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Subject:	Final Report on the Salt Spray Testing comparative analysis of hot-dip galvanised carbon steel and 3CR12 stainless steel friction rock stabilizer split-sets ex Mpumatube	
Keywords:	Salt Spray Testing; hot-dip galvanised carbon steel; 3CR12; friction rock stabilizer split-sets; roof bolts; ASTM B117-97; Mpumatube	

1. BACKGROUND:

Two friction rock stabilizer split-set samples of hot-dip galvanised carbon steel and 3CR12 stainless steel were submitted by L Mashigo (TCS) via Mpumatube for a 1000hr salt spray exposure comparative analysis. This request was deemed quite feasible due to the fact that 3CR12 3mm No.1 material had been identified as a replacement for the hot-dip galvanised carbon steel friction-rock stabilizer split-sets in the mining application. The main reason for the material substitution was for the additional benefit of increased corrosion resistance. Although this open section bolt design was initially considered, other designs particularly of a 'Kidney tube design' (involved reverse bending of the split-sets) was also considered. However, it was noted that the testing of this design in this environment would be considered at a later stage. Prior to testing, it was clear that the exposure to this continuous salt spray environment was not ideal, as this environment did not fully consider the external mining environment, where other corrosive solutions could potentially be present. Hence samples of mine effluent and run-off were requested to fully categorise the corrosive substance/solutions that could be present in this environment.

It must be noted that salt spray testing is not a very accurate means of determining the corrosion resistance of stainless steel exposed to the environment, due to the fact that it does not include alternate wet/dry cycles as well as the effect of UV light on the material surface.

It has been shown by workers that the corrosion product of actual environment testing and cyclic testing with UV exposure produce different corrosion products and types of corrosion.

However, due to the vast amount of data collected using Salt Spray Testing, it was deemed the best tool to use for comparative purposes as well as the fact that the testing cycle is quite short hence allowing for faster results and comparisons.

This report summarises the final comparative findings of the 1000 hour (6 week) continuous salt spray testing that is currently being conducted on the hot-dip galvanised and 3CR12 friction rock stabiliser split-sets.

2. METHOD:

A Weiss SC 450 Salt Spray Test Chamber was used for the testing. The experimental procedure was based on the following ASTM Standard Practices viz. ASTM B 117 – 97 - Practice for Operation Salt Spray (Fog) Apparatus, ASTM G 85-02 - Practice for Modified Salt Spray (Fog Testing) as well as to the ASTM Standard G1-03 - Practice for Preparing, Cleaning and Evaluating Corrosion test specimens.

(A) APPARATUS:

Salt Spray Test Chamber – Weiss Type SC 450

- Air Supply: Clean compressed air was applied to the atomizing nozzle at a pressure of 100 kPa ± 20 kPa.
- Water: All the reagent water used conformed to type IV water in ASTM specification D 1193. The pH value measured was between 6 - 7 and the conductivity between 5-20 µS/cm.
- Temperature: The exposure zone temperature for the salt fog test was set at 35ºC according to ASTM B 117-97. The temperature set point was set to 35^0C which was well controlled.

| Photograph 1 showing the Weiss Salt Spray Test Chamber. | Photograph 2 showing the instrumentation used to control temperature and humidity. |

(B) PREPARATION OF TEST SPECIMENS:

Minimal sample preparation was required prior to placing the samples in the chamber. However an 'X' was inscribed on the exposed tube surface of both tube bolts by using an angle grinder. The tubes were then thoroughly cleaned with ethanol after inscribing, and prior to insertion into the salt spray chamber. The samples were placed on a piece of polystyrene to support the samples and prevent contact between them. This also aided in propping up the samples at the required angle of 60^0 according to the Standard ASTM Procedure B117-97.

Sample Label	Tube	Surface Finish	Test Position
Sample 1	Mild Steel	Standard	Vertical
Sample 2	3CR12	Standard	Vertical

