BAM
Bundesanstalt für Materialforschung und -prüfung

TEST REPORT

REUTER CLEANOX 5.0 - PASSIVE LAYER CONDITION AFTER WELD SEAM TREATMENT

BAM reference	22029018 (English translation)
Copy	Electronic version, legally binding is exclusively sealed german version
Customer	Reuter GmbH & Co. KG Schimmelbuschstraße 9e 40699 Erkrath
Order date	23.08.2022
Reference	-
Receipt of order	03.01.2023
Test samples	Stainless steel 1.4301 and 1.4404 Reuter Cleanox 5.0 Polisher electrolyte
Receipt of samples	09.02.2023
Test date	April/May 2023
Test location	Bundesanstalt für Materialforschung (BAM) Unter den Eichen 87, 12005 Berlin
Test procedure according to	BAM STAA-76-08-FES ASTM A 967 KorroPad Prüfanleitung

This test report consists of page 1 to 26.

1 The subject of the examination

The subject of the order is corrosion investigations and tests to check the passive layer condition of stainless steel weld specimens after treatment with a weld cleaning device. The aim is, on the one hand, to evaluate the effectiveness of the process concerning passivation of the surface left behind with the KorroPad test and, on the other hand, to confirm the absence of ferrite using two tests by ASTM A967 [2] (Practice D+E).

2 Test material

The customer provided BAM with stainless steel test sheets made of 1.4301 (X5CrNi18 10) with ground surface and 1.4404 (X2CrNiMo17-12-2) with a cold-rolled surface. The specimen sheets made of 1.4301 are used specifically in the KorroPad tests, but because of the surface finish, they are only used for exemplary proof tests in the ASTM tests.

To check the chemical composition of the material, a spectral analysis was carried out using a Spectrolab spark emission spectrometer (FES) from Spectro. The values given are mean values from 5 individual measurements.

Based on the FES analysis, the alloy compositions for the stainless chromium-nickel steels of grades 1.4301 and 1.4404 could be confirmed. The alloying elements are within the limits of the alloy contents specified in DIN EN 10088-2 [1] for these materials. The deviation in the measured value of the nickel content of 1.4301, which is marginally below the nominal value, is not of corrosion-relevant magnitude.

Tab. 1: Chem. composition from FES analysis (MW n=5) with standard deviation SD and normative composition of the two steel grades according to DIN EN 10088 2:2014 [1]; in wt.%.

Measurement		C	Si	Mn	P	S	Cr	Mo	Ni	N
1.4301 (Specimen 23) MW n=5		0,0211	0,459	1,52	0,0366	0,0024	18,15	0,364	7,87	0,075
SD		*0,0007*	*0,0038*	*0,0125*	*0,0020*	*0,0002*	*0,0487*	*0,0091*	*0,0356*	*0,0025*
1.4404 (Specimen 26) MW n=5		0,0216	0,410	1,21	0,0297	0,0023	16,73	2,03	10,01	0,039
SD		*0,0002*	*0,0039*	*0,0046*	*0,0014*	*0,0001*	*0,0147*	*0,0114*	*0,0399*	*0,0015*
DIN EN 10088-2 [1]	1.4301 X5CrNi18-10	*<0,07*	*<1,00*	*<2,00*	*<0,045*	*<0,015*	*17,5 - 19,5*	*-*	*8,0 - 10,5*	*<0,10*
	1.4404 X2CrNiMo17-12-2	*<0,03*	*<1,00*	*<2,00*	*<0,045*	*<0,015*	*16,5 - 18,5*	*2,00 – 2,50*	*10,0 – 13,0*	*<0,10*

3 Test device and test procedure

A Cleanox 5.0 unit (Fig. 1), which was provided by the customer, was used for the post-weld treatment.

Fig. 1: Mobile worktable (l.), Cleanox 5.0-unit front (bottom left) and rear (bottom right), polisher electrolyte and distilled water (top, top right)

The specimen sheets were cleaned with ethanol and distilled water before testing and photographically documented.

The "Cleanox 5.0" weld cleaning device was set up according to the customer's specifications and filled with the "Polisher" cleaning solution supplied. **The unit was operated exclusively in the "Cleaning" mode.**

In the BAM laboratories, the weld seam cleaning was carried out manually in light circular movements according to the customer's specifications (Fig. 2). The cleaning time was 10 seconds. After cleaning, the heat-affected zone was metallic-bright and free of tarnish. In individual tests, the cleaning time was extended to 20 seconds, which resulted in a significant change in the surface structure. Exemplary surfaces are compared in Fig. 3.

During the process, the surface temperature of the test sheets in the weld area was measured and documented using an infrared temperature gauge (Tab. 2). Subsequently, the test sheets were sprayed with distilled water and thus cooled down to approx. 25 °C for the subsequent tests.

