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Use of Safurex® thin foil for Pressure, Level and Flow transmitters

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In the urea plant carbamate corrosive environments are commonly found in the HP synthesis, MP and LP sections. Carbamate, especially in the HP synthesis section, is very corrosive, even for stainless steels.

For delicate diaphragms used for pressure and differential pressure measurements this proves to be an even bigger challenge as there is (almost) no corrosion allowance, the diaphragm's thickness is somewhere near to 100 µm (0.1 mm/0.004 inch). The conditions are even worse for diaphragms in the vapor phase of such sections due to a phenomena called condensation corrosion.

Therefor the choice of material, the processing (shaping, welding) of such diaphragms and the application is particularly important.

The repair/replacement of a failed diaphragm type pressure or differential pressure instrument usually requires the shutdown of the process or the process unit. This leads to expensive downtime, capacity turn down or even potentially unsafe operations.

In 1996 Sandvik and Stamicarbon introduced Safurex®, a super duplex stainless steel specially developed for urea service. Now, after more than 25 years of operational experience, it is confidently confirmed that most of the typical corrosion problems that are present in urea plants, can be solved by using Safurex®.

This paper presents the development of Safurex® thin foil diaphragms for pressure and differential pressure measurements in carbamate corrosive environments in urea plants and other corrosive environments.

INTRODUCTION

Safurex® (UNS S32906) is a super duplex stainless steel for the Stamicarbon urea processes that was introduced in 1996[1]. Safurex® has gained an excellent track record since the introduction and has showed several advantages over traditionally used austenitic stainless steels [2-5]. The material shows excellent corrosion-resistance at low oxygen levels and active corrosion has never been observed in any urea equipment, not even when passivation air has been absent.

Condensation corrosion in the gas phase, crevice corrosion, chloride induced stress corrosion cracking (SCC) and strain induced intergranular cracking (SIIC) are not a problem in Safurex® plants as the material is practically immune to these forms of corrosion. Nowadays Safurex® is also applied in non-Stamicarbon urea plants and several other processes.

In urea plants worldwide and over more than 25 years, Safurex® has proven to be the best choice of material. Using Safurex® reduces the total cost of ownership, improves the reliability and availability and makes urea plants less

vulnerable for upset conditions. The option of extended blocking-in of the synthesis section makes urea plant operations highly flexible. Finally, the use of Safurex® allows low levels of passivation air which further improves plant performance and safety.

THE PROBLEM

At Stamicarbon we are regularly confronted with (extended) diaphragms from almost all sections of the urea plant, that have suffered corrosion damaged.

Picture 1: Corrosion damage HAZ Tantalum

Picture 2: Corrosion damage SS 316L

Picture 3: Weld damage Zirconium

To mitigate such consequences operators have opted to use exotic (and expensive) materials such as zirconium and tantalum or have applied technically, sub-optimal, system designs.

Zirconium is well known to resist the carbamate corrosive atmosphere. However zirconium has specific design requirements related to operating temperature and comes at a high price.

Tantalum also has good carbamate corrosion properties, however is not suitable for extended diaphragms as the heat affected zone (HAZ), at the diaphragm to extension weld, will become subject to corrosion due to material changes.

These factors can lead to early failure if not mitigated or to sub optimal designs (e.g. Zirconium lined extension body)

Technical designs used to mitigate the consequences of a diaphragm failure (think of nozzles with pancake type diaphragm seals with block and bleed arrangement, draining and/or flushing provisions etc. instead of pads with extended diaphragms) will introduce other, practical problems (plugging) and safety issues.

OUR SOLUTION

Stamicarbon as an innovative company is continuously striving to improve its technology and the applicability of Safurex®. Based on the exceptional corrosion properties of the Safurex® material, full Safurex® (extended) thin foil diaphragms for pressure and differential pressure measurements in (carbamate) corrosive environments have been developed. For this an innovation consortium was formed with Alleima (former Sandvik Material Technology) and co-inventor of Safurex®, from Sweden and Badotherm, a leading manufacturer of high quality process instrumentation, from The Netherlands. These full Safurex® diaphragms come at a competitive price and are now available for the market.