Table 1: showing the sample labelling convention.
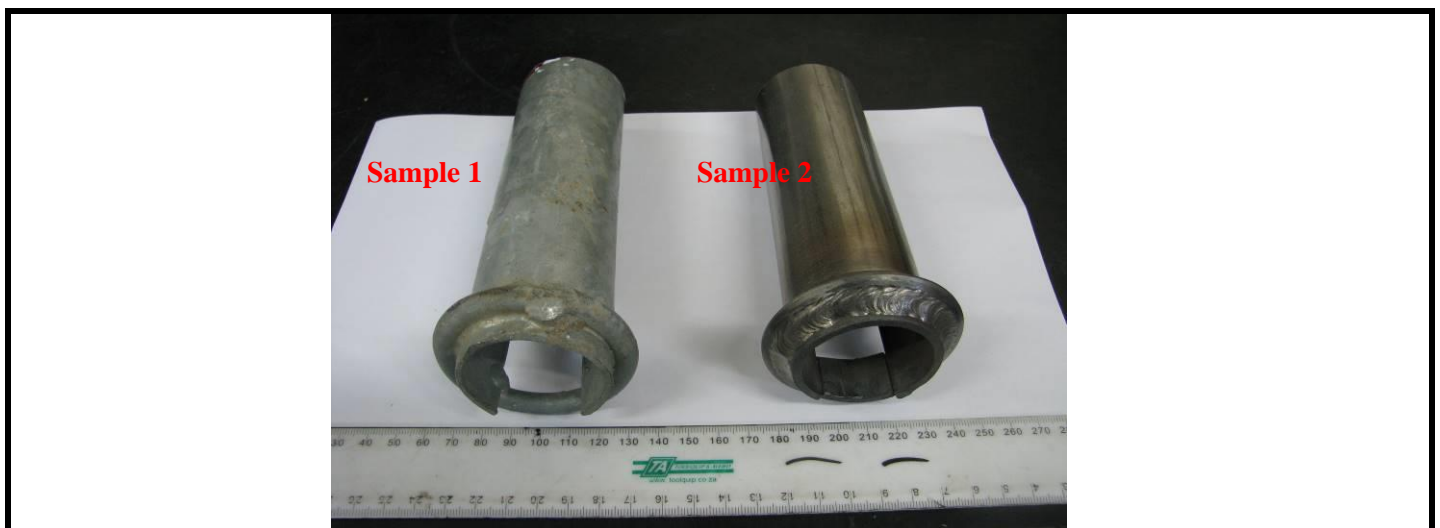
Photograph 3 showing the submitted sample labelling after cleaning prior to placement in the chamber.

Photograph 4 showing a close-up of the Sample 1 surface.

Photograph 5 showing a close-up of the Sample 2 surface.

Photograph 6 showing the 'X' marking on the samples after cleaning.

Photograph 7 showing the sample placement in the chamber prior to testing.

Photograph 8 showing another view of the sample placement into the chamber prior to testing.

(C) REAGENTS USED & MEASUREMENTS TAKEN:

Test Solution:

The pH of the salt solution in the reservoir was: At 25ºC = 5.8 and at 35ºC = 6.1
The average pH of the atomized solution collected in the fog collectors at 35ºC was 6.2. The solution collected in the fog collectors was boiled for 30 seconds before the pH reading was taken to get a close estimate of the pH of the atomized solution at 35ºC.
The salt solution was prepared according to ASTM Practice B 117-97 using 5 parts by weight of sodium chloride (1.25 kg of laboratory grade NaCl – free of bacteriological endotoxins) in 95 parts of reagent water (25 litres of RO water) and the solution had a specific gravity of 1.0341. This conformed to the specification given in the ASTM G 85-02 Practice for Modified Salt Spray (Fog) Testing.

Temperature:

The temperature in the exposure zone was taken once a day without opening the chamber. The average temperature over the entire testing period was 35.2ºC with a standard deviation of 0.4ºC.

Exposure Time:

As there is no mention of a specified exposure time in the ASTM Standard for the submitted samples, it was decided to expose the samples to at least 1000 hours of continuous Salt Spray. In this case, if no corrosion took place on any sample, then the samples would remain exposed until the onset of corrosion

Fog Collector Tests:

These tests as specified in the standard were conducted to obtain the correct spray nozzle setting in the chamber. In brief, the amount of fog condensate collected in a beaker with a funnel was used to set the spray nozzle for the correct amount of fog into the exposure chamber.

3. SURFACE VISUAL EXAMINATION AFTER 1000 HOUR EXPOSURE:

The submitted samples were exposed to continuous Salt Spray (fog) for a period of 1000hrs (6 weeks). Photographs are presented below to illustrate the effect of the salt spray testing on the samples after the continuous 1000 hour exposure. A summary of photographs is presented in Appendix 1 later in this report to illustrate the effect of the progressive week by week exposure of the samples in the Salt Spray Chamber.

| Photograph 9 showing the placement of the samples in the chamber prior to exposure. | Photograph 10 showing the samples after removal from the chamber post 1000 hour salt spray exposure. |

Photograph 11 showing the severe corrosion deposits present in the scribed section of Sample 1 after the 1000 hour exposure.

