KorroPad testing - applications from industry and research

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Abstract. The pitting corrosion resistance of stainless steels can be assessed with the KorroPad test in a very simple, quick and inexpensive way. The KorroPad is therefore an interesting alternative to time-consuming long-term tests and complex electrochemical investigations for manufacturers, processors and users of stainless steels. In this article, the basic functionality of the KorroPad is described and selected examples from industry and research illustrate its application. The assessment of grinding processes and post-weld treatment are first presented as relevant examples regarding industrial surface treatments on the pitting corrosion resistance. Subsequently, current research to the modification of the KorroPad test is presented, which shows the specific detection limit of KorroPads on binary Fe-Cr reference alloys. Finally, modified KorroPads are used to detect microstructure related susceptibility to pitting corrosion induced by sensitizing heat treatment. The KorroPad enables the creation of a sensitization T-t diagram based on the pitting corrosion resistance.

1 Introduction

The so-called "KorroPad" was developed in the research project "Detection of corrosion-sensitive surfaces of stainless steels by processors" [1,2]. The KorroPad consists of a gel-like electrolyte, which indicates corrosion-prone surface conditions on stainless steels by means of a visible color reaction. The principle of the KorroPad test is presented schematically in Figure 1.

The method was originally developed to detect an incompletely formed or instable passive layer on the austenitic stainless steels X5CrNi18-10 (1.4301). The KorroPad is a water-based agar gel, which contains 3 wt.-% agar, 1 mM potassium hexacyanoferrate III (K₃[Fe(CN)₆]) and 0.1 M sodium chloride.
(NaCl). The agar forms a gel matrix, which holds and immobilizes the electrolyte, while the $K_3[Fe(CN)_6]$ causes the KorroPad to achieve a redox potential of up to $+320 \text{ mV}_\text{Ag/AgCl}$ (measured on platinum) and is thus able to polarize the surface of a stainless steel up to $+200 \text{ mV}_\text{Ag/AgCl}$. The redox potential can be shifted by increasing or decreasing the $K_3[Fe(CN)_6]$ content [1]. In addition to the anodic polarization, the NaCl dissolved in the KorroPad also ensures that pitting corrosion is initiated and eventually stabilized.

Due to this composition, the KorroPad has a defined corrosion load, which can trigger localized metal dissolution (mostly through pitting corrosion) at weak points and defects at the surface. If the critical pitting corrosion potential is reached or exceeded by the anodic polarization iron ions pass into the KorroPad at the phase boundary and react with a blue colour change with $K_3[Fe(CN)_6]$ to Prussian Blue ($Fe_4[Fe(CN)_6]_3$). This insoluble pigment remains within the gel matrix of the KorroPad and the corrosion resistance can be estimated due to the permanent blue discoloration.

Three typical appearances can be observed in the KorroPad test, which are shown as examples in Figure 2. This classification enables a visual assessment of the susceptibility to corrosion.

![Figure 2. Typical appearances in the KorroPad test:](image)

- a) no pitting corrosion (stable passive layer) $\approx$ no indication;
- b) metastable pitting corrosion (with repassivation) $\approx$ many small indications;
- c) stable pitting corrosion (without repassivation) $\approx$ one large indication.

An assessment of the susceptibility to pitting corrosion of the stainless steels is also possible based on the percentage area of the blue indications [1, 3]. The susceptibility to pitting corrosion increases with the proportion of the coloured area. Values of more than 1% reflect an increased susceptibility to pitting corrosion in atmospheric conditions [3]. However, the image processing required for this is significantly more complex than a visual assessment according to Figure 2.

When assessing the formation of a passive layer, which is significantly influenced by the surface treatment, it is useful to consider the proportion of the area in order to investigate the kinetics of the formation of the passive layer [4] or to compare the quality of the passive layer regarding different surface conditions. However, it should be noted that the number of metastable pitting indications is reduced by passivation (e.g. at high relative humidity) reducing especially the smaller metastable indications and the overall area fraction of indications [4]. This must be considered when comparing different surface conditions by determining the area fraction. The sensitivity of the KorroPad method regarding the characterization of the pitting corrosion resistance is comparable to electrochemical methods and correlate with standard tests such as the alternating immersion tests [5], the salt spray tests [1, 3] and long-term exposure tests in natural atmospheres [1]. Research results on austenitic stainless steels [3, 4, 6-8] and martensitic stainless steels [5, 9-14] were recently published. The KorroPad was able to characterize reduced pitting corrosion resistance caused by increased sulphur content [8] and unfavourable surface treatments [3, 7, 15].