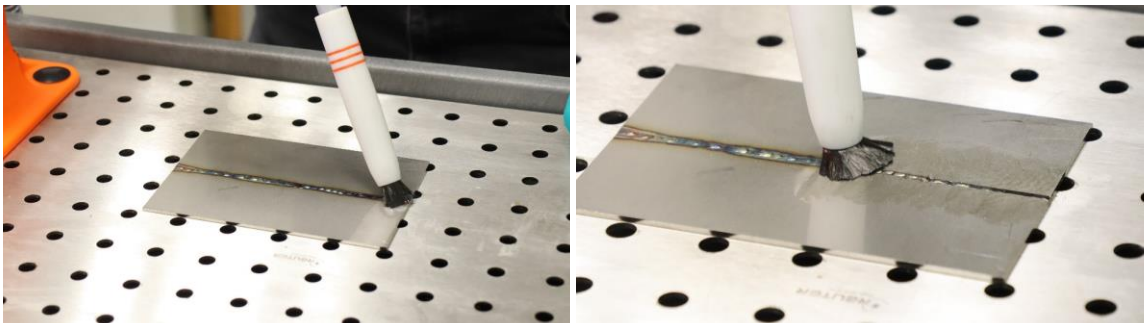


Fig. 2: Execution of weld cleaning on a sheet metal coupon 1.4404

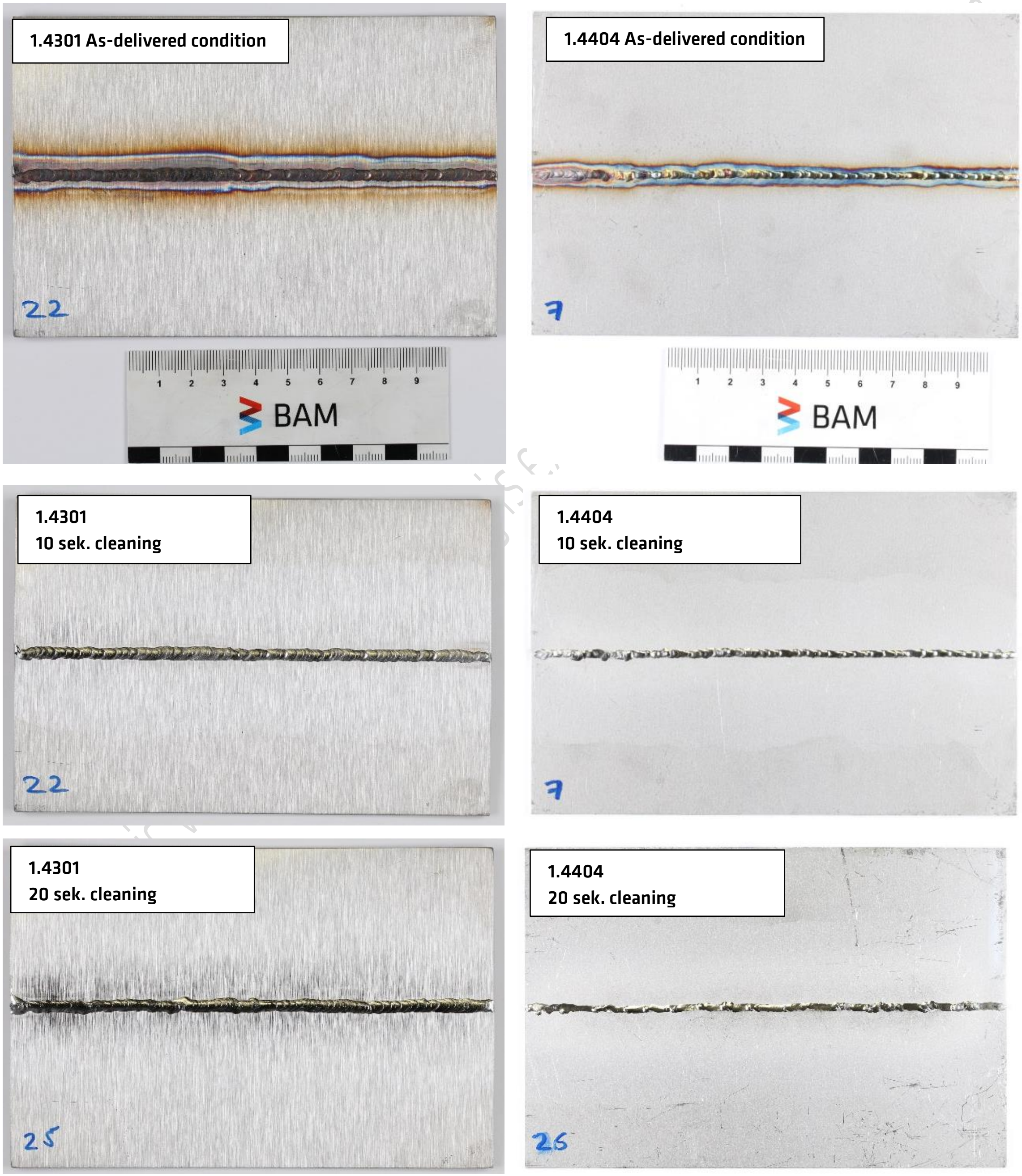


Fig. 3: Sheet metal coupons made of 1.4301 (l.) and 1.4404 (r.) as delivered with tarnish (top), after 10 sec. (middle) and 20 sec. (bottom) post-weld treatment with Reuter Cleanox 5.0

Tab. 2: Surface temperatures after weld post-treatment

Sheet coupon	Material	Duration of weld cleaning in seconds	Temperature IR in °C
1	1.4404	10	90,0
2	1.4404	10	91,5
3	1.4404	10	85,6
4	1.4404	10	79,8
5	1.4404	10	74,5
6	1.4404	10	79,4
7	1.4404	10	77,2
8	1.4404	10	70,3
9	1.4404	10	68,5
10	1.4404	10	70,5
11	1.4404	10	67,1
12	1.4404	10	82,1
13	1.4404	10	86,2
14	1.4404	10	71,8
15	1.4404	10	74,6
18	1.4404	20	109,0
26	1.4404	20	102,0
22	1.4301	10	85,0
23	1.4301	10	87,8
24	1.4301	10	107,0
25	1.4301	20	103,0

4 ASTM A967 [2] Practice D (Copper Sulfate Test)

This test is suitable for the detection of free iron on the surface of stainless austenitic steels, including the 300 series (here 304 = 1.4301 and 316L = 1.4404).

The test solution is prepared by dissolving 4 g of copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$ in 250 ml of distilled water, to which 1 ml of 95-100% sulfuric acid (H_2SO_4) was added.

The test solution was applied by pipette to the test plates in the area of weld post-processing and left on them for a period of 6 minutes. In order not to influence or rinse off possible copper deposits, the surfaces were cleaned under running water. The tested sample must not show any copper deposits.

5 reference samples were tested. The individual results for material 1.4404 are shown in Fig. 4 to Fig. 8 and for material 1.4301 in Fig. 9.

Result: none of the specimens showed copper deposition, and no free iron could be detected. The test was passed!

