding [5].

It is believed that in order to achieve a good adhesion the surface roughness must be increased to a sufficient level [3, 6-9], although evidence is also found for the opposite [10-12]. The increase of adhesion strength with surface roughness has been linked to an increase in surface area and specific surface energy [11].

It has been stated that corrosion is not possible as long as adhesion persists [13]. The corrosion induced coating degradation is initiated by processes that lead to the formation of a water phase on the surface of the metal, e.g. mechanical damage, osmotic blistering (salts present on the surface during coating), low film thickness or cracking of the coating. Corrosion may then propagate under the coating by three recognized mechanisms [4, 14, 15]

1) Cathodic disbonding (cathodic front alkalizes the water phase and disbonds the coating)
2) Anodic undermining (bonding sites on the substrate disappear due to corrosion)
3) Tearing of the coating by corrosion products with increased volume

This spreading of corrosion is commonly known by the names "corrosion creep", "underfilm corrosion" or "scribe creep". It is seldom seen to be initiated on undamaged surfaces.

Previous studies have reported about the effect of surface roughness on the initiation of a corrosion on painted steel [3, 7-9, 14, 16-23]. Although there is some agreement about the importance of an increased interfacial area between the coating and steel in order to diminish the rate of delamination, the exact mechanism for the adhesion loss or what parameters that may determine the delamination rate are not agreed upon. The roughness necessary to provide sufficient disbonding resistance in corrosive environments is also disputed.

The recent work of Sørensen *et al.*[8] suggests that the commonly used surface roughness parameters Ra, Ry or Rz do not describe the surface topography precisely. Substrates blasted with finer grit showed lower rates of cathodic disbonding than substrates blasted with larger grit particles, even for substrates with comparable profile height and peak densities. The experimental study indicated that the tortuosity of the surface is a parameter of greater importance compared to the traditional surface roughness parameters, as it describes the actual interfacial area more precisely than the profile height and peak density. Tortuosity is defined as the actual length of the steel-coating interface along a cross section, divided by the length of a straight line between the two end points.

However, wet adhesion is key to understanding corrosion resistance of coatings, as few coatings are able to resist hydrolysis over time [4]. The presence of water in the coating affects adhesion negatively, weakening it to a partially reversible value known as wet adhesion. Adhesion in wet conditions is hence lower than in dry, but it is stated that due to residual adhesive forces not all adhesion is lost. Wet adhesion is not seen as a failure mechanism but as a coating property [24].

This paper presents results for corrosion resistance and adhesion of organic coatings on machined surfaces with varying surface roughness. Four different machining processes have been evaluated with respect to surface topography and tested with respect to coating adhesion (ISO 4624, ISO 16276-2), impact toughness (ISO 6272-1) and corrosion creep resistance (ISO 20340). Blast cleaned and zinc metallized (TSZ) samples were used as reference surfaces in all tests.

The preliminary conclusion from the study is that the poor coating performance on machined surfaces is due to low wet adhesion strength, which caused little resistance against corrosion creep. Cathodic disbonding seems to be the mechanism by which adhesion loss spreads over the surface. Corrosion then follows behind.

The long term objective with this study is to improve coating lifetime in terms of a durable corrosion protection on machined surfaces by finding the right combination of coating and machining process. The objective with the work reported here was to investigate the properties of coatings applied on machined surfaces and to propose one or more hypotheses to why they degrade so rapidly.

2 Experimental

2.1 Materials

Samples representing four different machining processes – end milled, rolled milled, angle grinded and turned – have been tested for dry and wet adhesion, creep corrosion and impact toughness properties. Blast cleaned and zinc metallized (TSZ) samples were used as reference surfaces in all tests.
Information about the samples is given in Table 1. Test panels, 75x150 mm large and 4 - 5 mm thick, were prepared from various species of steel and cast iron. The samples were delivered by two different suppliers: supplier A delivered samples which had been end milled, rolled milled or turned, see systems A and B in Table 1. They applied an epoxy based antifouling coating system used for submerged parts on ships. Samples from supplier B had been angle grinded or end milled to a defined surface roughness R_a=1,6 µm and R_a =12 µm, see system C and D in Table 1. They applied a coating system used for equipment on boat decks.

Table 1: Materials, surface preparation and coatings.