Photograph 12 showing a close-up of the corrosion and run-off staining in the scribed section of Sample 2 after the 1000 hour exposure.
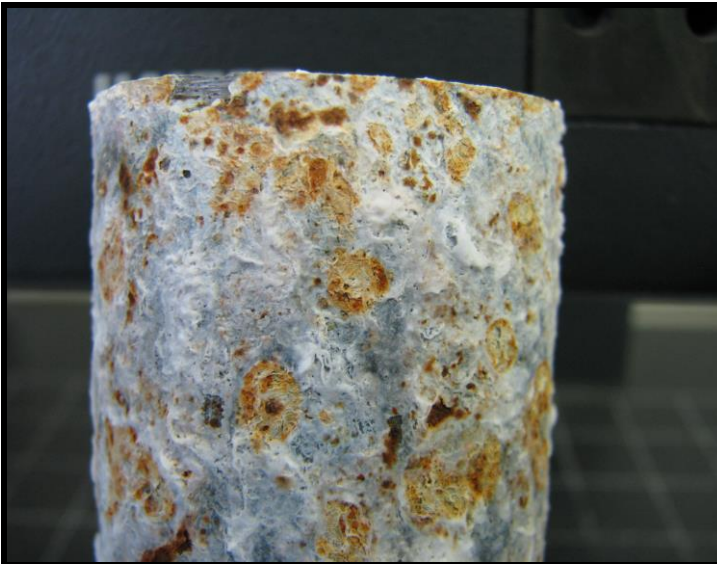
Photograph 13 showing the corrosion at the top section of Sample 1 after the 1000 hour exposure.
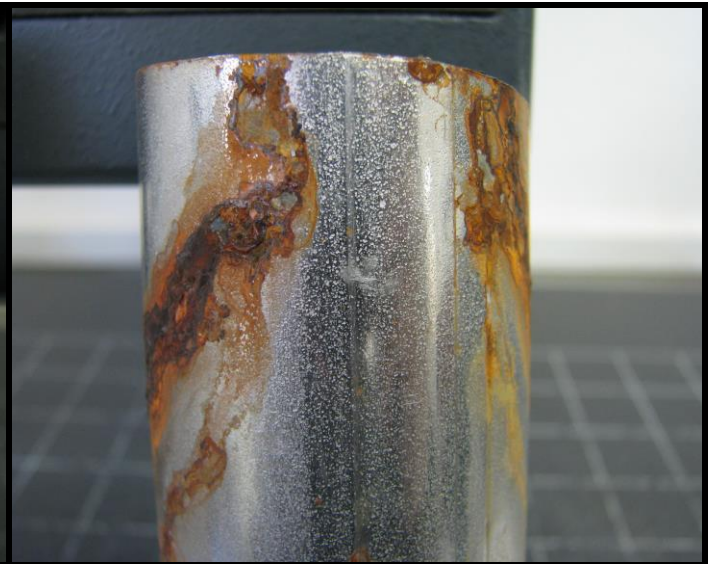
Photograph 14 showing the run-off staining at the top of Sample 2 after the 1000 hour exposure.

Photograph 15 showing the corrosion on the bottom section of Sample 1 after the 1000 hour exposure.

Photograph 16 showing the corrosion due to the run-off staining at the bottom section of Sample 2 after the 1000 hour exposure.

| Photograph 17 showing the inner surface of Sample 1 after the 1000 hour exposure. | Photograph 18 showing the inner surface of Sample 2 after the 1000 hour exposure. |

4. RESULTS & CONCLUSIONS:

The visual examination of the friction rock stabiliser split-set revealed that the submitted Sample 1 had some minor damage to the galvanising on the surface (see Photograph 4 - this would have most likely occurred during the insertion of the sample during operation). There were also a few minor scratches on the Sample 2 surface, but this would not have significantly influenced the corrosion rate. The tubes were of similar gauge.