4.1 Sheet coupon 2 – 1.4404

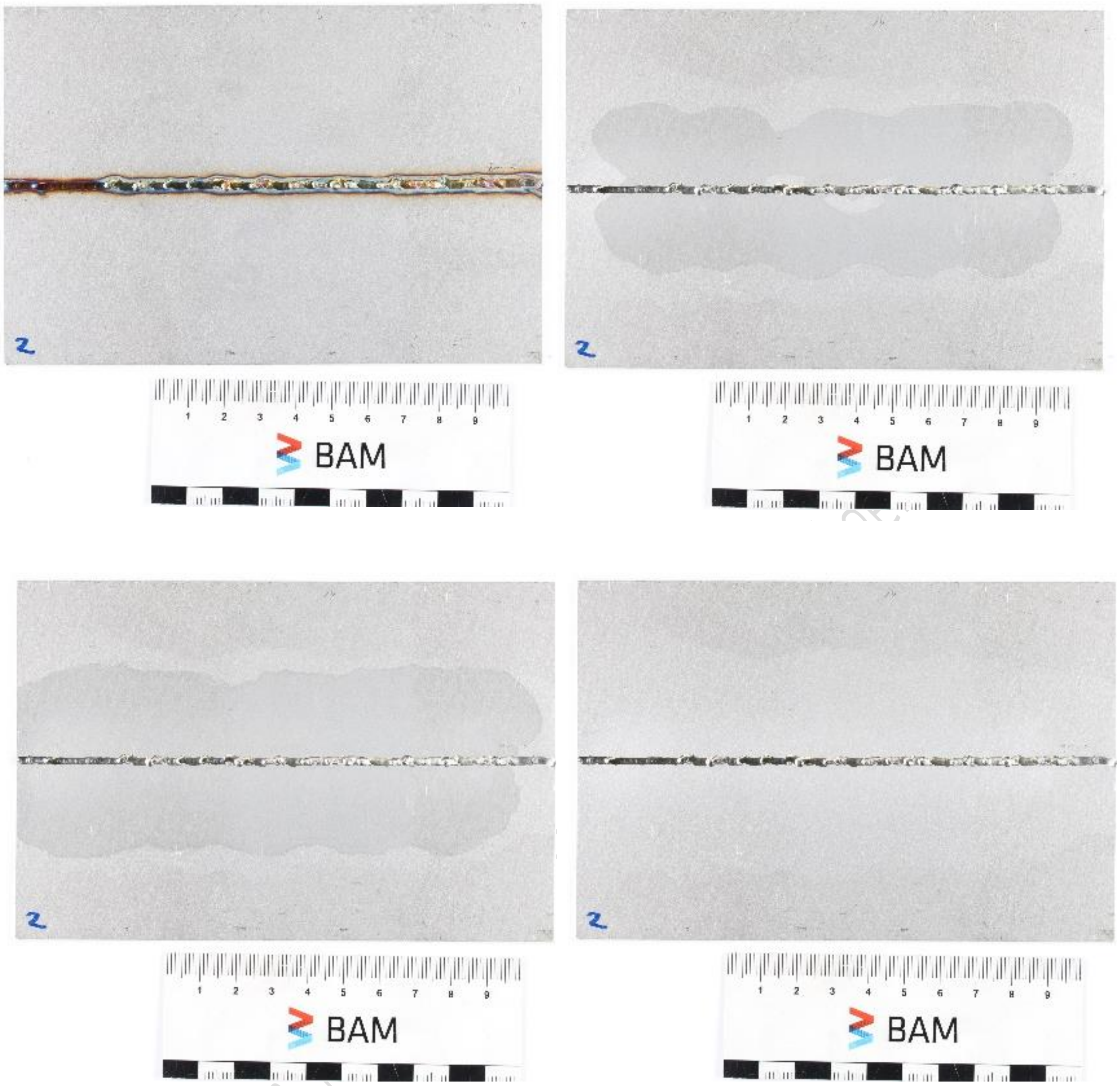


Fig. 4: ASTM A967 Practice D, sheet coupon 2, 1.4404, initial condition (top left), test solution applied after weld post-treatment (top right), test result after 6 minutes (bottom left) after rinsing with distilled water (bottom right). water (bottom right); no indications