	Material	Machining	Coating system	
			Generic type	**Thickness [µm]**
A1	Mild steel S355 EN10025	End milled	Epoxy primer Epoxy Epoxy vinyl tie-coat Antifouling coat	50 150 150 150
A2		Rolled milled		
A3		Turned		
A4		Blast cleaned		
B1	Cast iron SG400-12	End milled		
B2		Rolled milled		
B3		Turned		
B4				
		Blast cleaned		
C1	S355 J2G3	End milled R_a=1.6 µm	Epoxy primer Polysiloxane Polysiloxane	40 125 75
C2		End milled R_a=12µm		
C3		Angle grinded		
C4		Zinc metallized		
D1	S690QL	End milled R_a=1.6 µm		
D2		End milled R_a=12µm		
D3		Angle grinded		
D4		Zinc metallized		

2.2 Adhesion

It is difficult to investigate adhesion phenomena with the test methods available today. Several parameters have been found to influence the reliability of results [25, 26], as skills of the test operator, the coating thickness, curing time of the coating and adhesive. For the cut tests, both the tape used and the angle the knife cuts the coating is found to impact the results. For tensile tests the type of dollies used affects the result [27]. In the present study we performed a pull-off test according to ISO 4624 [28] and a cross-cut test according to ISO 2409 [29]. Pull-off tests are usually not measuring adhesive fracture since the coating typically fails cohesively, therefore a cross-cut test was included. The latter also gives more reliable wet adhesion results. The tests were performed both in dry and in wet conditions. For wet adhesion testing, samples had been immersed in tap water at 10 °C to room temperature for one week for saturation of the coating prior to testing.

For pull-off tests, three test dollies were glued to each coated surface and pulled off after one day of curing. The dry adhesion test measures the minimum tensile stress necessary to detach the coating in the perpendicular direction to the substrate. Results are reported in MPa. A right-angle lattice pattern was cut into the coating for the cross-cut test and the cut-area was examined and classified from 0 (good adhesion) to 5 (bad adhesion) by a visual comparison with the illustrations in the standard. The test does not assess the adhesion quantitatively but gives a qualitative indication on the adhesion of the coating to either the preceding coat or the substrate.

2.3 Impact toughness

To evaluate the resistance of the dry film of paint to cracking or peeling, an impact toughness test was performed according to ISO 6272-1 [30]. A 20-mm-diameter spherical indenter was dropped on the samples under standard conditions and about 50% relative humidity. The mass of the falling weight used was 2 kg, and the drop height was increased until deformations were produced. The test measures the minimum drop height which will cause cracking or peeling when coating is subjected to a falling weight, and results are reported in Joules (energy absorbed).

2.4 Accelerated weathering laboratory test

To evaluate the resistance to underfilm corrosion creep and cathodic disbonding, an accelerated weathering test was performed according to ISO 20340 [31]. Samples were scribed with a 2 mm milling cutter down to the steel substrate in order to initiate corrosion. The length of the scribe was 50 mm. Three parallels of each machining process and coating system were exposed to cyclic tests intended to have a duration of 25 weeks. However, due to rapid degradation the samples were collected after 7-8 weeks, photographed and degraded coating scraped away for measurement of corrosion creep under the coating from the scribe. The amount of cathodic disbonding of the coating in front of the corrosion was also evaluated.

2.5 Surface characterization

The surface roughness was measured with a profilometer on unpainted machined samples. The profilometer is a contact method where the stylus tip moves on the surface in order to measure traditional surface parameters as Ra, Rq and Rz. Scanning Electron Microscopy (SEM) of cross sections was used to examine the surface morphology of the different machined substrates studied.

3 Results

3.1 Adhesion

The results from dry adhesion tests showed contrary to expectation that adhesion was just as good for the machined surfaces as it was on the blast cleaned surfaces. Both pull-off adhesion tests and cross-cut tests revealed only minor differences.

Pull-off testing gave fracture strengths between 16 and 21 MPa for all samples. The coatings generally failed cohesively, so the test did not actually measure adhesion. The metallized reference samples gave cohesive fracture in the zinc layer. There was no difference between machined samples and blast cleaned samples. Pull-off strength of wet coatings was also in this range. The pull-off test was performed on samples with glued dollies cured for 24 hours. The wet pull-off test is recommended to be conducted within 24 hours of removal from water as coating adhesion is found to recover most of its initial value as the water evaporates from the film [27]. Apparently samples regained adhesive strength.

The cross-cut test on the other hand differentiated better between the surface preparation methods. The wet adhesion was much lower on machined surfaces with the epoxy/polysiloxane coating than on the blast cleaned and metallized surfaces, see results in Table 2. For the antifouling coating the wet adhesion was comparable to the dry adhesion.

