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CAN PASSIVATION AIR IN UREA PLANTS BE MINIMIZED THROUGH THE USE OF SAFUREX® AND CONSEQUENTLY IMPROVE PLANT SAFETY ? (a)

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1. Preface

Stamicarbon has been established in 1947 and is the wholly owned licensing subsidiary of the Dutch chemicals producer DSM. Stamicarbon licenses proprietary processes, know-how and expertise, developed and commercially proven by licensees and its mother company.

The Chemical group DSM NV is a private corporation with its main offices in the Netherlands. DSM is globally active in several areas of the chemical process industry and has about 22,000 employees worldwide.

2. Summary

Urea plant safety in relation to flammable gas mixtures has been an important issue for the entire history of the urea process industry. The risk of flammability of gas mixtures is a result of the necessity to introduce passivation air in the process to avoid excessive corrosion to plant equipment. Minimizing the amount of introduced passivation air reduces the (already small) chance that the gas composition in the urea plant comes within the flammability ranges of such gas mixtures, thus making the plant safer.

The traditional construction materials in urea plants however did not allow minimized passivation air. A new construction material, suitable to operate with far lower oxygen passivation requirements, had to be found.
Sandvik and Stamicarbon developed this material together. The so developed material has been given the name Safurex®, which was registered.
Safurex® was extensively tested on laboratory scale and in plant experience on commercial scale has been obtained for several years already.

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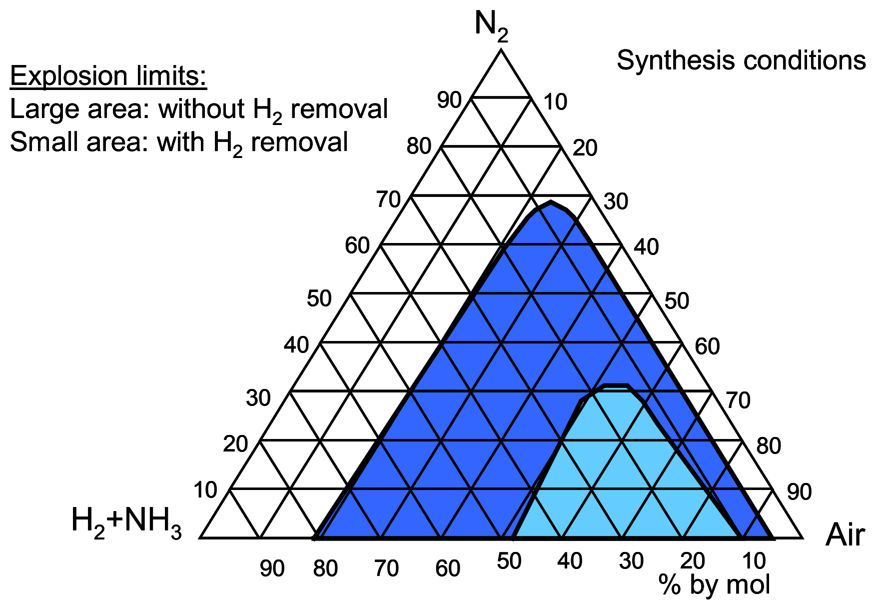
Safurex® does have far lower oxygen passivation requirements and is therefore the material of construction needed to improve plant safety. Moreover this material has other advantages in the field of investment, environment and maintenance as well.

