A three month accelerated test according to NACE standard TM108-2008 Item No 21252 on iridium oxide, tantalum oxide mixed metal oxide coatings on titanium for impressed current anodes

By

Professor David Scantlebury, MA PhD FICorr
and
Ms Yuanfeng Yang BSc MSc

For Titanium Electrode Products Inc.
PO Box 2467
Stafford, Texas 77497-2467
United States of America

14th August 2009

Professional Biography of Professor David Scantlebury

Professor Scantlebury's research interests and his major teaching are closely interrelated. For the whole of his academic career (nearly forty years!), he has been interested in the strange situation between a potentially corroding metal and the organic coating that is placed on that metal with the intention of preventing corrosion of that metal. There is a fascinating mixture of knowledge and disciplines overlapping, that Professor Scantlebury finds attractive, including metallurgy, surface science, adhesion, electrochemistry, and polymer science. And all this arises from real problems with real solutions.

The Professor' other related research interests include marine corrosion, cathodic protection, and the corrosion and corrosion control of rebar steel in concrete. He teaches all these subjects mainly in Unit 4 in the MSc in Corrosion Control Engineering at the University of Manchester but as well gives most of the year two undergraduate lectures in the Materials Science degree course. Since 1989, he has organised a five yearly international conference on corrosion protection by organic coatings at Christ's College Cambridge and two major Conferences on Cathodic Protection in Manchester. He is a recipient of the U.R. EvansAward from the Institute of Corrosion for outstanding Internationally recognised achievements in Corrosion Science and Engineering.

His administrative duties include being the course director for the MSc course in Corrosion Engineering and Control and the one week annual Short Course in Corrosion Engineering, now in its 35th year.

Professor Scantlebury also currently holds a visiting Chair in the Department of Chemistry, University of Xiamen, China.

CONTENTS:

1.0 Executive summary

Four coated titanium anodes were supplied to us by Titanium Electrode Products Inc and tested for three months according to **NACE standard TM108-2008 Item No 21252 "Testing of catalysed titanium anodes for use in soils or natural waters"**.
The acceleration factor in this accelerated test is 100; the maximum operating current density is scaled up from 100A/m^2 to 10,000A/m^2. The test period is therefore reduced by a factor of 100, i.e. a 91 day test equates to a 25 year life.

According to TM108, three anode failure criteria are given;

"Anode failure can be detected by a significant rise in cell voltage and anode potential while operating at the required constant current density". [Page 1]

"Anode failure is marked by a rapid escalation in both cell voltage and anode potential". [Page 5]

"The time of failure shall be recorded when the anode potential increases by 4.0V above its initial value". [Page 5]

In all four specimens tested for the 91 day period;

- there was a steady linear increase in cell voltage with no rapid escalation.
- there was a steady linear increase in anode potential with no rapid escalation.
- the anode start and end potential differences in all cases were less than 4.0 volts.

The anodes clearly have passed the test.

According to NACE standard TM108-2008 Item No 21252; these anodes will therefore exceed their design life of 25 years.

2.0 Introduction

Appendices A B and C are copies of three journal papers written by the author of this report which sets the background to these materials, their compositions, manufacture, performance and accelerated testing.

2.1 Acceleration factor

The acceleration factor in this test is anode current density. As stated in TM108;

"For the total design service charge density, it is the current density for the anode in the intended service times the desired service life for the anode".
In this test the current density is increased by a factor of 100 from a maximum of 100A/m^2 to 10,000A/m^2. In our case this equates to 5A over the specimen area of 500 mm^2. The test period is therefore reduced by a factor of 100, i.e. a 91 day test equates to a 25 year life.

3.0 Minor modifications to NACE standard TM108-2008 Item No 21252

Section 3.1. Beakers were open topped and vented into an open and well ventilated laboratory
Section 3.1.4 The Luggin probes were solid state poly(acrylamide) salt bridges
Section 5.6 As soon as the power was energised, the solutions began heating up and immediately required placing in a cooling bath. The bath consisted of a water-filled stainless steel tank in which was placed a copper cooling coil through which tap water was passed. This system was found to maintain the electrolyte solutions within the recommended range 30±5^{o}C. The water bath was kept topped up daily.