Table 2: Cross-cut adhesion measurements in wet and dry conditions.

	Material	**Machining**	**Cross-cut [score]**	
			Dry	**Wet**
A1	Mild steel S355 EN10025	End milled	2	3
A2		Rolled milled		
A3		Turned	2	0-1
A5		Blast cleaned		
B1	Cast iron SG400-12	End milled	2	0-1
B2		Rolled milled	2	0-1
B3		Turned	2	0-1
B4		Blast cleaned		
C1	S355 J2G3	End milled R_a=1.6 µm	1-2	3
C2		End milled R_a=12µm	1-2	4-5
C3		Angle grinded	1-2	5
C4		Zinc metallized	1-2	1-2
D1	S690QL	End milled R_a=1.6 µm	1-2	4
D2		End milled R_a=12µm	1-2	5
D3		Angle grinded	1-2	4
D4		Zinc metallized	1-2	1-2

3.2 Impact toughness

Results from impact toughness tests can explain some of the experienced problems with organic coating on machined surfaces. The impact resistance testing showed only small differences among the different machining methods. However, impact resistance was found to be about four times higher on blast cleaned surfaces, see Table 3. It is to be noted the considerable difference between the two reference samples C4 and D4. The substrates are of different materials, and D4 was prepared of a harder material which probably explains the large difference between C4 and D4.

Table 3: Impact toughness measurements.

	Material	Machining	Impact toughness [J]
A1	Mild steel S355 EN10025	End milled	5
A2		Rolled milled	7.5
A3		Turned	5
A4		Blast cleaned	18
B1	Cast iron SG400-12	End milled	7.5
B2		Rolled milled	5
B3		Turned	7
B4		Blast cleaned	18
C1	S355 J2G3	End milled R_a=1.6 µm	3
C2		End milled R_a=12µm	4
C3		Angle grinded	3
C4		Zinc metallized	19
D1	S690QL	End milled R_a=1.6 µm	3
D2		End milled R_a=12µm	3
D3		Angle grinded	3
D4		Zinc metallized	3.5

3.3 Corrosion

Very poor corrosion creep resistance was found on all machined samples already after 7-8 weeks in the ISO 20340 test, with some minor differences between the various machining methods. On all the machined samples several mm cathodic disbonding was found in front of the corrosion creep. No corrosion creep was found on the zinc metallized reference samples. Table 4 shows information about the measured corrosion creep on all samples. Values are obtained by averaging over 9 measurements on three parallels according to the test standard.

While the measured corrosion creep was 7 mm on the angle grinded samples C3 and D3, it was found to be twice as high on the other machined samples. Cathodic disbonding was believed to be the precursor of the corrosion process on all samples, but on angle grinded also some blistering was identified.

A close visual inspection of samples after removal of disbonded coating layers, see Figure 1, shows that red rust has formed uniformly at the defect spreading under the coating. Also black rust having formed in circular shapes can be seen in the close vicinity of the defect. However, no corrosion products can be seen in the areas nearest the disbonding front.

Table 4: Corrosion creep measured on samples.

	Material	Machining	Corrosion creep [mm]	Cathodic disbonding [mm]
A1	Mild steel S355 EN10025	End milled	11	3
A2		Rolled milled	13	6
A3		Turned	14	5
A4		Blast cleaned		
B1	Cast iron SG400-12	End milled		
B2		Rolled milled		
B3		Turned		
B4		Blast cleaned		
C1	S355 J2G3	End milled R_a=1.6 μm	12	2
C2		End milled R_a=12μm	13	2
C3		Angle grinded	7	0
C4		Zinc metallized	0	0
D1	S690QL	End milled R_a=1.6 μm	13	1
D2		End milled R_a=12μm	13	1
D3		Angle grinded	6	0
D4		Zinc metallized	0	0

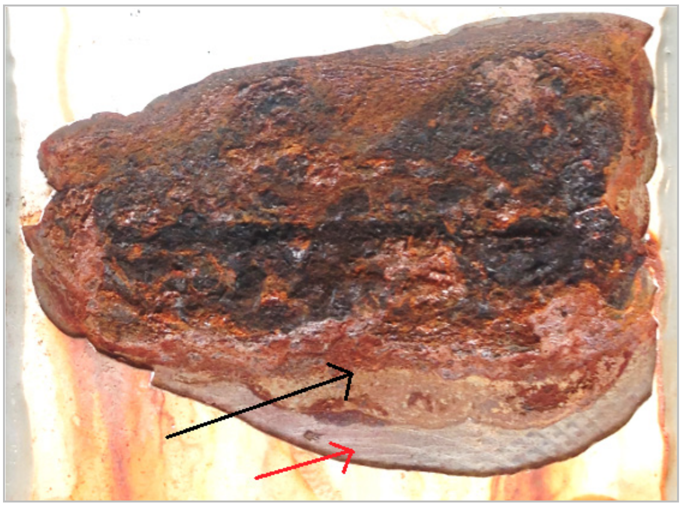

Figure 1: Corrosion creep identified on milled sample C1. Red arrow indicates cathodic disbonding while the black arrow points out the area with corrosion products.