DESIGN FEATURES

- All wetted parts in Safurex®: Safurex® diaphragm welded to a full Safurex® (extended) body.
- Safurex® diaphragm (100 µm/0.1 mm/0.004 inch) having a very fine grain structure (prerequisite for excellent corrosion resistance)
- Safurex® diaphragm with the ductility and flexibility necessary for the application as diaphragm seal in (differential) pressure applications
- Specialty welding to bond Safurex® diaphragm to full Safurex® extension body

- Welded construction extensively tested:
    - Streicher tested for welding and welder qualification
    - Autoclave tested under urea synthesis conditions without oxygen
- Metallurgically examined:
    - Corrosion of diaphragm and weld is extremely low.
    - Excellent microstructure in the foil, HAZ and weld metal
- (Extended) diaphragms design according Badotherm FSO-EXT and FSO-DF or to custom specification.
- Facilitates further digitalization (IIoT) and connectivity (e.g. Stami Digital process Monitor)

OBTAINABLE ADVANTAGES: COST SAVINGS AND DESIGN FREEDOM

- The application of the (extended) diaphragm seals in Safurex® provides a cost attractive alternative for pressure and differential pressure measurements in the HP synthesis, MP section(s) and LP section of any UREA plant. It now offers the possibility to have all wetted parts of the synthesis equipment and instrumentation to profit from the excellent corrosion resistant's to carbamate of Safurex®.
- Safurex® has a distinct price advantage over solutions in suitable alternative materials without typical drawbacks as high price and limited design freedom.
- Installation in the process can be simple, by direct mounting on the equipment or pipe line (preferably pad type nozzle or standard HP adapter) without any blocking, bleeding and/or flushing provisions.
- As an option the measurement can be executed as a wireless industrial sensor for monitoring purposes beyond the standard process monitoring and control, ready for further digitalization of your plant and IIoT.
- Suitable solution for other (corrosive) processes, such as melamine, nitric acid, etc. Discuss your case with Stamicarbon specialists.

THIN FOIL DEVELOPMENT

As manufacturer of Safurex® Alleima was the partner to lead the develop of the thin foil.

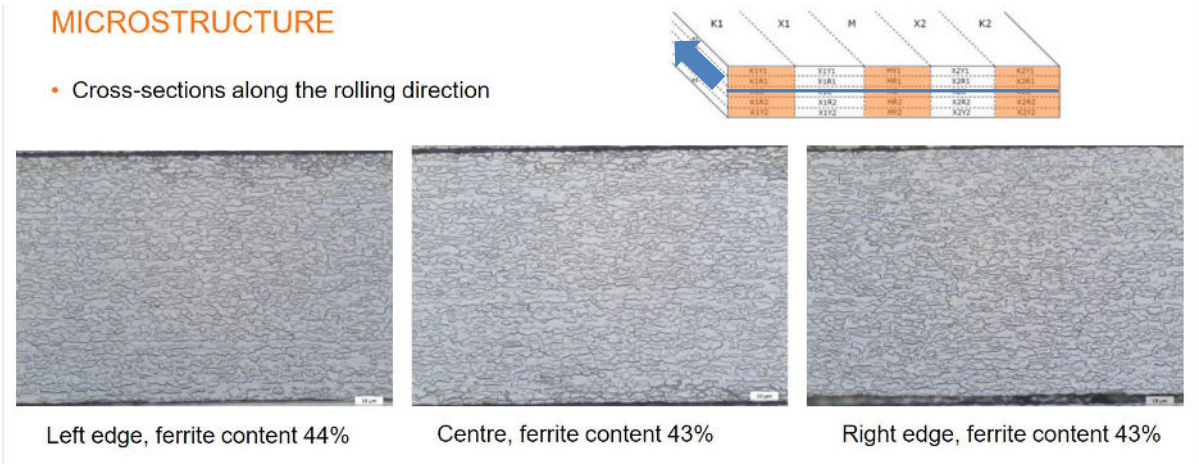
Based on their specific knowledge of the material, its properties and of possible processing steps they were able to develop a reproducible processing sequence to manufacture thin foil of the required specifications.

To achieve the correct formulation, the governing (and conflicting) parameters had to be balanced:

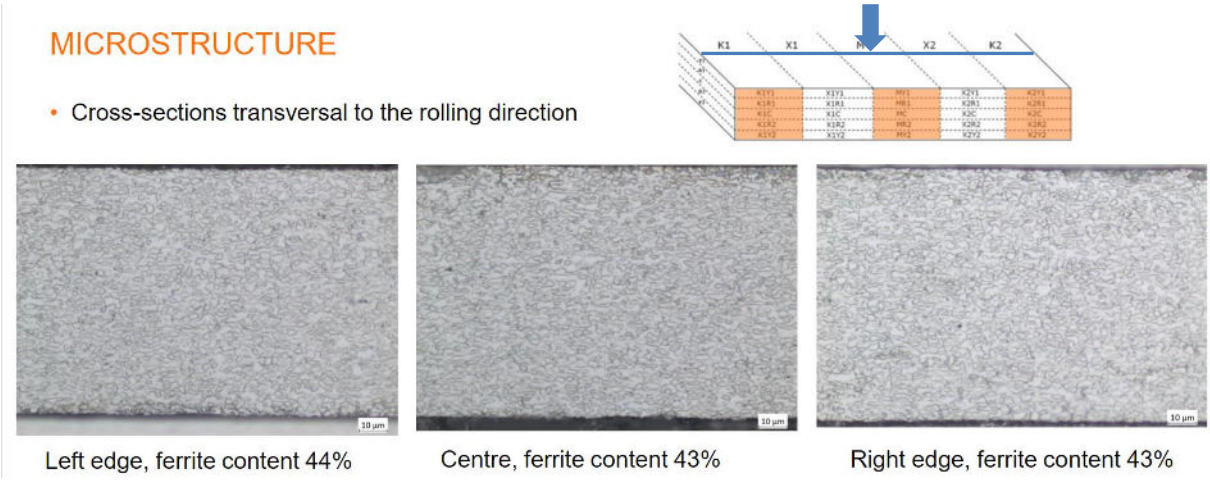
- For the manufacturing of the diaphragm the ductility and weldability of the foil.
- For the accurate operation of a diaphragm seal type measurement the flexibility of the diaphragm.
- For reliable operations the longevity and thus the corrosion resistance of the diaphragm seal.

The result:

Picture 4: The foil



Picture 5: Micro structure of strip along the rolling direction.



Picture 6: Micro structure of strip transversal to the rolling direction.

CUTTING AND SHAPING OF SAFUREX® DIAPHRAGMS

To provide the necessary knowledge to produce a diaphragm seal for the use in (differential) pressure measurements, Badotherm, a leading manufacturer of diaphragm seals, was involved.

They provided the knowledge and operations to transform a Safurex® flat thin foil into a cut wafer and into a convulved diaphragm seal.

The cut, convolved result:

Picture 7: Cut Safurex® wafer

Picture 8: Convoluted diaphragms

WELDING SAFUREX® DIAPHRAGM TO SAFUREX® BODY

From this point on Badotherm and Stamicarbon focus was on a welding procedure to fuse the thin Safurex® diaphragm to a full Safurex® body, machined from bar.
A suitable pWPS was developed and under the supervision of Lloyd's and Stamicarbon the pWPS, in a test welding session, was promoted to WPS.
After additional corrosion testing (Streicher) of the welds, also the WPQR was certified and Badotherm welders were certified by Lloyd's as well as by Stamicarbon.

Picture 9: Badotherm welder welding the Safurex® diaphragm

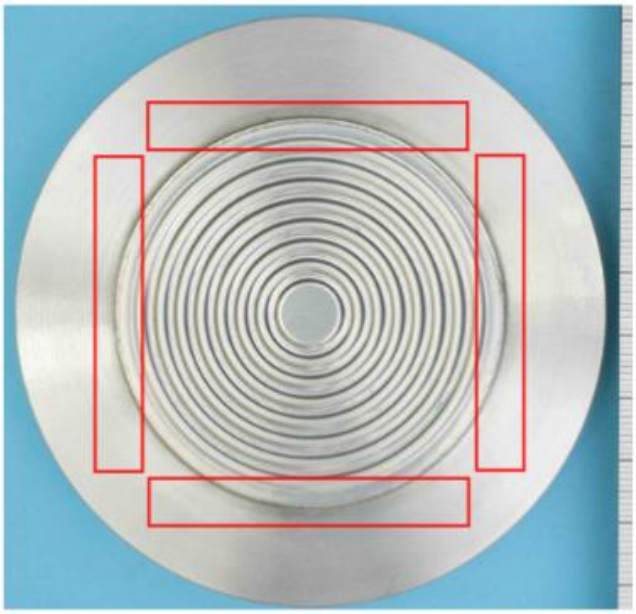
AUTOCLAVE CORROSION TESTING

Although the Streicher test is a good test method to evaluate general corrosion resistance of steels, it has been proven to be not selective/predictive enough to distinguish the corrosion resistant properties of steels under carbamate corrosive conditions. Therefore a more selective test was required, exposing the welded samples to severe HP urea synthesis carbamate environment. Such a test had to be done in a specialized autoclave unit.
Autoclave test set-up:
The equipment used for the evaluation of corrosion-resistance in ammonium carbamate was an existing dedicated designed autoclave, see Picture 10. The autoclave was rated at a maximum pressure of 300 bar (4350 psi) and a maximum temperature of 250°C (482 °F). It is equipped with an agitator to ensure that the liquid is constantly renewed at the specimen surface and to maintain a homogeneous environment for all samples. During the est the autoclave was monitored and controlled from a separate room, except during loading and evacuation which was always performed at ambient pressure and temperature.