The following four chapter present selected application examples from industry and research, in which the KorroPad test was successfully applied. The first two examples deal with the possibility of assessing the quality of industrially manufactured surfaces and the detection of corrosion-prone surface conditions. The two final chapters show how the KorroPad test can also be used to verify material and structural causes of pitting corrosion induced by sensitizing heat treatment.
2 Reduced corrosion resistance and staining of industrially ground stainless-steel surfaces

Impairment of the visual appearance and staining of industrially ground stainless steel surfaces in non-critical atmospheric exposure conditions has led to complaints in the manufacturing industry. In many of these complaints, abrasive belts with aluminium oxide ($\text{Al}_2\text{O}_3$) were used for the final surface treatment. This is the reason why it was suspected that $\text{Al}_2\text{O}_3$ abrasive belts are responsible for the problem of staining [1]. Systematic studies on the influence of the used abrasive belt on the corrosion resistance of the austenitic stainless steel X5CrNi18-10 (1.4301) were carried out in order to investigate this assumption [16, 17]. The abrasive belt (single-layer grain / granulate grain) and the grain type ($\text{Al}_2\text{O}_3$ / SiC) were varied and the grinding parameters like cutting speed 24 m/s, counterrotation and mineral oil-free cooling lubricant are kept constant. The aim was to create critical surface conditions, detect them with the KorroPad and identify the cause of staining. Figure 3 shows the applied abrasive belts (240 grit) in the unused condition, the ground surfaces and the associated results of the KorroPad test on these surfaces.

The surfaces ground with granulate abrasive belts show more and larger KorroPad indications than the surfaces ground with single-layer grain. This effect can be demonstrated for products of several manufacturers and no significant differences were observed with regard to the surface quality (roughness and visual appearance). In addition, surfaces ground with SiC abrasive belts show less and smaller indications in direct comparison to surfaces ground with $\text{Al}_2\text{O}_3$ abrasive belts.

A major advantage of the KorroPad test is the localization of critical defects at the surface, which can be detected by the position of the large blue indications. This was used to determine the cause of the
higher susceptibility of the surfaces ground with granulate to staining. Figure 4 shows SEM images (SE contrast) of the ground surface areas where the largest KorroPad indications emerged.

![SEM images of the ground surfaces](image)

**Figure 4.** SEM images of the ground surfaces, where large KorroPad indications emerged

a) granulate abrasive belt with corundum grain [17] b) granulate abrasive belt with SiC grain.

Large particles (200 µm to 600 µm) of the ground steel can be seen at these surfaces, which are located on the top of the surface and are firmly attached to it. These particles create a large number of geometric crevices and contain microparticles of the respective abrasive grain (Al₂O₃ or SiC). The formation of these particles can be traced back to the pronounced topography of the granulate abrasive belt, which favours the remains of chips in the abrasive belt, while this is not the case for single-layer grain. The particles are merged to the surface in the grinding process and remain on the ground surface. The abrasive chip particles represent local defects, which impairs the formation of the passive layer. In the case of atmospheric exposure, this leads to the local release of iron ions and cause the phenomenon of brownish staining, since iron ions are converted to rust in subsequent reactions. Clarifying the cause of the increased susceptibility to corrosion was significantly facilitated by the KorroPad test, which is due to the good differentiability between different surfaces and the localization of critical defects.

### 3 Influence of weld post-treatment on corrosion resistance

There has been a significant increase in the use of duplex stainless steels in construction [18], since low Ni and Mo steels (1.4062, 1.4162 and 1.4362) reduce material costs compared to austenitic stainless grades for different technical applications [19, 20]. At the same time, duplex steels possess higher yield and ultimate tensile strength and an equivalent or better corrosion resistance compared to austenitic stainless steels 1.4301 and 1.4404 [19, 21]. The disadvantage is that production, processing and especially welding of duplex stainless steels is more demanding and requires adaptations.