From the salt spray (fog) testing conducted on the submitted samples when considering the progressive week on week corrosion as well as the final exposed samples the following became evident:

- After only the initial 24 hour salt spray exposure, there was clear evidence of white corrosion product being present on the Sample 1 surface (see Photograph 19). This white corrosion product is typical of the corrosion product experienced on galvanized steel exposed to continuous moist conditions (without drying) and consists mainly of zinc oxide which is quite voluminous (occupies between 20-25% more volume than zinc) and is loosely adherent to the surface. Reddish-brown staining was detected on the inscribed section of Sample 2, as well as the top cut edge of Sample 2 (see Photograph 20). Some run-off was also detected on the top cut edge and the bottom markings of the inscription. This reddish-brown staining was also detected in the bottom crevice between the tube and the lip (see Photograph 20).
- It was clear after 72 hours of salt spray exposure that the white corrosion product areas present on Sample 1 had increased. This corrosion product was present on the entire sample (see Photograph 21). The reddish-brown staining and run-off staining on the Sample 2 exposed surface had also increased (see Photograph 22).
- After 96 hours of salt spray exposure, the white corrosion product areas had again increased (see Photograph 23). The reddish-brown run-off staining from both the top cut edge and the bottom inscribed edges of Sample 2 exposed surface had also increased (see Photograph 24).
- Similarly after the 168 hour (1 week) salt spray exposure, the white corrosion product areas had increased, with the exception that there was now some slight reddish corrosion product at the center of the inscription (see Photograph 25), indicating that the corrosive attack of the underlying carbon steel had initiated. The reddish-brown run-off staining from both the top cut edge and the bottom inscribed edges of Sample 2 exposed surface had also increased (see Photograph 26).
- Similarly after the 336hour (Week 2) and 504hour (Week 3) exposure, there was again increases in the white corrosion product areas (see Photographs 27 and 29). The reddish corrosion product at the center of the inscription had also increased, indicating that the corrosive attack of the underlying carbon steel was progressing. The reddish-brown run-off staining from both the top cut edge and the bottom inscribed edges of Sample 2 exposed surface had also increased (see Photographs 28 and 30).

- After the 672 hours salt spray exposure time (Week 4), the white corrosion product had increased on the Sample 1 surface, with increases of the reddish brown corrosion product at the inscription. Of note was the significant increase in the brown corrosion product through the entire surface of Sample 1 (see Photograph 31). This again confirmed that the corrosion of the underlying surface of the metal was progressing. The Sample 2 surface appeared quite similar to the previous examination with only a slight increase in the run-off staining (see Photograph 32).
- After 840 hours salt spray exposure (Week 5), the white corrosion product as well as the reddish brown corrosion product had again increased on the inscription as well as the entire surface (see Photograph 33). The Sample 2 surface appeared quite similar to the previous examination (see Photograph 34).
- After the 1008 hours of salt spray exposure (Week 6), the entire surface of Sample 1 was covered with the white corrosion product (consisting mainly of Zinc oxide) – see Photograph 10. The reddish brown corrosion product had increased significantly over the entire Sample 1 surface (see Photograph 10). The Sample 2 surface showed typical run-off staining with the absence of any pitting or general corrosion present (see Photograph 10). The close-up examination of the inscription on the Sample 1 surface revealed significant reddish brown corrosion product confirming that the metal had been severely attacked (see Photograph 11). Similarly, there was excessive run-off staining emanating from the Sample 2 inscription (see Photograph 12). The top and bottom Sample 1 surface exhibited significant reddish brown corrosion product over the entire surface confirming that the underlying metal was corroding (see Photographs 13 and 15). The run-off from the top and to the bottom of Sample 2 was quite excessive (see Photographs 14 and 16). The inner surfaces of both samples showed no major corrosive attack (see Photographs 17 and 18).

Hence from the salt spray testing conducted after the 1008 hour exposure on the submitted friction rock stabilizer split-set samples tube samples, it was clearly evident that white corrosion product areas in the form of zinc oxide was heavily present on the entire Sample 1 surface. There was also evidence of reddish brown corrosion product present in the inscription of this sample as well as the entire surface, confirming that the underlying corrosion of the base metal (carbon steel) was progressing rapidly. The Sample 2 surface had evidence of heavy run-off staining present at the top cut-edge as well as the bottom inscription areas of the sample. There was also evidence of heavy staining at the crevice created by the lip of the sample. Of note was the lack of pitting or general corrosion visually present on the Sample 2 surface, after the 1000 hour exposure.