Picture 10: Autoclave used for the HP experiments

Picture 11: Sample holder* with *Samples

The autoclave was loaded with the chemicals, calculated to simulate the concentration of ammonium carbamate that would be expected in a HP synthesis at extreme operating conditions of 170 bar (2466 psi), 205°C (401 °F) and without oxygen. In total 6 specimens (Cut as indicated in Picture 12) were exposed for 330 hours (~14 days) in the test batch. Two (2) Safurex®, two (2) zirconium 702 and two (2) AISI 310MoLN (25-22-2). See Picture 11.



Picture 12: Overview of a diaphragm showing the positions of the extracted corrosion test sample

CORROSION TEST RESULTS OF AUTOCLAVED SAMPLES

After the autoclave test all samples were cleaned and the loss in weight was determined.

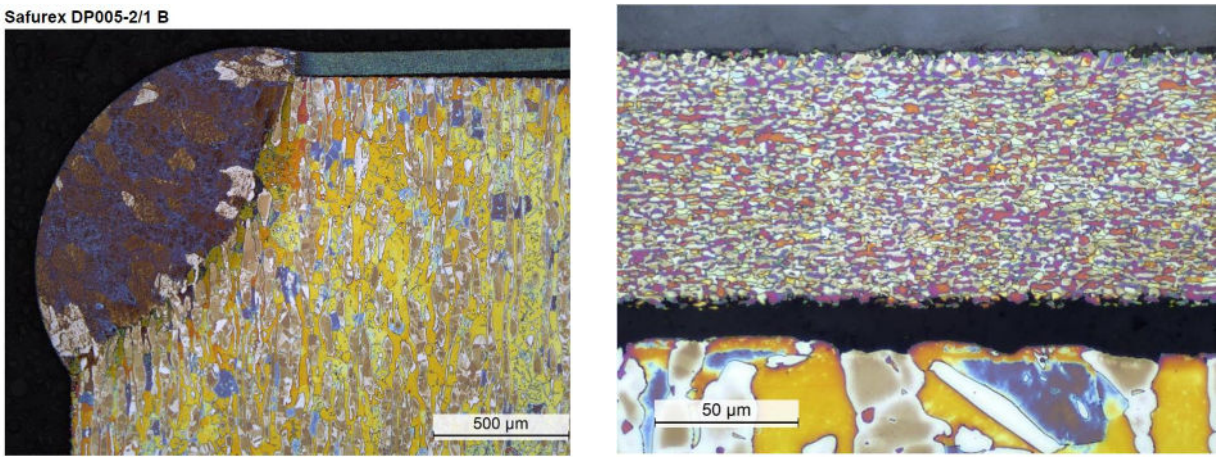
Table 1
Weight decrease (%) of all specimens

Sample ID	Surface area [cm^2]	Before exposure [g]	After exposure [g]	Difference [g]	Weight decrease [%]
Safurex DP005-2/1 B	8.786	5.9540	5.9093	0.0446	0.75
Safurex DP005-3/1 B	10.044	8.8040	8.7531	0.0509	0.58
AISI 310MoLN-C	10.158	8.5121	7.7901	0.7220	8.48
AISI 310MoLN-D	9.329	7.3637	6.6933	0.6705	9.1
Zirconium 702 C	9.575	5.8338	5.8337	0.0001	0.001
Zirconium 702 D	10.068	7.4823	7.4822	0.0001	0.001

From the beginning it was anticipated that zirconium could outperform Safurex® and, if only taking notice of the data in Table 1, it seems like it did.
However, from further microscopic examination it became clear that the (already very low) weight loss of the Safurex® samples originated predominantly from the Safurex® body part (disk), machined from Safurex® bar material and that the diaphragm showed only very very slight attack. Refer to Table 2 for a summary of the microscopic examinations.
The corrosion that was observed on the diaphragms was predominantly at the crosscut end, due to the cutting of the samples. Such crosscut does not exist when the diaphragm is applied as operational process seal.
Furthermore, in the autoclave test both sides of the diaphragm were exposed due to the cutting. The attack on an operational process seal will only occur on one side.
The very low corrosion of the diaphragm can be explained by the fact that the micro structure of the diaphragm shows a much finer grain than the disc, made of forged bar Safurex®. This is clearly illustrated in Picture 13 and 14.