3. Safety Aspects

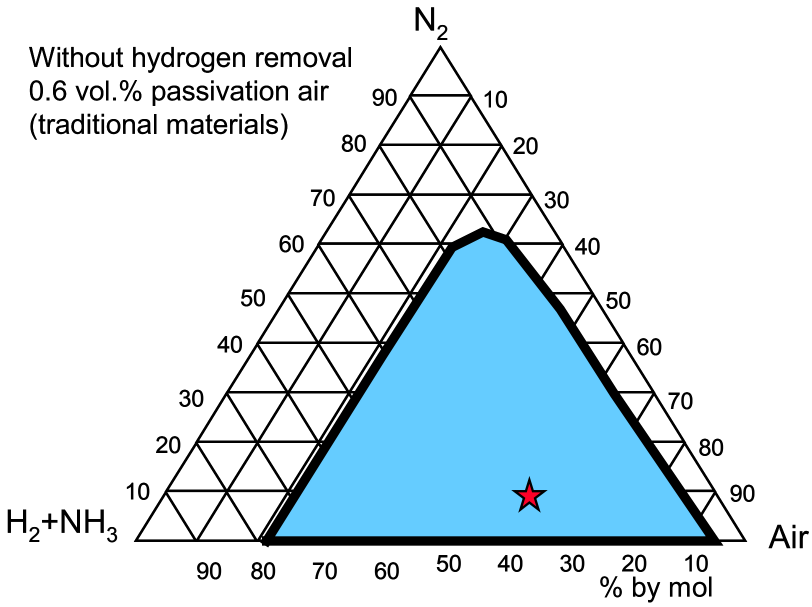
In the seventies of last century most urea plants were fed with carbon dioxide containing a considerable amount of hydrogen. A hydrogen content of close to 1 % was by no means an exception. This hydrogen in combination with the passivation air needed to protect the construction material from corrosion made flammable gas mixture in the plant inert vent a possibility. The removal of hydrogen from the carbon dioxide as introduced by Stamicarbon in the early nineteen eighties reduced the flammable area applicable for the inert gas vent, as can be seen in the composition diagram in Figure 1.
Introduction of hydrogen removal resulted in a safer plant as can be seen from Figure 2 and 3.

Figure 1.



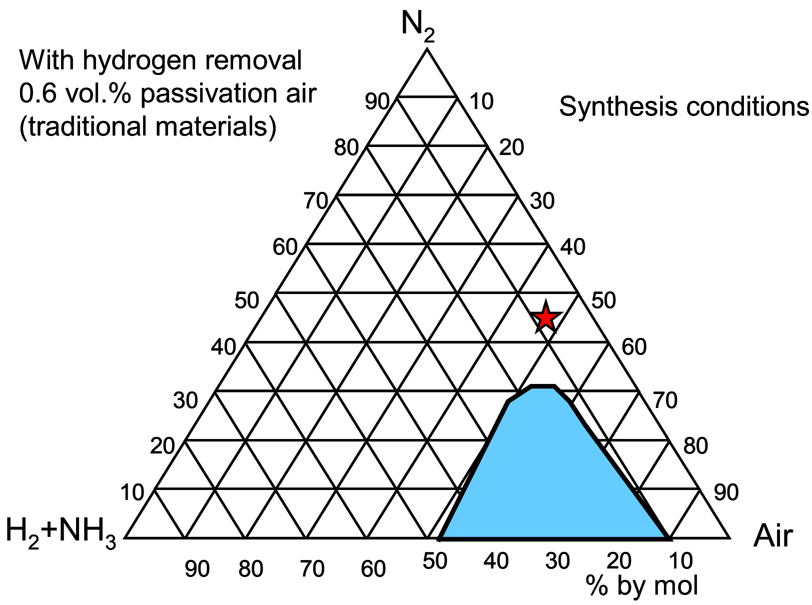
Without a hydrogen removal system the high pressure inert purge of a urea plant can be of a composition which is flammable. See Figure 2.

Figure 2.



By removing the hydrogen the flammable area became smaller and the inert purge gas composition changed to a more safe N_2 / air ratio # note

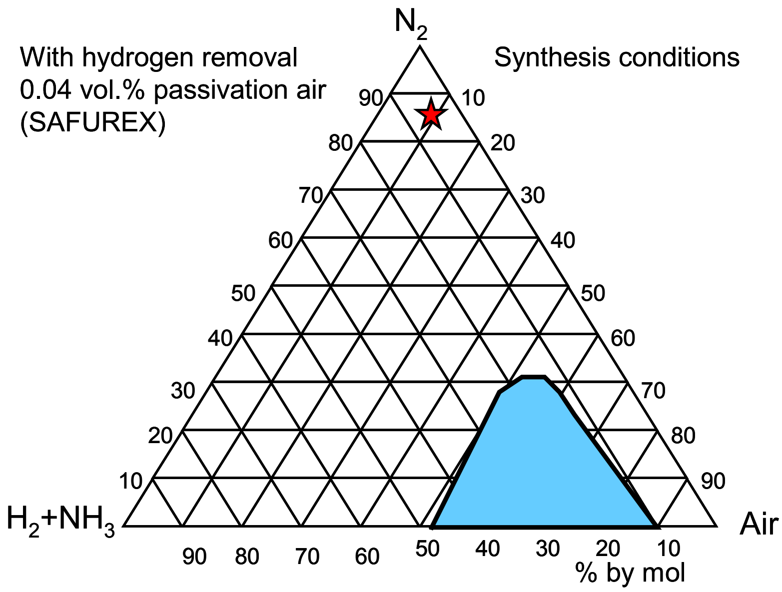
Figure 3.