The corrosion resistance of welds is influenced by the base material, the filler metal, the welding process and the weld seam surface finish [22, 23]. The tempering colours from welding significantly reduce the corrosion resistance of welded joints and must therefore be removed by suitable surface treatment [19, 21, 23]. This requires a process-related optimization of the production considering the corrosion resistance [19, 24, 25].

The aim of these investigations was to determine the influence of different surface treatments on the corrosion resistance of duplex steel weldments using the KorroPad test. One main question was whether blasting with glass-bead is equivalent to blasting with corundum grain regarding the weldment’s corrosion resistance. Weldments of 1.4062 with pickled, blasted, electropolished and plasma-polished surfaces were provided by Wilhelm Modersohn GmbH & Co.KG for different characterizations. Figure 5 shows welds in different surface states.
Figure 5. Weldments with different surface states: a) without treatment, b) pickled, c) blasted and d) electropolished [15].

The KorroPad test was used to check whether a stable passive layer was formed after passivation for 24 h at 95% relative air humidity on the different surfaces. The KorroPads were applied to areas directly at the weld seam (SN) and with 60 mm distance to the weld seam to additionally detect the sole effect of surface treatment. Figure 6 shows the results of the KorroPad test for the various conditions.

![KorroPad Test Results](image)

Figure 6. Results of the KorroPad test after various surface treatments at the welding seam (WS, top line) and at 60 mm distance to the welding seam (bottom line).

If the tempering colours are not removed after welding (untreated, directly at the welding seam), large areas with blue indications arise at the welding seam (WS) on the right side of the KorroPad. This indicates that iron ions are released at a high rate and a high susceptibility to pitting corrosion or a loss of passivity can occur. The untreated sheet material (60 mm distance to the welding seam) shows no KorroPad indications and therefore the high pitting corrosion resistance of the used duplex steel 1.4062. Surfaces blasted with Al₂O₃ (corundum) show a very high number of small (metastable) indications within the KorroPad at the welding seam and also with 60 mm distance. The indications are larger at the weld seam, which indicates a more critical condition.

Glass-bead blasting shows a significantly lower susceptibility to pitting corrosion, since the KorroPad has only isolated small indications. Polished and pickled surfaces are comparable to the untreated sheet and do not show any indications during the KorroPad test. The supplementary determination of critical pitting potentials resulted in significantly lower values for blasted surfaces compared to pickled or polished surfaces, which is presented in Figure 7 [15]. The blasted surfaces were examined by SEM in order to determine the cause of the lower pitting corrosion resistance, see Figure 8.
Figure 7. Pitting potentials after various surface treatments at 60 mm distance to the welding seam (blue) and directly at the welding seam (red), Electrolyte: 3000 ppm Cl\(^-\), pH 4; dU/dt: 1 mV/s [15].

Figure 8. SEM images of surfaces: top - glass-bead blasted a) SE contrast, b) BSE contrast; bottom - blasted with Al\(_2\)O\(_3\): c) SE contrast, d) BSE contrast [15].

The SEM images with SE contrast (topography) show rough surfaces for both blasting agents (corundum: R\(_s\) = 26 ± 2 µm, R\(_a\) = 3.8 ± 0.2 µm / glass-bead: R\(_s\) = 29 ± 8 µm, R\(_a\) = 4.1 ± 0.5 µm). If the same area of the surface is compared with element contrast (BSE), many dark areas can be seen, which indicate lighter elements. An analysis of these areas using EDX spots shows silicon and oxygen for glass-bead blasted surfaces, while aluminium and oxygen are present on surfaces blasted with corundum. At these points, when looking carefully at the SE contrast images, blasting particles can be recognized within the surface as result of the blasting process. These particles hinder the formation of a stable passive layer, which explains the increased susceptibility to pitting corrosion. Blasting (especially with Al\(_2\)O\(_3\)) may be suitable to remove tempering colours, but it generates surfaces that are more
susceptible to pitting corrosion than other post-treatment processes for weldments made of 1.4062 duplex stainless steels.