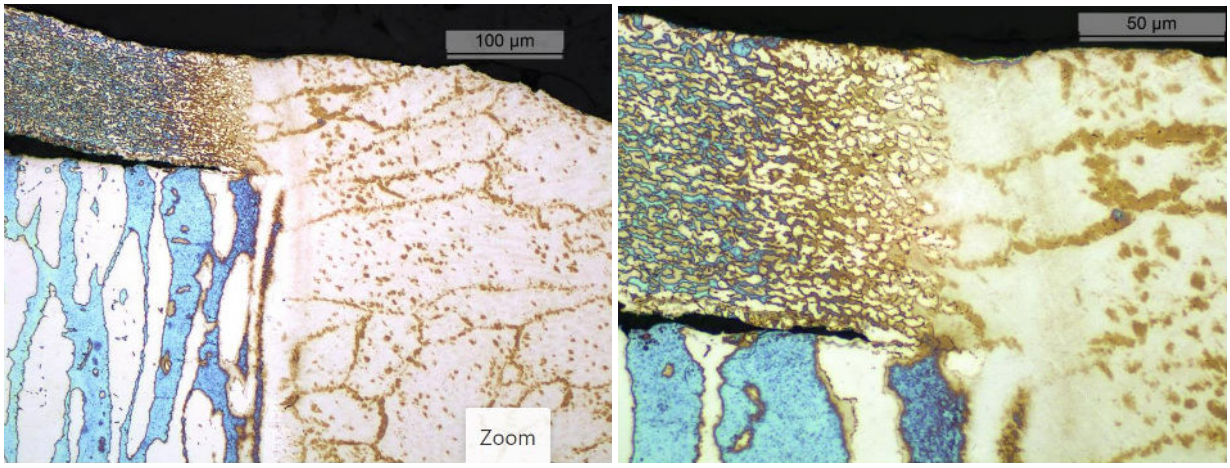
Table 2
Typical result of the microscopic examination of a Safurex® sample after autoclave exposure

Item		Microstructure	Depths [µm]	
			General	Max
HAZ	Disc	The selective attack occurred via austenite phase constituent and secondary austenite at the ferrite – ferrite grain boundaries.	15 – 20	52
	Diaphragm	Compared to the base material, there is almost no deviation and/or peculiarities. The selective attack occurred via austenite phase.	2 – 5	≤ 5
Weld		Relative coarse grained solidification microstructure of mostly ferrite and a network of acicular austenite at the grain boundaries. Locally an interdendritic solidification defect was present near the fusion line.	1 – 2	81
Stamicarbon specification A4-18005 RevAB accordance with material BE.06			In any direction ≤ 100 µm	



Picture 13/14: micro cross section of the weld joint and of the diaphragm. The disc shows a coarse banded ferritic matrix with island of austenite. The ferrite –austenite microstructure of the diaphragm is very fine grained.

The weld itself showed very little attack and proof of proper fusion as illustrated in Picture 15.



Picture 15/16: Details of the welt joint show the proper fusion and heat affected zone of the diaphragm (and disc) of the weld joint.

CONCLUSIONS

- Alleima, Badotherm and Stamicarbon have jointly developed thin foil Safurex® diaphragms for pressure and differential pressure measurements.
- These are a cost effective and improved alternative for (differential) pressure measurements in urea plants.
- Safurex® can now be used as **the** single material of choice in contact with the urea synthesis environment.
- Safurex® diaphragms can also be specified for other (MP and LP) sections of the urea and melamine plant and for other processes suffering from (carbamate) corrosion. Discuss your case with Stamicarbon specialists.
- Safurex® diaphragm type instruments are cost effective (Capex as well as Opex).
- Extended Safurex® diaphragm seals can be mounted directly into the process.
- Optionally the measurement can be executed as a wireless industrial sensor for monitoring purposes ready for further digitalization of your plant and IIoT.
- Safurex® diaphragm seals are exclusively available from Stamicarbon.

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Picture 17: Typical diaphragm type differential pressure transmitter arrangement (Badotherm)