4.2 Sheet coupon 7 – 1.4404

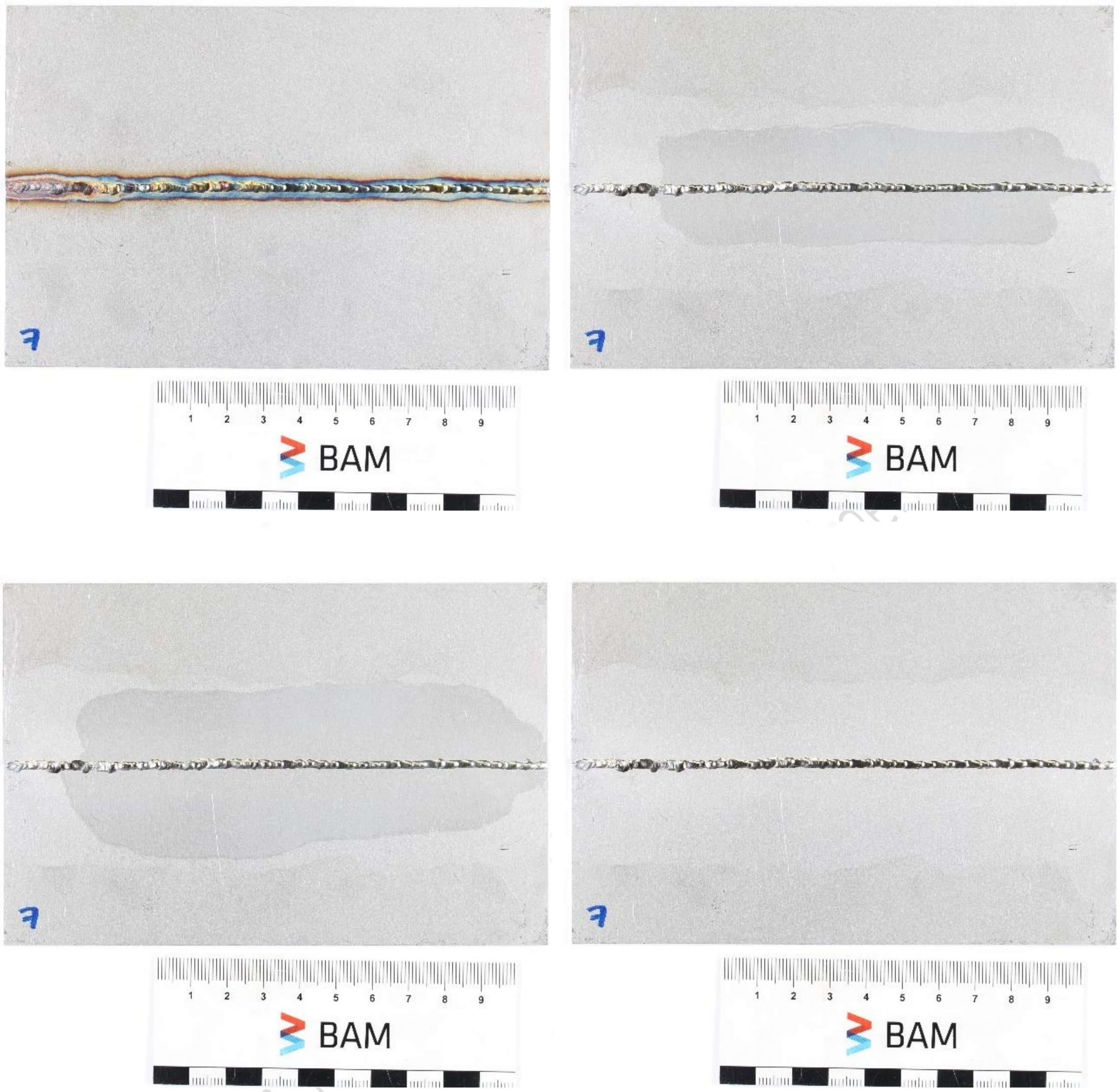


Fig. 5: ASTM A967 Practice D, sheet coupon 7, 1.4404, initial condition (top left), test solution applied after weld post-treatment (top right), test result after 6 minutes (bottom left) after rinsing with distilled water (bottom right). water (bottom right); no indications

4.3 Sheet coupon 8 – 1.4404

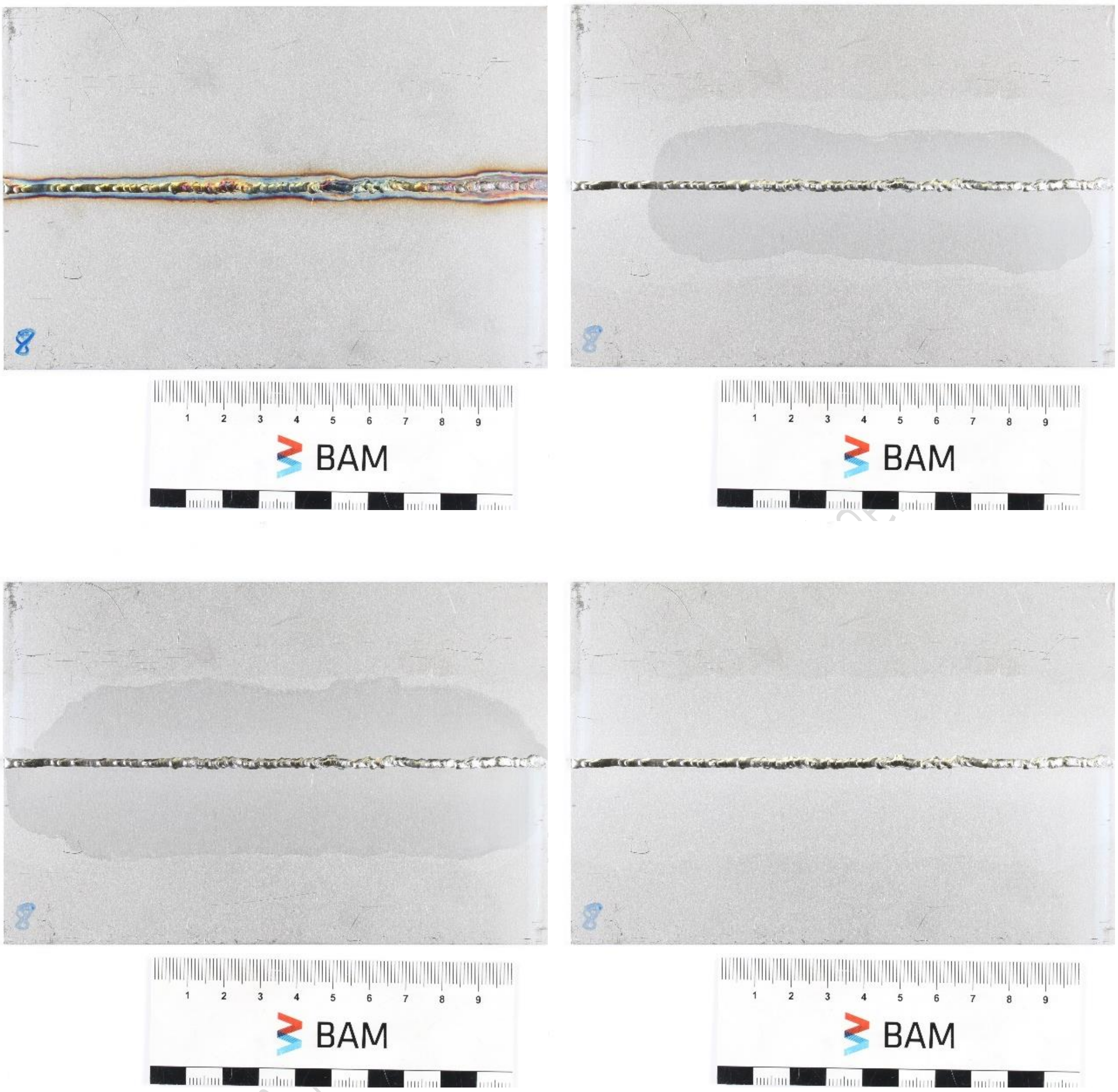


Fig. 6: ASTM A967 Practice D, sheet coupon 8, 1.4404, initial condition (top left), test solution applied after weld post-treatment (top right), test result after 6 minutes (bottom left) after rinsing with distilled water (bottom right). water (bottom right); no indications