The result of the hydrogen removal from the carbon dioxide is a plant operation whereby the actual gas composition of the inert purge is definitely outside the flammable range and the likelihood of an explosion is remote. Upset conditions or the unnoticed failure of the hydrogen removal reactor could however still cause a dangerous situation. Stamicarbon has ensured that even in such case safety precautions (e.g. scrubber sphere) are in place.

In the case that passivation air could be reduced from an oxygen content of 0.6 % in the carbon dioxide to a value of 0.04 %, the change in the gas composition of the inert purge of the plant would move the gas composition of the inert purge in the graph to a point which is really far away from the flammable limits. See Figure 4.

Figure 4.



Consequently a reduction of air for passivation purposes in the urea plant reduces the chance of mishaps in case of upset conditions considerably further.

Safurex® provides the possibility to reduce the oxygen content in the carbon dioxide to a value of 0.04 % without exposing the urea plant process equipment to more than normal corrosion.

note: In the presented composition diagrams the total of N_2, CO_2 and H_2O in the inert vent (inerts with respect to flammability) are presented as N_2.

4. Can the Passivation Air Be Minimized?

The materials in contact with the process fluid in a urea plant are exposed to corrosion because the process fluid is quite aggressive, especially at higher temperatures. In the synthesis section, specifically in the stripper, temperatures are at around 200 ^oC.
At this temperature the corrosion rate to the stainless steels normally in use in urea plants is very high. Protection from corrosion is found in the passive layer on the stainless steel, which is maintained through a continuous supply of oxygen via the process streams in contact with the stainless steel. This reduces the overall corrosion rate to acceptable values of some 0.05 to 0.1 mm per year. The installed equipment has sufficient corrosion allowance to secure life times of above 20 years.
The oxygen required to maintain the passive layer is fed to the process via air injection into the carbon dioxide stream. The oxygen content required in the CO_2 to maintain the passive layer on the normal urea quality stainless steel is 0.6 % (vol).

Stamicarbon and Sandvik have developed a new duplex stainless steel material, which does not require the high concentration of oxygen in the process streams to maintain a passive layer. Corrosion tests have shown that very low oxygen concentrations are sufficient to keep the oxygen layer intact. This material has been named Safurex®.
Using Safurex® as construction material in the urea synthesis allows the oxygen content in the CO_2 to be safely reduced to 0.04 % (vol) without running into corrosion problems.
Current testing indicates that the figure of 0.04 % (vol) O_2 is not even needed and may be further reduced in the future.
It should be noted that reduction of the O_2 concentration to 0.04 % (vol) can only be done in case all materials in contact with the process medium in the synthesis section are made out of Safurex®.

The passivation air can therefore be minimized. Consequently the chances that during upset conditions the inert vent gas composition enters into the flammable area, in Fig 4 of the previous chapter, are further reduced.

5. Results of Safurex®Testing

Safurex® is a high alloy ferrite-austenite stainless steel, developed to withstand the corrosive environment present in the urea process. This material belongs to the group of stainless steels that traditionally was divided in two different groups.

- Ferrite stainless steels, with chromium as the sole alloying element. It has the same microstructure as carbon steel. Chromium is the element responsible for the formation of the passive layer that gives the material corrosion resistance
- The addition of nickel produces an austenitic structure and allows welding.

The latest development has created a new major group:

- The ferrite-austenite (Duplex) stainless steels to which Safurex® belongs.