3.4 Surface characterization

Only small differences in surface roughness were found among the machining methods, all measuring Ra between 0.6-2.6 μm. Images taken by SEM confirmed this, see Figure 2.

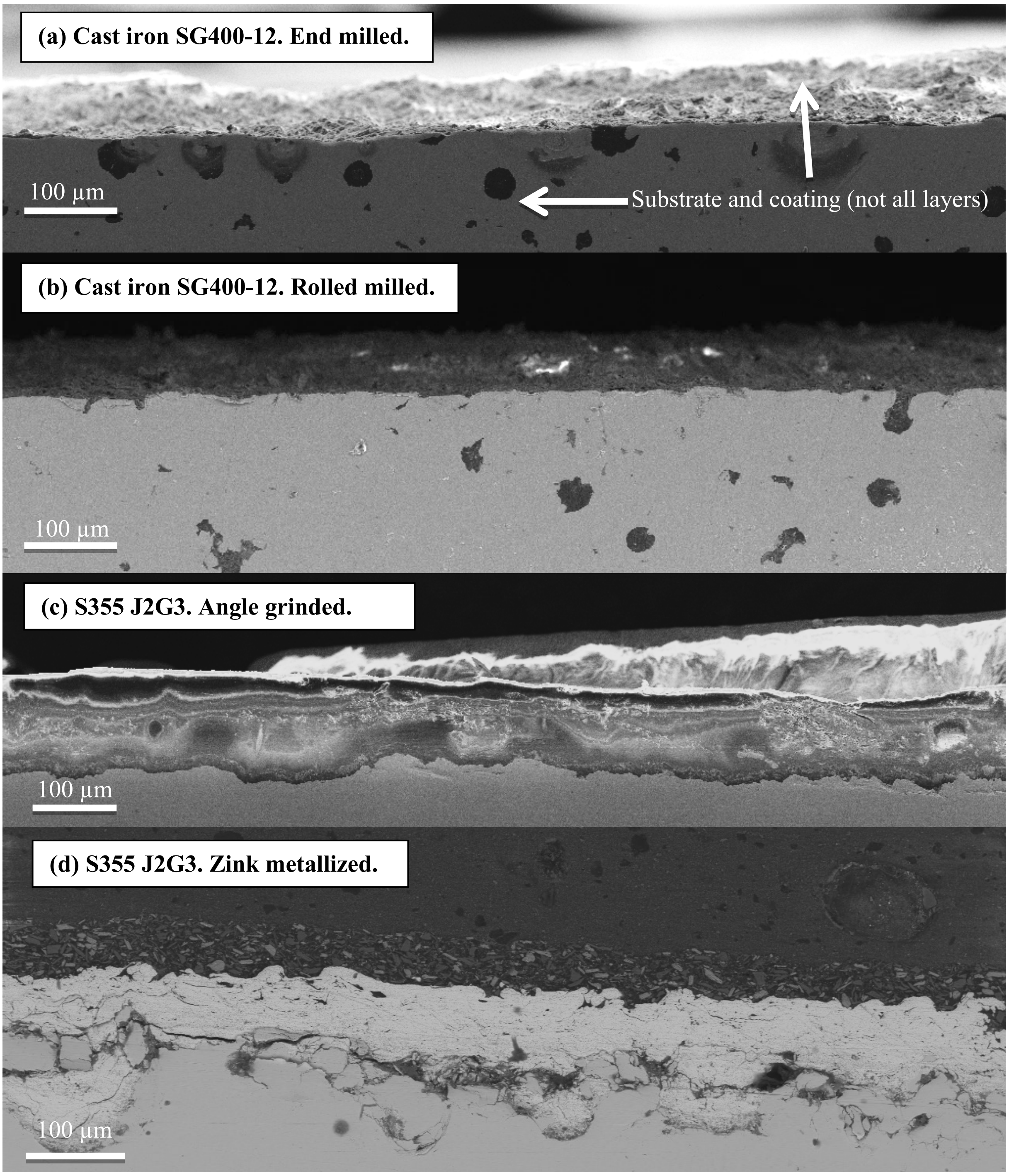

Figure 2. SEM images of machined surfaces (a-c) and the metallized surface (d). Some corrosion may be seen on substrate due to preparation for microscopy. White spots are areas with charging of electrons, possibly due to organic materia (coating) being smeared out during polishing.