4.4 Sheet coupon 9 – 1.4404

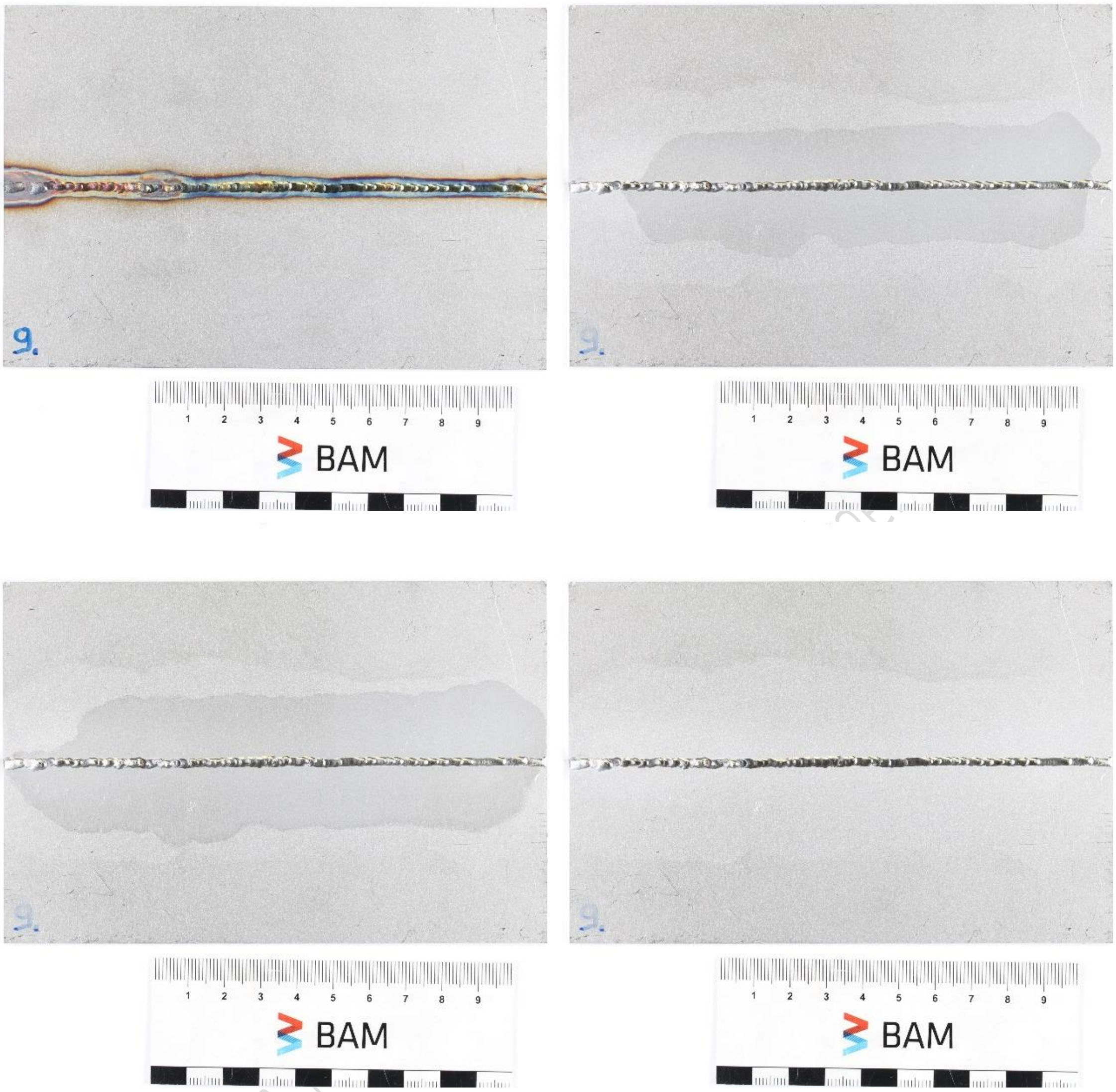


Fig. 7: ASTM A967 Practice D, sheet coupon 9, 1.4404, initial condition (top left), test solution applied after weld post-treatment (top right), test result after 6 minutes (bottom left) after rinsing with distilled water (bottom right). water (bottom right); no indications