4 Analysis and modification of the detection limit of the KorroPad test

The KorroPad was used in research projects on the corrosion resistance of martensitic stainless steels with 13-15% chromium in order to evaluate the influence of the heat treatment on the corrosion resistance and to detect suboptimal heat treatment conditions [11, 13, 14]. Non-critical surface conditions were produced by laboratory grinding (wet, SiC abrasive, single-layer grain, 180 grit) and passivated (24 h at > 95% relative air humidity) in order to exclude the influence of surface treatment and passivation. This preparation technique allows to study microstructure and alloy related factors influencing the pitting corrosion resistance with the KorroPad test. Based on the extensive studies on martensitic stainless steels, it was concluded that stable pitting corrosion occurs when the chromium content falls locally below 11 to 13 wt-% [13, 14]. This low chromium content was therefore initially adopted as the microstructure related detection limit of the KorroPad test. This statement should be viewed critically in terms of its general validity since martensitic stainless steels possess a high carbon content.

In order to determine the detection limit of the KorroPad more precisely, ferritic reference alloys with chromium contents between 7 and 18 wt.-% were manufactured and examined electrochemically as well as with the KorroPad. It was also examined how an increase in the KorroPad composition affects the detection limit. The aim of this adjustment was a higher detection limit in the range of 16 to 18 wt.-% chromium, in order to use the KorroPad test to detect sensitized states in ferritic, austenitic and duplex stainless steels.

For the preparation of the reference alloys, various chromium contents were specifically produced in an arc melting furnace (Compact Arc Melter MAM-1, Buehler GmbH). The melting chamber was previously evacuated three times and subsequently flushed with argon to minimize oxidation during melting. 20 g of each reference alloy were melted twice in the arc and the melt was cooled on a water-cooled copper plate. The chemical composition of all reference alloys is shown in Table 1.

Table 1. Chemical composition of the ferritic reference alloys in wt.-% [26].

| Element | Cr  | Ni  | Mo  | Mn  | Si  | N   | C   | P   | S   |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 7.3 %   | 7.32| 0.28| 0.01| 0.78| 0.28| 0.005| 0.019| 0.011| 0.003|
| 9.3 %   | 9.34| 0.36| 0.01| 0.99| 0.37| 0.009| 0.025| 0.013| 0.002|
| 11.2 %  | 11.19| 0.43| 0.01| 1.17| 0.44| 0.007| 0.029| 0.015| 0.002|
| 13.6 %  | 13.56| 0.33| 0.02| 0.90| 0.46| 0.019| 0.025| 0.016| 0.002|
| 15.8 %  | 15.80| 0.25| 0.03| 0.68| 0.49| 0.015| 0.029| 0.020| 0.002|
| 17.4 %  | 17.36| 0.18| 0.03| 0.48| 0.49| 0.024| 0.022| 0.018| 0.002|
| 18.0 %  | 17.96| 0.16| 0.03| 0.42| 0.51| 0.023| 0.025| 0.019| 0.002|

The reference alloys were solution annealed at 1050 °C for 0.5 h, quenched in water and then annealed at 800 °C for 1 h to transform the martensitic microstructure at chromium contents of less than 10 wt.% chromium to ferrite. The alloys were then cold rolled to 50% of their initial height and annealed again at 800 °C for 1 h to achieve a recrystallized microstructure.

The corrosion resistance of the ferritic reference alloys was characterized regarding the passivation behaviour in 0.1 M H2SO4 solution and in relation to the critical pitting potentials in 0.1 M NaCl solutions [26] to verify the increasing corrosion resistance with the Cr content. The samples were ground with 180 SiC paper in preparation for these electrochemical investigations and in case of critical pitting potentials also passivated for 24 h at more than 95% relative humidity. This should enable the formation of a stable passive layer and minimize the influence of surface treatment and passivation on the pitting
corrosion resistance. The results regarding critical pitting potentials and passivation current density are presented in Figure 9.

![Figure 9](image)

**Figure 9.** Critical pitting potential and passivation current density of the ferritic reference alloys [26].

The critical pitting potential shows a linear increase with the chromium content and the passivation current density exhibit a logarithmic reduction with increasing chrome content. This is exactly the expected behaviour in relation to the chromium content and this allows the investigation of the detection limit of the KorroPad test. The reference alloys with increasing corrosion resistance were finally examined using the KorroPad test. In addition to the "standard" KorroPad with 0.1 M NaCl and 1 mM K,\(_3\)[Fe(CN)\(_6\)], modified KorroPads with 0.2 M NaCl and 2 mM K,\(_3\)[Fe(CN)\(_6\)], 0.5 M NaCl and 5 mM K,\(_3\)[Fe(CN)\(_6\)] and 1.0 M NaCl and 10 mM KHCF were used to determine the resulting detection limit. All KorroPads were applied for a test period of 15 minutes, scanned optically with 600 dpi and assessed according to their visual appearance (Figure 2). The results of the KorroPad tests with increasing corrosivity on alloys with different concentrations of chromium are presented in Figure 10.