4 Discussion

It is stated that corrosion cannot happen as long as adhesion persists [4, 13, 32]. It is therefore believed that a combination of weak wet adhesion and poor impact toughness is of importance for the observed coating failures on machined surfaces in the field. From impact toughness tests we see that coatings on machined surfaces are more prone to damage caused by impacts, as impact toughness was on the average four times higher on blasted compared with the machined surfaces. We believe that a substrate profile with low surface roughness on machined surfaces will not be able to absorb lateral forces. This probably partly explains the poor performance of coatings on machined surfaces.

Wet cross-cut tests showed that coatings on machined surfaces loose much of their adhesion strength when saturated with water. Cross-cut tests on machined surfaces gave quite different results, as the score on three of the wet samples was actually very good. Wet pull-off tests measured high pull-off strength even when testing happened within 24 hours of exposure. All dry adhesion tests gave good results and therefore little information about the different corrosion behavior of machined surfaces compared to blasted. It is to be noted that both the pull-off and the cross-cut test are characterized by several disadvantages: They may overestimate adhesion as a result of energy loss in deformation of the coating. When performed in wet conditions, dollies have to be glued with the same glue used for dry conditions, but the glue may be affected by the water in the film. Water may also evaporate during curing of the glue. Pull-off usually results in cohesive fracture. The main disadvantage with the cross-cut test is that it is a qualitative test. Besides, the state of the coating (wet/dry, degree of curing etc) may significantly affect how the forces from the knife are transmitted to the substrate/coating interface.

These results, and results from other studies [33], have shown that moisture in the coating reduce adhesion strength. However, it is not clear why this has such dramatic effect on machined surfaces and not on blast cleaned ones. It is hard to see how the physical forces and chemical bonds between the coating and the steel substrate can differ between machined and blasted surfaces.

On all machined surfaces significant cathodic disbonding was found in front of the corrosion creep. Hence, it seems that cathodic reactions first cause the coating to detach from the steel, and corrosion comes after. The observed red rust Fe_2O_3 (hematite) usually forms due to high oxygen and water exposure, while black Fe_3O_4 (magnetite) usually forms when oxygen diffusion to the steel surface is limited. Alkalis generated at cathodic sites inhibit corrosion but destroy adhesion, hence eventually leading to more and more exposed steel. Oxygen is during this process depleted by the cathodic reaction, while the value at the defect is higher. Concentration differences in oxygen will form aeration cells, resulting in a polarity switch: the initial cathodic site will become anodic. It has been shown in several studies [34-36] that for unpolarized samples, cathodic disbonding propagates by a series of electrochemical cycles where initially cathodic sites under the coating get depleted of oxygen and switch polarity and become anodic. The previously anodic areas – covered by red rust - will now deliver cathodic currents to the electrochemical circuit. Possible cathodic reactions will foremost be the oxygen reduction, and the reduction of hematite Fe_2O_3 to magnetite Fe_3O_4. Cathodic reactions might also happen at other sites close to the anodic, hence resulting in an advancing disbonding front.

The transport of cations to cathodic sites will be greatly enhanced by adhesion loss, as rate determining step (RDS) for the cathodic disbonding has been found to be the transport of cations along the metal-coating interphase and not the path through the coating [8, 37]. According to Sørensen (2009) the findings could mean that the abrasive blasting, to some extent, can be used for minimizing the rate of cathodic delamination. It seems likely that the degree of wet adhesion, related to the surface roughness of the substrate is of major influence on cathodic disbonding and corrosion creep rate.

5 Conclusions

Cathodic disbonding seems to be a precursor to corrosion creep, so increasing the resistance against cathodic disbonding in some way may improve coating durability. From the above results it is possible to suggest four hypotheses for coating degradation on machined surfaces.

1. The low surface roughness affects impact toughness resistance: defects are easily created.
2. The wet adhesion properties of the coating are low
3. Cathodic disbonding spreads easily on machined surfaces.
4. Machining activates the surface, and cathodic and anodic local spots will more readily be formed on the surface.

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