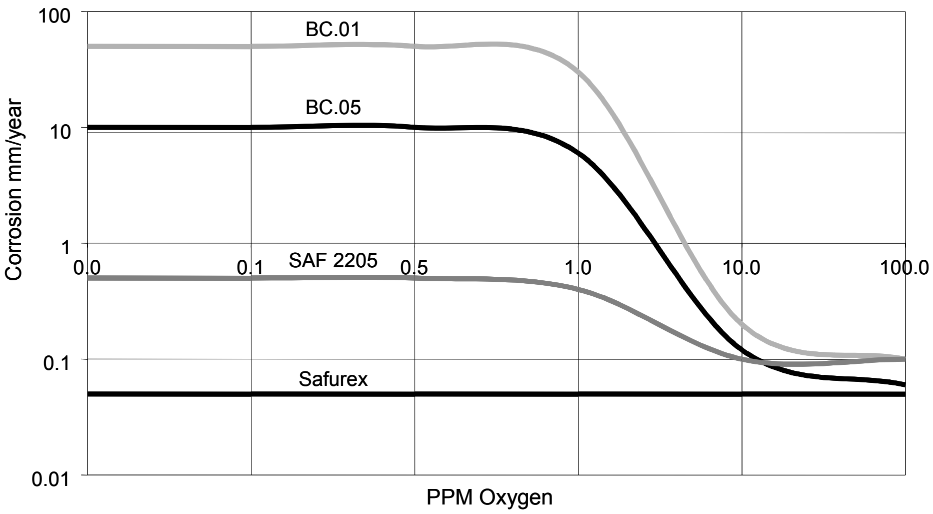
Duplex steels combine the good stress corrosion cracking properties of the ferrite stainless steels with the good ductility and weldability of the austenite stainless steels.

The duplex structure also results in high mechanical strength properties compared to the traditional austenitic stainless steels (AISI 316L and 2RE69), see page 3 of the attached Sandvik data sheet.

Many laboratory tests and exposure tests to the process fluid were performed since 1996, the year Safurex® was introduced. Tests were done on tube, bar, HP pipe, and plate.
Since no equipment can be built without welding, weld consumables were optimized and weld deposits were subjected to corrosion tests as well. Also samples of internal bore welding without the addition of filler wire were corrosion tested. Weld deposits after Post Weld Heat Treatment were also tested. All the aforementioned tests indicated that Safurex® is the ideal material for use in urea plants.

Testing of corrosion rates of Safurex® in comparison to other urea quality material under conditions with hardly any oxygen present resulted in the data given in Figure 5 here below.

Figure 5: Corrosion rates in oxygen free carbamate solutions



The corrosion performance of Safurex® as shown in the afore going figure brought the realization that reduced air introduction would be a serious option for plants constructed in Safurex®

Further elaborate testing of Safurex® under conditions with reduced oxygen was done.
The results of these tests have made it clear that an O_2 content of 0.04 % (vol) in the CO_2 feed to the plant is more than enough to operate a plant constructed out of Safurex® safely.
Having determined the lower O_2 concentration, the resulting material balances around the inert gas vent of the urea plant showed that the composition of these gasses had shifted far away from the flammable area as indicated in Figure 4 of this paper.

6. Practical Equipment Fabrication Experience with Safurex®

It is commercially proven that HP Equipment can be fabricated in Safurex® using conventional techniques

The following equipment has been contracted.
- HP Carbamate Condenser, 1850 tubes, 1998
- HP Carbamate Condenser, 1300 tubes, 1998
- Liquid distribution system stripper, 1990
- Stripper, 3100 tubes, 1999
- HP Carbamate Condenser, 1000 tubes, 1999
- High efficiency reactor trays, 2000
- Stripper, 2400 tubes, 2001
- HP Carbamate Condenser, 2150 tubes, 2001
- Syphon Jet reactor trays, 2001
- HP Carbamate Condenser, 1850 tubes, 2001
- HP Carbamate Condenser, 1300 tubes, 2001
- Syphon Jet reactor trays, 2002
- HP Carbamate Condenser, 1865 tubes, 2002

Fabrication experience shows that:
- Welding Safurex® is less critical than welding other materials used in urea plants
- Risk for hot cracking is minor
- Fabrication as per the most common design codes can be done, also in relation with post weld heat treatment.

7. Other Advantages of Safurex® and the Reduced Passivation Air Injection

7.1 Investment

Safurex® has a significantly higher mechanical strength than the austenitic stainless steels traditionally used in urea plants. This property allows designers to reduce the thickness of liner plates, tube walls in heat exchangers and HP pipe wall thickness while maintaining even better life time expectations of the equipment.
The overall investment costs of Safurex® equipment, as a consequence of this increased material strength, are lower than the costs of traditional equipment (about 5 to 10%).