4.5 Sheet coupon 10 – 1.4404

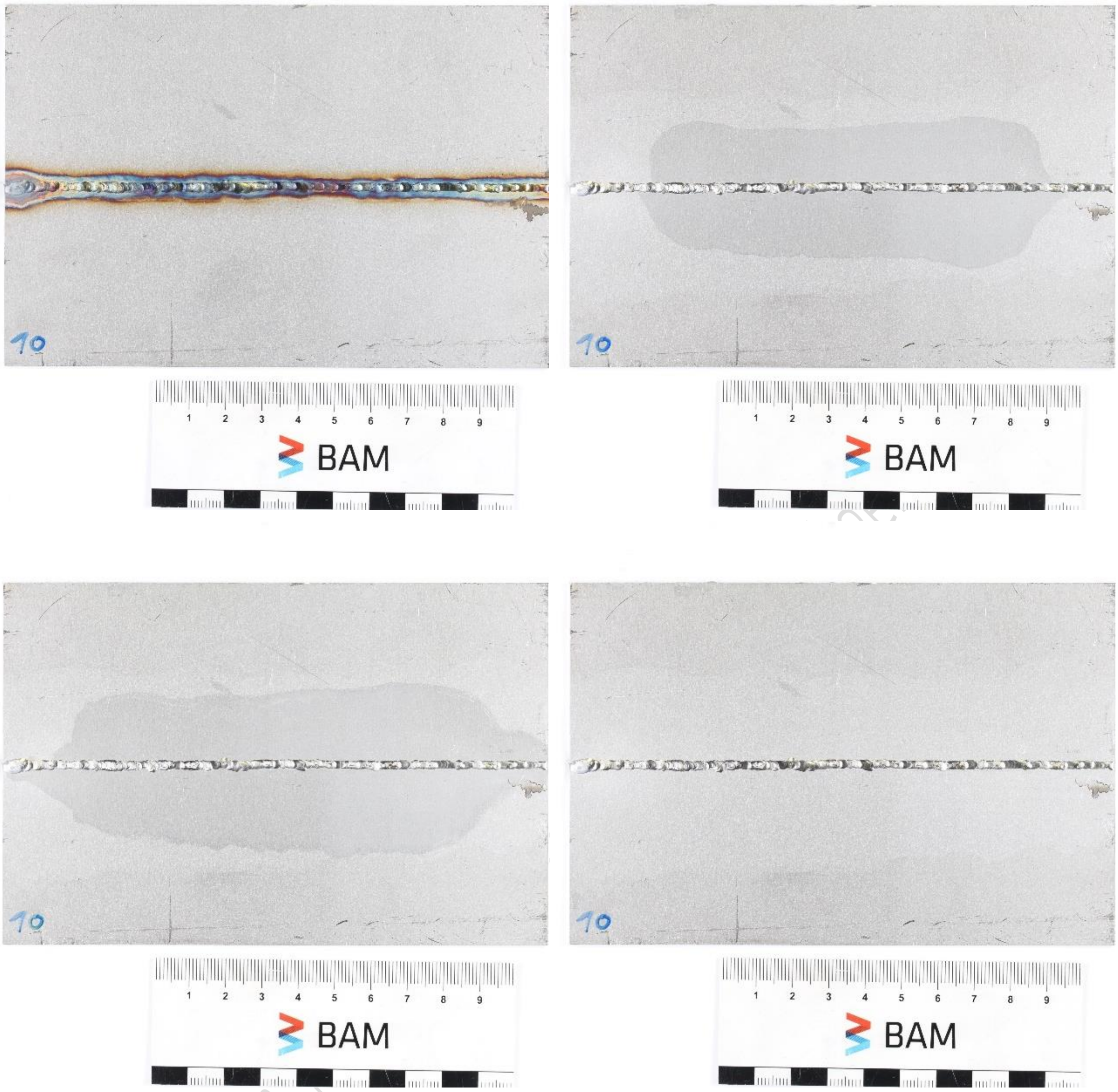


Fig. 8: ASTM A967 Practice D, sheet coupon 10, 1.4404, initial condition (top left), test solution applied after weld post-treatment (top right), test result after 6 minutes (bottom left) after rinsing with distilled water (bottom right). water (bottom right); no indications

4.6 Sheet coupon 24 – 1.4301

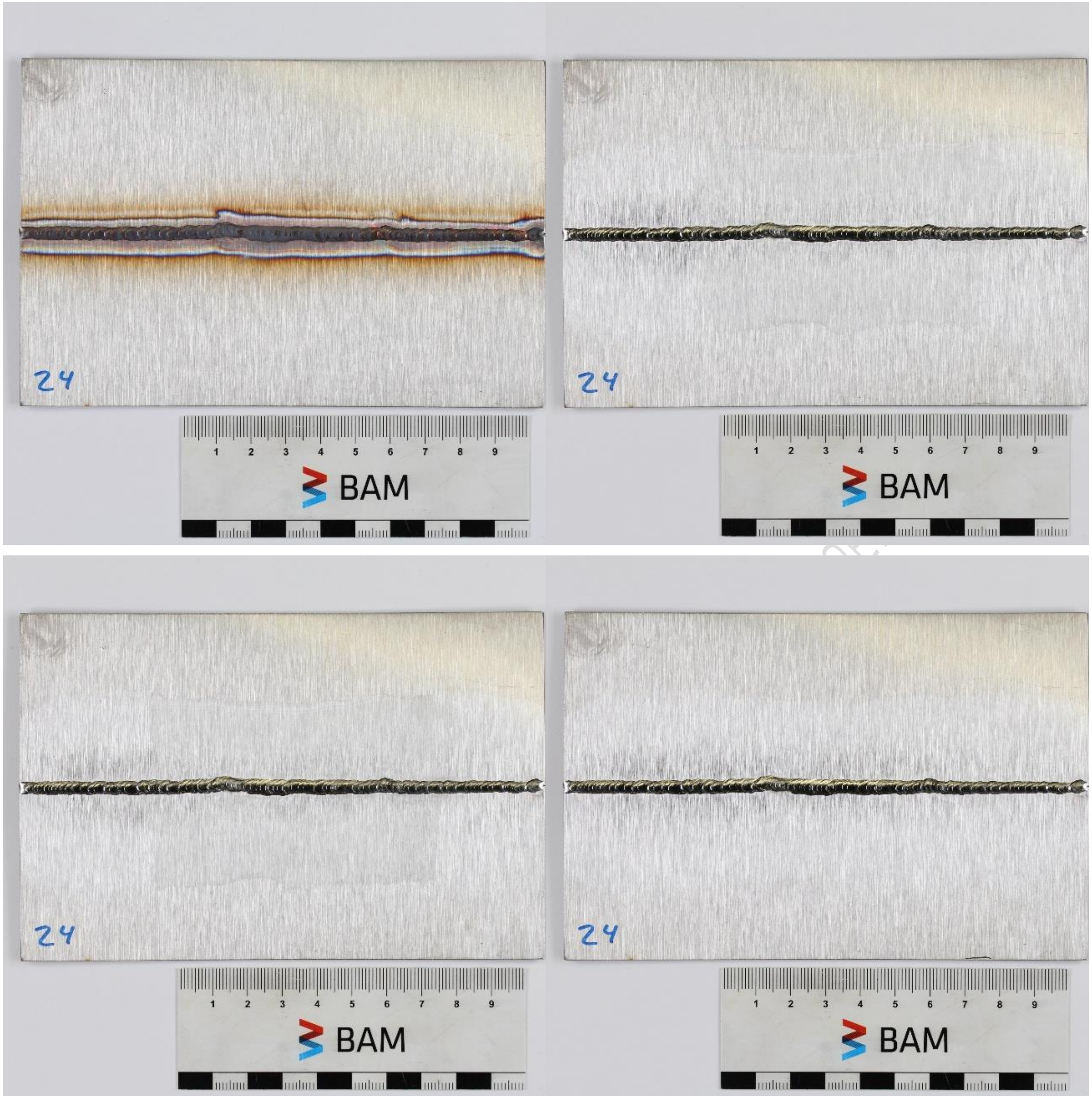


Fig. 9: ASTM A967 Practice D, plate coupon 24, 1.4301, initial condition (top left), test solution applied after weld post-treatment (top right), test result after 6 minutes (bottom left) after rinsing with distilled water (bottom right). water (bottom right); no indications

5 ASTM A967 [2] Practice E (Potassium Ferricyanide-Nitric Acid Test)

This test is recommended when the detection of very small amounts of free iron is required on 200 and 300-series austenitic stainless steels and duplex stainless steels.

The test solution is prepared by adding 10 g of chemically pure potassium ferrocyanide to 500 ml of distilled water, to which 30 ml of 70% nitric acid is added and stirred until all the ferrocyanide is dissolved and diluted to 1000 ml with distilled water.

The test solution must be freshly prepared on the day of testing as it will discolor with time. The test solution is applied to the specimen, and the formation of a dark blue color within 30 seconds indicates the presence of metallic iron. A slowly developing paler blue color usually indicates the presence of iron oxides. The test solution was applied with a pipette to the surface of the sheet metal coupons in the weld finishing zone. Five reference specimens were tested. The individual results are shown for material 1.4404 in Fig. 10 to Fig. 14 and for material 1.4301 in Fig. 15.

Result: none of the specimens showed blue coloration, and no free iron could be detected. The test was passed!

