# Applicability and Limitations of the EPR-Test as a Substitute for ASTM G28 A Streicher-Test

<u>Gregor Mori<sup>1</sup></u>, Manuel Prohaska<sup>2</sup>, Guido Tischler<sup>3</sup>

<sup>1</sup>CD Laboratory of Localized Corrosion, Montanuniversitaet Leoben, Franz Josef Strasse 18, A-8700 Leoben Austria, <u>mori@unileoben.ac.at</u> <sup>2</sup>BHDT GmbH, Werk-VI-Strasse 52, A-8605 Kapfenberg, Austria <sup>3</sup>voestalpine Grobblech GmbH, voestalpine-Strasse 3, A-4020 Linz, Austria

## Introduction

Among intergranular corrosion tests there are standard tests which have been standardized many years ago as, for instance, Streicher-, Huey- and Strauss-test<sup>1</sup>. All these tests are immersion tests with a testing time between 24 and 240 h. This test procedure generally results in a pronounced degradation of the analysed samples by means of grain dropping if the investigated material conditions are sensitized. As an alternative test method, Cihal et al.<sup>2,3,4</sup> have developed an electrochemical method to characterize sensitization of materials, the so-called electrochemical potentiokinetic reactivation method (EPR-test). This test reduces testing time dramatically (a few minutes).

## Experimental

Tests have been done with material Alloy 926 with 0.01% C, 20.3% Cr, 24.9% Ni, 6.4% Mo, 0.9% Cu, 0.2% N and balance Fe. Different conditions have been produced. Solution annealed material was used as good benchmark, whereas isothermal sensitized specimens treated at 760°C for 20 h and 900°C for 120 h served as susceptible benchmarks. Additionally several thermomechanically treated specimens have been investigated. They were produced with different end rolling temperatures (850 and 950 °C) and cooling rates (water and air quench). Two specimens (950°C-water quench) have been further heat treated to investigate the impact of an additional heat treatment at 950 °C for 0.5 h, water quench, temper at 600 °C for 1 h (HT1) and 1000 °C for 0.5 h, water quench, temper at 600 °C for 1 h (HT2).

Corrosion tests were one the on side Streicher-tests according to ASTM G28A and on the other hand double-loop EPR-tests. EPR-test conditions are described elsewhere<sup>5</sup>.

Specimens have been characterized prior to corrosion testing with energy filtered TEM (EF-TEM), with EDS line scans in STEM mode and with electron diffraction to identify precipitated phases and quantify chemical composition of depletion zones. Type of attack after corrosion testing was characterized with a high resolution SEM.

#### **Results and Discussion**

Results of EF-TEM characterization of investigated alloys are combined in Table 1. All alloys except the solution annealed condition contain various amounts of different  $\sigma$ - and  $\chi$ -phases. All are enriched in Mo and some are enriched, some depleted in Cr. Chemical composition and width of depletion zones adjacent to precipitates are also presented in Table 1. Most critical are Mo-depleted zones near  $\sigma$ -phases of condition 760\_20. Chemical composition of  $\sigma$ - and  $\chi$ -phases say that after  $\chi$ -phase formation these precipitates transform into  $\sigma$ -phases during long term annealing. No EF-TEM images are included in the present extended abstract due to space limitation.

Corrosion results are presented in Figure 1. Streicher- and EPR-test give suitable degrees of sensitization. The only exceptions are isothermally annealed materials 760\_20 and 900\_120. While the material annealed at 760°C for 20 h shows pronounced grain dropping after Streicher-test due to continuous sensitization with Laves-phase, condition 900\_120 shows a

more uniform attack in both tests resulting in less weight loss during Streicher-test due to a lack of grain dropping (Figure 2).