Another advantage is that the reduction in tube wall thickness results in a larger inner diameter of the heat exchanger tube, which in turn has an impact on the dimensions and finally the weight of the equipment, or opens the possibility to increase capacity with same outer dimensions of the heat exchanger, which is an interesting option in case of replacement.

Due to the high mechanical properties, different schedules for HP piping can be used. This has a large effect on the wall thickness leading to longer pipe length and because of that fewer welds needed in the HP piping. Moreover fabrication of very large pipe diameters becomes feasible.

Because a reduced passivation air injection causes an overall lower inert concentration in the urea synthesis section the urea reactor and/or the HP scrubber will benefit from it and become smaller in size, thus reducing the investment.

7.2 Environment

Urea plants built with Safurex® as construction material for the synthesis section will require only 0.04 % (vol) of O_2 in the CO_2 feed. The consequence of the reduction of the airflow, providing the oxygen, to the CO_2 compressor is that the amount of inerts to be vented from the synthesis section will decrease considerably as well. In case a lower amount of inerts from the plant will be vented also a proportionally lower amount of ammonia will leave the synthesis section.

7.3 Stress Corrosion Cracking

Boiler feed water contaminated with very small amounts of chlorides can cause serious damage to austenitic stainless steel tubes in the shell side of HP carbamate condensers, especially in the tube sheet area. The annular space between tube and tube sheet is a stagnant area and refreshment of BFW does not take place. Accumulation of chlorides may be the result.
The consequence of accumulated chlorides is very likely to be a case of stress corrosion cracking in the austenitic stainless steel tubes. Safurex® is hardly sensible to stress corrosion cracking and equipment fabricated out of Safurex® will not suffer.

8. Availability of Safurex®

To be able to reduce the O_2 concentration in the CO_2 to 0.04 % (vol) the complete plant must be constructed completely in Safurex®. Therefore, besides plates, bar, tubes, pipe, weld consumables and the like, also check valves, safety valves, block valves and control valves in contact with the process medium must be available in Safurex® too.

Stamicarbon and Sandvik have been discussing the availability topics with a number of suppliers of these parts, resulting in proper and competitive availability of these materials.

As Safurex® has been developed by Sandvik, together with Stamicarbon the material is principally available for use in projects using the Stamicarbon urea technology.

9. Conclusions

Safurex® makes it possible to reduce the passivation air stream to a urea plant. The more than tenfold reduction of the O_2 concentration in the carbon dioxide feed to a urea plant, which is the result thereof, shifts the Nitrogen/Air ratio in the inert vent gasses of the synthesis section in such a way that even in the case of upset operating conditions the chances of flammable conditions become virtually non existing. The safe operation of urea plants benefits from the use of Safurex®

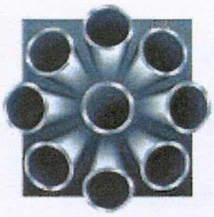
The use of Safurex® as construction material in urea plants is becoming more and more common. Fabrication of urea synthesis equipment is experienced as easy. The corrosion resistance of the material is better than the corrosion resistance of the traditional urea quality stainless steels. The resistance of Safurex® against stress corrosion cracking due to chlorides in tubes of HP carbamate condensers is far better than experienced with austenitic materials, which are sensitive there for. The investment in Safurex® equipment is below the investment in equipment using traditional materials. All equipment and parts needed for a complete grass roots urea plant in Safurex® are commercially available.

Investment in HP equipment and/or a complete plant in Safurex® is lower compared to using traditional austenitic stainless steels.

It is safer, better and cheaper.

Sources:
1) Explosion limits, by Jo Meessen, Stamicarbon, The Netherlands
2) Safurex® in urea plants, by Jo Eijkenboom, Bart Gevers, Stamicarbon, The Netherlands

Attachement



SANDVIK Steel

Seamless tube

Sandvik SAFUREX™

S-1847-ENG March 1999 • Cancels all previous editions

Sandvik SAFUREX™ is a high alloy duplex (austenitic-ferritic) stainless steel developed to withstand the corrosive environments present in the Stamicarbon urea process. Characteristic features are:

- *good corrosion resistance to carbamate solutions both with oxygen and with little or no oxygen*
- *excellent resistance to intergranular corrosion*
- *excellent resistance to pitting and crevice corrosion*
- *high resistance to stress corrosion cracking*
- *good weldability*

STANDARDS

Type of steel
UNS S32906

Product standards
ASTM A789, A790, A479

Approvals
ASME Code Case 2295.
Stamicarbon specification 18005S-BE 06/MS50

FORMS OF SUPPLY

Seamless tube – Finishes and dimensions
SAFUREX™ can be supplied as straight or U-bent tubing. The size range is up to OD 273 mm. Bigger sizes on request.