5.1 Sheet coupon 1 – 1.4404

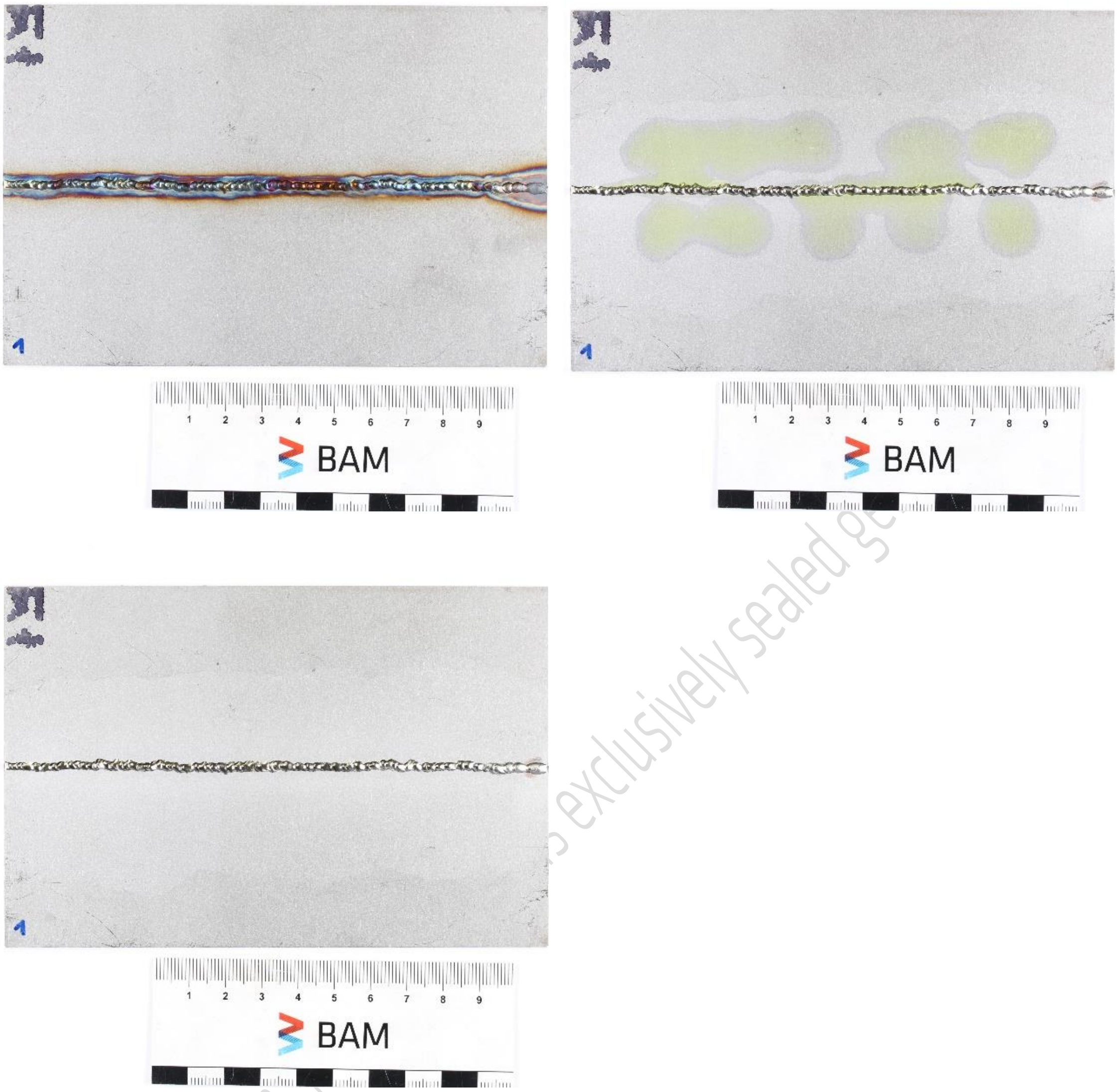


Fig. 10: ASTM A967 Practice E, sheet coupon 1, 1.4404, initial condition (top left), test solution applied after weld post-treatment, test result after 30 seconds (top right) after rinsing with distilled water (bottom left). water (bottom left); **no indications**