Finally one can conclude from the analysis conducted after the 1000 hour salt spray exposure, that the hot-dipped galvanised mild steel friction rock stabiliser split-set sample exhibited a much more severe type of corrosion with the initial galvanised surface being oxidised and then the subsequent mild steel underneath. It must also be clearly noted that once the hot-dipped galvanised layer has corroded away, the underlying metal corrosion is significantly increased exponentially, which does not bode well for the corrosion resistance of the underlying metal. This increase in corrosion has been found to be approximately 3 times more when compared to 3CR12 (from previous testing conducted). This was evident due to the surface oxidation corrosion product by visual examination present on the entire sample surface. Less severe corrosion of the 3CR12 rock stabiliser split-set was experienced, with mainly the run-off from the cut-edge having a significant corrosion effect on the stainless steel. Due to the lack of mass loss on the samples experienced, analysis could not be conducted for a more accurate corrosion rate calculation.

5. REFERENCES:

ASTM Standards: B 117 Practice for Operation Salt Spray (Fog) Apparatus; G 85-02 Practice for Modified Salt Spray (Fog) Testing; G1-03 Practice for Preparing, Cleaning and Evaluating Corrosion test specimens.

Kavith Nundlall, Metallurgical Services & Development, 29th May 2018

ADDENDUM:

A query involving the integrity of the welds on both samples after the completion of the salt spray testing conducted was generated after the distribution of this report. Below is a summary of photographs showing the welds of both samples after mechanical cleaning with a polypropylene brush to remove the excessive run-off, and the subsequent chemical cleansing by immersion of both the welds in a 20% Nitric acid solution for 20 minutes, to remove the remaining deposits.

Photograph 19 showing the Sample 2 weld after mechanical and chemical cleansing.	Photograph 20 showing another area of the Sample 2 weld after both mechanical and chemical cleansing.

Photograph 21 showing the Sample 1 weld after mechanical and chemical cleansing.	Photograph 22 showing another area of the Sample 1 weld after both mechanical and chemical cleansing.

Visual Analysis:

The visual examination conducted on both the welds of Samples 1 and 2, revealed the absence of any pitting or other detrimental corrosion effects present in the welds, after performing both the mechanical and chemical cleansing. This confirmed that both the welds showed good integrity.

Appendix 1:

The progressive visual examination and photographs of the corrosion on the submitted samples on an initial daily and weekly basis are presented below:

Day 1 – 20/03/2018 (after 24 hours exposure):

| Photograph 23 showing the Sample 1 surface after 24 hours of continuous salt spray exposure. | Photograph 24 showing the Sample 2 surface after 24 hours of continuous salt spray exposure. |

Day 3 – 22/03/2018 (after 72 hours exposure):

| Photograph 25 showing the Sample 1 surface after 72 hours of continuous salt spray exposure. | Photograph 26 showing the Sample 2 surface after 72 hours of continuous salt spray exposure. |

Day 4 – 23/03/2018 (after 96 hours exposure):

Photograph 27 showing the Sample 1 surface after 96 hours of continuous salt spray exposure.	Photograph 28 showing the Sample 2 surface after 96 hours of continuous salt spray exposure.

Week 1 – 26/03/2018 (after 168 hours exposure):

Photograph 29 showing the Sample 1 surface after 168 hours of continuous salt spray exposure.	Photograph 30 showing the Sample 2 surface after 168 hours of continuous salt spray exposure.

Week 2 – 03/04/2018 (after 336 hours exposure):

| Photograph 31 showing the Sample 1 surface after 336 hours of continuous salt spray exposure. | Photograph 32 showing the Sample 2 surface after 336 hours of continuous salt spray exposure. |

Week 3 – 09/04/2018 (after 504 hours exposure):

| Photograph 33 showing the Sample 1 surface after 504 hours of continuous salt spray exposure. | Photograph 34 showing the Sample 2 surface after 504 hours of continuous salt spray exposure. |

Week 4 – 16/04/2018 (after 672 hours exposure):

| Photograph 35 showing the Sample 1 surface after 672 hours of continuous salt spray exposure. | Photograph 36 showing the Sample 2 surface after 672 hours of continuous salt spray exposure. |

Week 5 – 23/04/2018 (after 840 hours exposure):

Photograph 37 showing the Sample 1 and Sample 2 surface after 840 hours of continuous salt spray exposure.

Photograph 38 showing the Sample 1 and Sample 2 surface after the 1008 hours of continuous salt spray exposure.