The tubes are supplied in the solution annealed and white-pickled condition or in the bright-annealed condition.

Other forms of supply
- Pipe
- Fittings
- Flanges
- Plate
- Round bar
- Filler metals for welding

MECHANICAL PROPERTIES

The mechanical properties are determined for heat exchanger tubes. If SAFUREX is exposed for prolonged periods to temperature ranges exceeding 280°C (540°F), the microstructure changes, which results in a reduction in toughness. This does not necessarily affect the behaviour of the material at the operating temperature. The listed values are guaranteed for tube and pipe.

At 20°C (68°F)

Wall thickness	Proof strength $R_{p0.2}$ [a]		Tensile strength R_m		Elong. A [b]
	MPa	ksi	MPa	ksi	%
mm	min.	min.	min.	min.	min.
<10	650	94.3	800	116	25
≥10	550	80	750	109	25

1 MPa = 1 N/mm²
a) $R_{p0.2}$ corresponds to 0.2% offset yield strength.
b) Based on $L_0 = 5.65 S_0$, where L_0 is the original gauge length and S_0 the original cross-section area.

At high temperatures

Metric units

Temperature °C	Wall thickness mm	Proof strength $R_{p0.2}$ MPa min.	Tensile strength R_m MPa min.	Elong. A % min.
100	<10	550	750	25
	≥10	500	730	25
200	<10	470	720	25
	≥10	430	700	25
300	<10	450	710	25
	≥10	410	690	25

SAFUREX™ is a trademark owned by Sandvik AB

Imperial units

Temperature °F	Wall thickness inch	Proof strength $R_{p0.2}$ ksi min.	Tensile strength R_m ksi min.	Elong. A % min.
200	<0.4	81.0	109	25
	≥0.4	74.4	107	25
400	<0.4	68.0	104	25
	≥0.4	62.2	101	25
600	<0.4	64.8	103	25
	≥0.4	59.0	100	25

According to ASME Code Case 2295, the following design values are recommended for SAFUREX (UNS S32906)

Temperature		Stress			
		Tube wall thickness <10 mm		Tube wall thickness ≥10 mm	
°F	°C	ksi	MPa	ksi	MPa
100	38	33.1	228	31.1	214
200	93	33.1	228	31.1	214
300	149	31.5	217	29.6	204
400	204	30.6	210	28.7	197
500	260	30.1	207	28.3	195
600	316	30.1	207	28.3	195

Impact strength

SAFUREX possesses a good impact strength. The ductility to brittle transition temperature is approximately -100°C (210°F). Figure 1 shows the typical impact energy for SAFUREX.

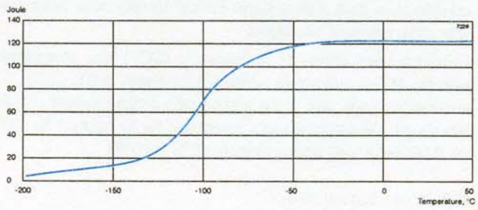


Figure 1. Typical impact energy curve for SAFUREX using half size

PHYSICAL PROPERTIES

Density, 7.7 g/cm³, 0.28 lb/in³

Thermal conductivity

Temperature °C	W/m °C	Temperature °F	Btu/ft h °F
20	13	68	7
100	14	200	8
200	16	400	9
300	18	600	10
400	19	800	11

Specific heat capacity

Temperature °C	J/kg °C	Temperature °F	Btu/lb °F
20	470	68	0.11
100	500	200	0.12
200	530	400	0.13
300	560	600	0.14
400	600	800	0.14

Thermal expansion, mean values in temperature ranges ($x10^{-6}$)

SAFUREX has a coefficient of thermal expansion close to that of carbon steel. This gives SAFUREX definite design advantages over austenitic stainless steels. The values given in the table are average values in the temperature ranges.