Table 1: Precipitates in superaustentite stanness steel Alloy 926					
Sample designation	Phases	Composition	Amount	Location	Depletion zone (width and chem. composition)
Sol. ann.	-	-	-	-	-
760_20	Laves	$Mo\uparrow, Cr\downarrow (Fe_{29}Cr_{18}Ni_9Mo_{44})$	high	GB	300 nm Mo from 6.4 to 2.5 %
900_120	$\sigma_1$	$\begin{array}{c} \text{Mo}\uparrow, \text{Cr}\downarrow\\ (\text{Fe}_{11}\text{Cr}_{19}\text{Ni}_{22}\text{Mo}_{48})\end{array}$	high high	GB and G	>2000 nm Mo ( $6.4 \rightarrow 3.5 \%$ )
	$\sigma_2$	$\begin{array}{c} \text{Mo}\uparrow, \text{Cr}\uparrow\\ (\text{Fe}_{36}\text{Cr}_{32}\text{Ni}_{12}\text{Mo}_{20})\end{array}$		GB	>2000 nm Mo (6.4 → 3.5 %), little Cr↓
850_W	$\chi_1$	Mo $\uparrow$ , Cr $\uparrow$	medium	GB	300 nm Mo (6.4 $\rightarrow$ 3.5 %), little Cr $\downarrow$
850_A	$\chi_1$	Mo $\uparrow$ , Cr $\uparrow$	high	GB	300 nm Mo (6.4 $\rightarrow$ 3.5 %), little Cr $\downarrow$
950_W	$\chi_1$	Mo↑, Cr↑ (Fe <sub>36</sub> Cr <sub>30</sub> Ni <sub>12</sub> Mo <sub>22</sub> )	low	GB	300 nm Mo (6.4 $\rightarrow$ 3.5 %), little Cr $\downarrow$
950_A	$\chi_1$	Mo $\uparrow$ , Cr $\uparrow$	medium	GB	300 nm Mo (6.4 $\rightarrow$ 3.5 %), little Cr $\downarrow$
950_W_HT1	$\chi_1 \ \chi_2?$	$\begin{array}{c} Mo\uparrow, Cr\uparrow\\ (Fe_{36}Cr_{32}Ni_{12}Mo_{20})\\ Mo\uparrow, Cr\downarrow \end{array}$	high low	GB GB and G	300 nm Mo (6.4 $\rightarrow$ 4 %), little Cr $\downarrow$ not investigated
950_W_HT2	$\chi_1$	Mo↑, Cr↑	high	GB	1500 nm Mo ( $6.4 \rightarrow 5.5 \%$ ), little Cr $\downarrow$

Table 1: Precipitates in superaustenitic stainless steel Alloy 926

GB...grain boundary, G...Grain

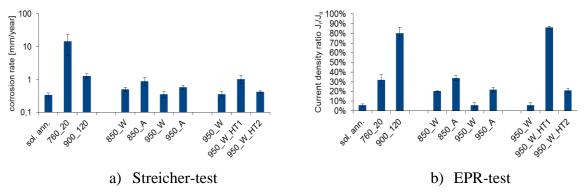
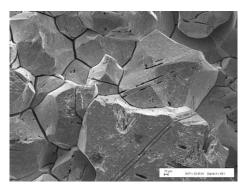


Figure 1: Corrosion rates of differently sensitzed conditions of Alloy 926

#### Conclusions

Mass loss in Streicher-test is determined by grain dropping which occurs when a continuous sensitization zone at grain boundaries is present. EPR-test gives large sensitization values when a large number of precipitates with a depleted zone of Cr and/or Mo is present.

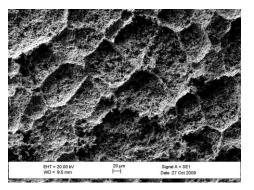
An advantage of EPR-method is its higher sensitivity at lower degrees of sensitization (DOS) when compared to standard immersion tests. As a disadvantage, highly-skilled laboratory staff is required to execute the EPR-test in a proper way. EPR-test is capable to replace Streicher-test when accepting that it has to be optimized for each single material and performed by skilled technicians.



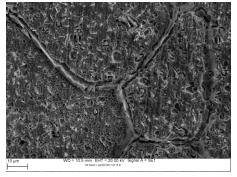
a) 760\_20 after Streicher-test: large extent of grain dropping



c) 760\_20 after EPR-test: continuous intergranular corrosion



b) 900\_120 after Streicher-test: more uniform attack, grain boundaries exhibit minor attack



d) 900\_120 after EPR-test: intergranular and uniform attack

Figure 2: SE images of isothermally annealed samples after Streicher- and EPR-test

Precipitates that result in sensitization of Alloy 926 are different  $\sigma$ - and  $\chi$ -phases, all are Mo enriched, some contain large amounts of Cr.

# References

- 1. ASTM Annual Book of ASTM Standards; Section 3: Metals Test Methods and Analytical Procedures; Volume 03.02: Wear and Erosion, Metal Corrosion, ASTM, 2000
- 2. V. Čihal, T. Shoji, V. Kain, Y. Watanabe, R. Stefec, Electrochemical Polarization Reactivation Technique: EPR-a Comprehensive Review, Fracture and Reliability Research Institute, Graduate School of Engineering, Tohoku University, 2004
- 3. V. Čihal, Intergranular Corrosion of Steels and Alloys, Elsevier Science Publishers, BV, 1984, 368-382
- 4. V. Čihal, R. Štefec, On the development of the electrochemical potentiokinetic method, Electrochimica Acta 46 (2001) 3867-3877
- M. Prohaska, G. Mori, H. Hofstätter, G. Tischler, R. Grill, Corrosion Properties of Different Highly Alloyed Clad Materials for Offshore Applications Manufactured by a New Thermo-Mechanical Rolling Process, proceedings of a conference Corrosion 2011, March 10<sup>th</sup> – 15<sup>th</sup> 2010, NACE, Houston, 2011, 1-13