![Figure 10](image)

**Figure 10.** Results of the KorroPad test with four different KorroPad corrosiveness on ferritic reference alloys with different chromium content.
The standard KorroPads (0.1 M NaCl, 1 mM K$_3$[Fe(CN)$_6$]) shows a transition of stable pitting to metastable pitting is between 9.3 and 11.2 wt.-% Cr. With 13.6 wt.-% or more Cr, pitting corrosion no longer occurs and the passive layer remains stable (passive).

Increasing the corrosivity of the KorroPad by applying higher NaCl and K$_3$[Fe(CN)$_6$] concentrations shifts the detection limits to higher chromium contents. In addition, less metastable pitting corrosion indications can be seen, since the higher redox potential favours the differentiation between stable pitting and the passive state. The KorroPad-results of all four KorroPad modifications allows an easy comparison of different alloy or microstructure related corrosion resistances. Furthermore, the KorroPads with 0.5 M NaCl, 5 mM K$_3$[Fe(CN)$_6$] are more suitable for the detection of material related pitting susceptibility of stainless steels with 18 % Cr due to the higher detection limit. Figure 11 shows schematically how a change in the KorroPad concentration affects the open circuit potential and the critical pitting corrosion potential for a KorroPad test on the alloy with 15.8 wt.-% Cr.

![Figure 11](image)

**Figure 11.** Change in the anodic polarization and the critical pitting potential when increasing the corrosivity of the KorroPad for a ferritic stainless steel with 15.8 wt.-% Cr.

The standard KorroPad (0.1 M NaCl, 1 mM K$_3$[Fe(CN)$_6$]) polarizes the surface to a potential within the passive range. The anodic polarization by 1 mM K$_3$[Fe(CN)$_6$] is not sufficient to polarize the reference alloy with 15.8% chromium up to the pitting potential in a 0.1 M NaCl solution. Consequently, no stable pitting corrosion is initiated and no KorroPad indications occur.

The higher concentrations of K$_3$[Fe(CN)$_6$] increase the anodic polarization and the distance to the pitting corrosion potential is reduced. At the same time, increasing the NaCl concentration lowers the critical pitting corrosion potential. The combination of 0.5 mM K$_3$[Fe(CN)$_6$] and 0.5 M NaCl is sufficient to reach the critical pitting potential, initiate pitting corrosion and generate large indications on the reference alloy with 15.8 wt.-% Cr.

Since the critical pitting corrosion potentials are shifted to higher potentials with increasing chromium content, stable pitting corrosion does not yet occur in the reference alloys with 17.4 and 18 wt.-% Cr. However, local chromium depletion, which reduces the pitting corrosion resistance, can be detected on ferritic, austenitic and duplex stainless steels using KorroPads with higher corrosivity. This can be demonstrated on sensitized stainless steels which are susceptible to intergranular corrosion and pitting corrosion.

5 Sensitization behaviour of the lean duplex stainless steel X2CrNiN23-4

Secondary phases (M$_2$3C$_6$, Cr$_2$N, σ, γ$_2$) can precipitate due to welding of the duplex stainless steel X2CrNiN23-4 (1.4062) within the heat affected zone. This can cause local chromium depletion at the ferrite-ferrite grain boundaries and reduce the corrosion resistance. The sensitization behaviour was
characterized by aging studies at 600 °C with ageing times from 0.1 h to 10 h. The generated microstructures are presented in Figure 12. Significant precipitation is visible in the SEM images after aging for 0.2 h. Longer durations result in precipitation growth. Finally, precipitation starts at 10 h within the ferrite grains.

Figure 12. Microstructure of the duplex stainless steel X2CrNiN23-4 after aging at 600 °C [27].