4.0 Equipment

The power supply was Thurlby Thandar TTi EX4210R 42V 10A unit. The Digital voltmeter was TENMA 72-7730 with an input impedance of greater than 10 MΩ. It was calibrated against a Standard Weston Cell Type 1268 with a cell voltage of 1.01859V at 20^{o}C. Specimen photos were taken with a Microcapture digital microscope

5.0 Data

The test programme begun at 1245 BST on Tuesday 7th April 2009. All potentials were measured versus the Saturated Calomel Electrode (SCE).

Open circuit unpolarised potentials were measured on each electrode and were; Electrode 1 +0.3572V (SCE); Electrode 2 +0.3396V (SCE); Electrode 3 +0.3408V (SCE); Electrode 4 +0.3199V (SCE). The programme finished at 1600 BST on Monday July 6th 2009. Cell current was maintained at 5±0.01A for the duration of the test (91d) by daily adjustment. Cell current drift was always less than 0.01A during the daily period.

All the data, time, date, cell voltage, anode potential was recorded and is reported at the end of this report.

The four daily anode potentials are plotted graphically during the test period as are the four cell voltages and are at the end of this report, pages 8-11.

It can be seen from the anode potential and cell voltage graphs that in all four specimens tested for the 91 day period there was a steady linear increase in cell voltage with no rapid escalation and there was a steady linear increase in anode potential with no rapid escalation.

5.1 Anode potential change

The first few data points up to day five are more scattered than the later data. This is not unexpected as explained in our published work (refer to Appendices A, B and C). The original mud-cracked surface is uneven; there will be surface layers; some regions will be more reactive than others and will dissolve rapidly by wear and erosion under the influence of the intense oxygen evolution reaction. Thus the value for the initial voltage cannot be picked out from the actual data.

Therefore to obtain this initial voltage and the true increase in anode potential during the test, we have statistically fitted the data to a linear equation Y=A + B*X. To obtain A, the initial voltage, we have extrapolated this straight line back to time zero. The increase in anode potential is therefore B*X, the line gradient B multiplied by X the time, 91 days.

Anode 1	3.37±0.20V	Anode 2	3.33±0.27V
Anode 3	2.47±0.15V	Anode 4	3.27±0.17V

None of the above is greater than the specified voltage in the NACE standard of 4V

5.2 Visual appearance

Plates 1-4 are the four specimens after testing pages 13-14. Plate 5 is from the same batch, untested which had been sectioned for microscopy, Plates 6 and 7 were also untested and sectioned. Plate 8 is also untested.

From the visual appearance of the specimens, those which have been tested are visually indistinguishable from those which have not.

6.0 Conclusions

According to TM108, three anode failure criteria are given;

“Anode failure can be detected by a significant rise in cell voltage and anode potential while operating at the required constant current density”. [Page 1]

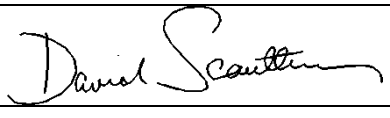
“Anode failure is marked by a rapid escalation in both cell voltage and anode potential”. [Page 5]

“The time of failure shall be recorded when the anode potential increases by 4.0V above its initial value”. [Page 5]

In all four specimens tested for the 91 day period;

- there was a steady linear increase in cell voltage with no rapid escalation.
- there was a steady linear increase in anode potential with no rapid escalation.
- the anode start and end potential differences in all cases were less than 4.0 volts.

The anodes clearly have passed the test according to NACE standard TM108-2008 Item No 21252; these anodes will therefore exceed their design life of 25 years.

	signature	legible	date
For Scantlebury Consultants		Prof. JD Scantlebury	14th August 2009