Metric units, (10^{-6}/°C)

Temperature, °C	30-100	30-200	30-300	30-400
SAFUREX	11.5	12.0	12.5	12.5
Carbon steel	12.5	13.0	13.5	14.0
AISI 316L	16.5	17.0	17.5	18.0

Imperial units, ($x10^{-6}$/°F)

Temperature, °F	86-200	86-400	86-600	86-800
SAFUREX	6.5	7.0	7.0	7.0
Carbon steel	7.0	7.0	7.5	8.0
AISI 316L	9.0	9.5	10.0	10.0

Modulus of elasticity, ($x10^3$)

Temperature °C	MPa	Temperature °F	ksi
20	200	68	29.0
100	194	200	28.2
200	186	400	26.9
300	180	600	26.0

CORROSION RESISTANCE

Urea carbamate found in the HP urea process is very aggressive and corrosion problems in form of intergranular or/and general corrosion are found when an inferior material is applied.

Variations in corrosion properties have been found especially on 316L Urea Grade type of material and the Huey test has been included as material test for Sandvik 3R60 U.G. and Sandvik 2RE69 to verify a low general corrosion rate and a low intergranular attack. Past experience has showed that a high corrosion rate in service corresponds to a high corrosion rate in the Huey test.

Under normal service conditions in the Stamicarbon urea process the chloride concentration in the boiler water is extremely low and no chloride induced corrosion will appear. If the chloride concentration is unintentionally increased, stress corrosion cracking can occur on austenitic stainless steels like 316L U.G. and 2RE69. (The risk for SCC increases with increasing chloride concentration, stresses and temperature.)

At the prevailing conditions even small concentrations like 10-50 ppm may be sufficient to cause SCC. Austenitic-ferritic (duplex) grades have in general a much improved resistance (see figure 2). Pitting and crevice corrosion are formed at much higher chloride concentrations, which are unlikely to appear in service. SAFUREX has superior pitting and crevice corrosion resistance to both 3R60 U.G. and 2RE69.

Stress corrosion cracking

SAFUREX has excellent resistance to chloride induced stress corrosion cracking. The resistance of various alloys to stress corrosion cracking determined by constant load testing in aerated 40% $CaCl_2$, pH 1.5, at 100°C (210°F), (modified ASTM G36 method) is shown in Figure 2.

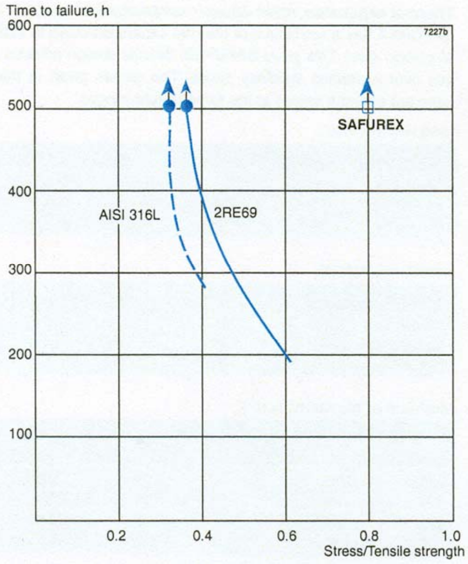


Figure 2. Constant load testing of SAFUREX and some other alloys in 40% $CaCl_2$ pH 1.5, at 100°C. Time to failure vs applied stress in percentage of tensile strength is shown.

Intergranular corrosion

SAFUREX has, owing to its high content of important alloying elements, a very high resistance to intergranular corrosion. The resistance to intergranular corrosion is often studied by means of either Huey testing (ASTM A262 practice C, 5x48h in boiling HNO_3) or Streicher testing (ASTM A262 practice B, 120h in boiling H_2SO_4 + $FeSO_4$). For SAFUREX, the Streicher test is a more relevant test method. In the table below, some results from this test are presented. Regarding Huey testing, SAFUREX meets the same requirements as 2RE69 according to specifications from Stamicarbon.

Streicher test results

Typical mm/year	Maximum mm/year	Selective attack µm
0.18	0.78	<70

Pitting and crevice corrosion

SAFUREX has a carefully balanced composition with a high content of chromium and nitrogen and a moderate amount of molybdenum. This enables a very high resistance to localised corrosion, such as pitting and crevice corrosion. An index for comparision of the theoretical pitting and crevice corrosion is the PRE number (Pitting Resistance Equivalent).