The corrosion resistance of the aging conditions was examined by the EPR method, by critical pitting temperatures (CPT) according to ASTM G150 and with the modified KorroPads described in the previous chapter. Figure 13 shows the results regarding the degree of sensitization (DOS), critical pitting temperatures (CPT) and the results of the four KorroPad modifications.

Figure 13. Results of the KorroPad test with various concentrations, the CPT values (°C) and the DOS [%] for the sensitization at 600 °C of the duplex stainless steel X2CrNiN23-4 [27]; dashed lines show the first reduction of pitting corrosion resistance.
KorroPads with 0.1 M NaCl and 1 mM K₃[Fe(CN)₆] are not suitable to detect the reduction in corrosion resistance due to sensitization at 600 °C, while decreasing CPT values and increasing degree of sensitization (DOS) show the expected behaviour. This can be explained by the fact that the depletion zones formed during annealing do not fall below the detection limit of the standard KorroPad. Increasing the corrosivity of the KorroPads up to 0.5 M NaCl and 5 mM K₃[Fe(CN)₆] detects the loss of pitting corrosion resistance with for aging times of 0.2 h or more. Therefore, sensitization can be detected with the modified KorroPad test and the EPR method with a comparable detection limit. The modified KorroPad test is therefore suitable to generate T-t-sensitization diagrams for different stainless steels. The critical pitting corrosion temperatures show the same trend. However, the modified KorroPad test can be carried out much more effectively using parallel tests. Electrochemical experiments (EPR method, ASTM G150) are much more time consuming and require suitable electrochemical devices.

6 Conclusions
The application examples of the KorroPad test from industry and research show that the pitting corrosion resistance of stainless steels can be assessed in a simple, quick and inexpensive way. Especially the visual indication of the pitting corrosion resistance is very advantageous, which is generated in a very short test duration (15 min) without a complex electrochemical test setup. The sensitivity of the KorroPad regarding the pitting corrosion resistance is comparable with other electrochemical methods and long-term experiments within artificial or natural atmospheres [1]. The KorroPad is therefore an interesting alternative to time-consuming long-term tests and complex electrochemical analysis methods for manufacturers, processors, users and researchers of stainless steels. The KorroPad is often used in current projects for screening tests and extensive parameter studies in order to limit the scope of systematic electrochemical investigations. Various questions and problems relating to stainless steels can be addressed, which are traced back to surface-related and material-related changes of the pitting corrosion resistance.

The current main application of the KorroPad test is the detection of surfaces prone to pitting corrosion, which can be correlated with an incomplete formation of the passive layer. The KorroPad can test whether the passive layer is formed properly or if there is a surface with critical defects. Furthermore, the KorroPad locates the most corrosion-prone surface areas, which was successfully illustrated by the example of ground surfaces. This location of critical defects makes it easy to clarify the causes of higher susceptibility to corrosion by further analytical examinations at these points.

If a defect-free surface is established by laboratory grinding and a stable passive layer is formed by suitable passivation, then the KorroPad can be used to examine the pitting corrosion resistance of alloys or microstructural states. KorroPad test on ferritic reference alloys with chromium contents from 7.3 to 18.0 wt.-% show, that chromium contents of less than 11.2 wt.-% result in stable pitting corrosion, even with non-critical surface and suitable passivation. This material related detection limit of the standard KorroPad can be shifted to higher chromium contents by increasing the concentrations of NaCl and K₃[Fe(CN)₆]. This modified KorroPads are suitable to detect material and microstructure related susceptibility to pitting corrosion for higher alloyed stainless steels like 1.4301, 1.4404 and 1.4062. This has been demonstrated using the example of sensitization of the duplex stainless steel X2CrNiN23-4. KorroPads with 5 times higher concentration (0.5 M NaCl, 5 mM K₃[Fe(CN)₆]) compared to standard KorroPad (0.1 M NaCl, 1 mM K₃[Fe(CN)₆]), characterised sensitization in perfect correlation to the well-established EPR method.

These results show that modified KorroPads can be used as a powerful tool to easily, quickly and effectively detect material-related causes of pitting corrosion and check the corrosion resistance of various stainless-steel batches. A vulnerability identified and localized with the KorroPad can then be investigated using electrochemical, metallographic and analytical methods to clarify the specific cause.
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