The PRE is defined as, in weight-%

PRE = %Cr + 3.3 x %Mo + 16 x %N

One of the most severe pitting and crevice corrosion tests applied to stainlesss steel is ASTM G48, i.e. immersion in 6% $FeCl_3$. When pits are detected following a 24 hours exposure, together with a substaintial weight loss (>5 mg), the test is interrupted. Otherwise the temperature is increased 5°C (9°F) and the test is continued with the same sample. In the table below, SAFUREX is compared with some other urea grades with regard to PRE and the critical pitting temperature (CPT) determined by ASTM G48A.

PRE-values and critical pitting temperature acc. to ASTM G48A for SAFUREX and some other urea grades

Alloy	PRE	CPT, °C	CPT, °F
SAFUREX	43	75	167
2RE69	34	45	113
3R60 U.G.	26	<10	<50

Potentiostatic tests in solutions with different chloride contents are reported in Figure 3. The applied high potential, 600 mV SCE, corresponds to very harsh conditions, thus resulting in conservative data with a lower critical temperature compared with most practical situations.

Critical crevice corrosion temperature (CCT) was determined with the MTI-2 procedure which is immersion in 6% $FeCl_3$, 12 crevices on each side of the test coupon and a moment of 0.28 Nm. Duplicate samples were immersed for 24 hours. The CCT for SAFUREX was approximately 40°C (100°F).

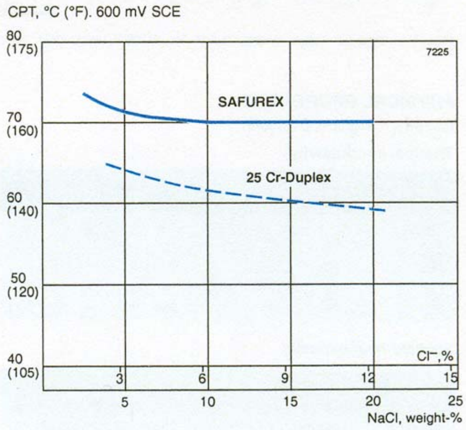


Figure 3. Critical pitting temperature (CPT) at varying concentrations of sodium chloride, from 3 to 25% (potentiostatic determination at +600 mV SCE with surface ground to 600 grit paper).

WELDING

SAFUREX has good weldability. Suitable welding methods are manual metal arc welding (MMA) and TIG welding, which is best performed with an addition of 1-2% nitrogen to the shielding gas. The electroslag welding process (ESW) can be used for strip overlay welding of e.g. tube sheets.

Welding should be conducted within the heat input range of 0.2-1.5 kJ/mm, and an interpass temperature of maximum 150°C.

Matching filler metals are recommended in order to obtain a weld metal with optimum corrosion resistance and mechanical properties. At tube to tube sheet welding the TIG process should be used with a shielding gas of Ar + 2% N_2 in order to get the correct weld metal structure. The covered electrodes and the filler metal for TIG welding have both designation SAFUREX. For more detailed information concerning welding, contact Sandvik for advice.

FABRICATION

Bending

The force needed for bending SAFUREX is higher than that for standard austenitic stainless steels which is a natural consequence of the higher proof strength.

Expanding

Compared with austenitic stainless steels, SAFUREX has a higher proof and tensile strength. This must be kept in mind when expanding tubes into tubesheets. Normal methods can be used, but the expansion requires higher initial force and should be undertaken in a one step operation. As a general rule, tube to tubesheet joints should be welded to ensure a leak free joint.

Machining

Being a dual phase materal (austenitic-ferritic) SAFUREX will present a different wear picture from that of a single phase material like 2RE69. The cutting speed must therefore be lower than that recommended for austenitic grades. Further information is available on request

APPLICATIONS

SAFUREX is suitable for the following applications in urea plants:

- Stripper tubes
- Carbamate condenser tubes
- Ferrules in strippers
- High pressure piping
- Scrubbers
- Lining
- Overlay welding
- Reactor trays

Recommendations are for guidance only, and the suitability of a material for a specific application can be confirmed only when we know the actual service conditions. Continuous development may necessitate changes in technical data without notice.

SANDVIK Steel

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