5.2 Sheet coupon 3 – 1.4404

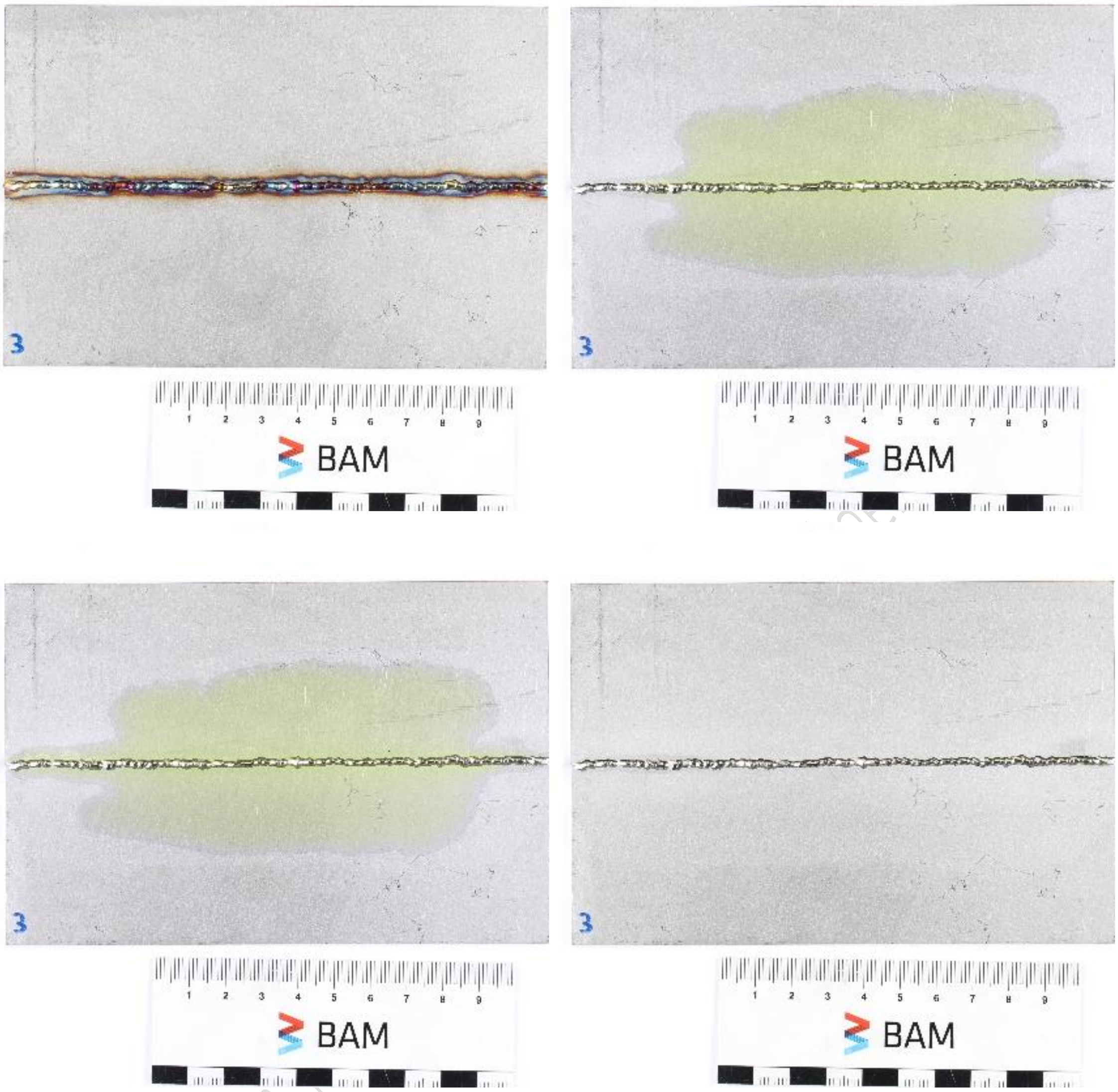


Fig. 11: ASTM A967 Practice E, sheet coupon 3, 1.4404, initial condition (top left), test solution applied after weld post-treatment (top right), test result after 30 seconds (bottom left) after rinsing with distilled water (bottom right). water (bottom right); no indications

5.3 Blechcoupon 4 – 1.4404

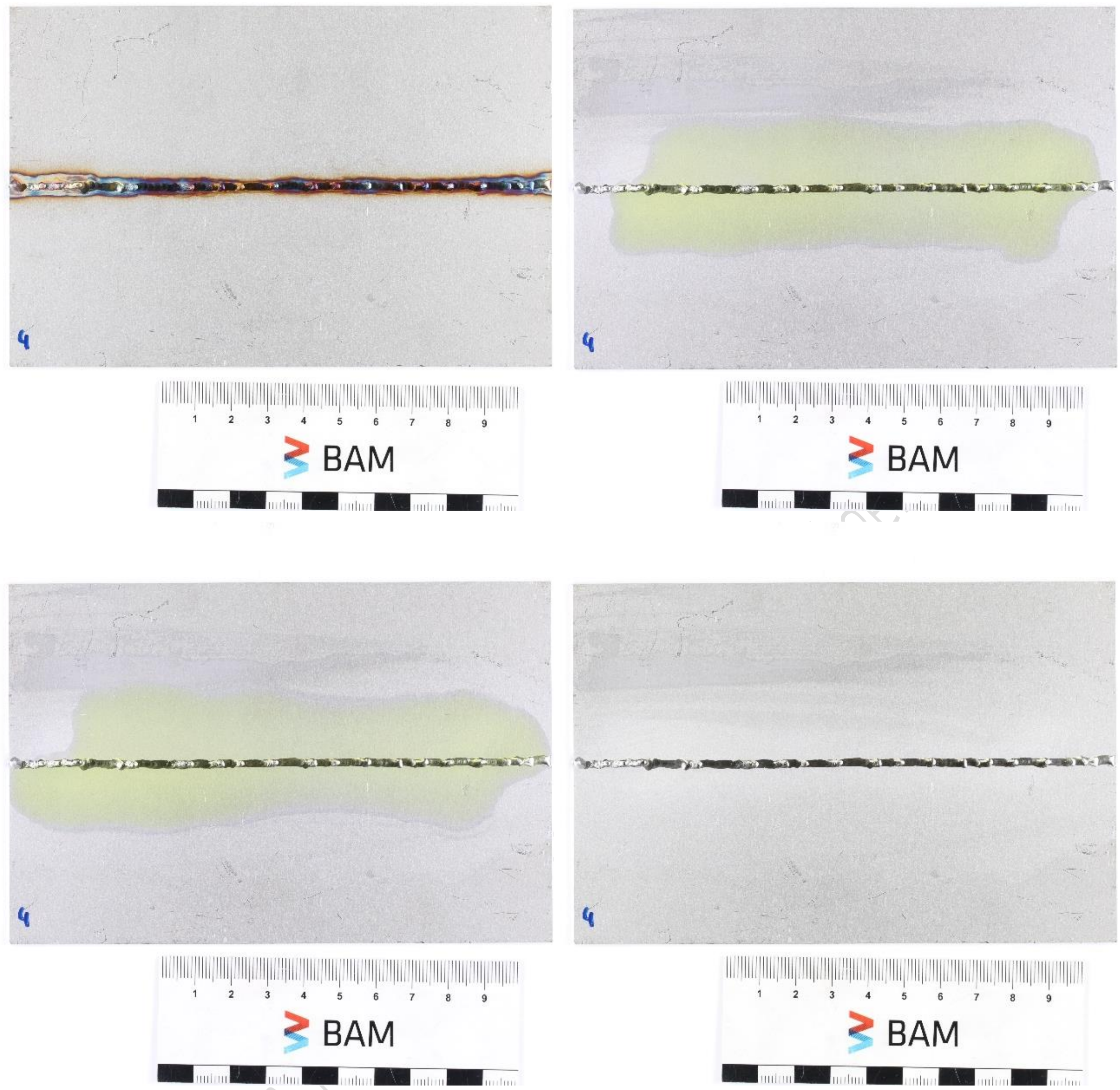


Fig. 12: ASTM A967 Practice E, sheet coupon 4, 1.4404, initial condition (top left), test solution applied after weld post-treatment (top right), test result after 30 seconds (bottom left) after rinsing with distilled water (bottom right). water (bottom right); no indications

5.4 Sheet coupon 5 – 1.4404

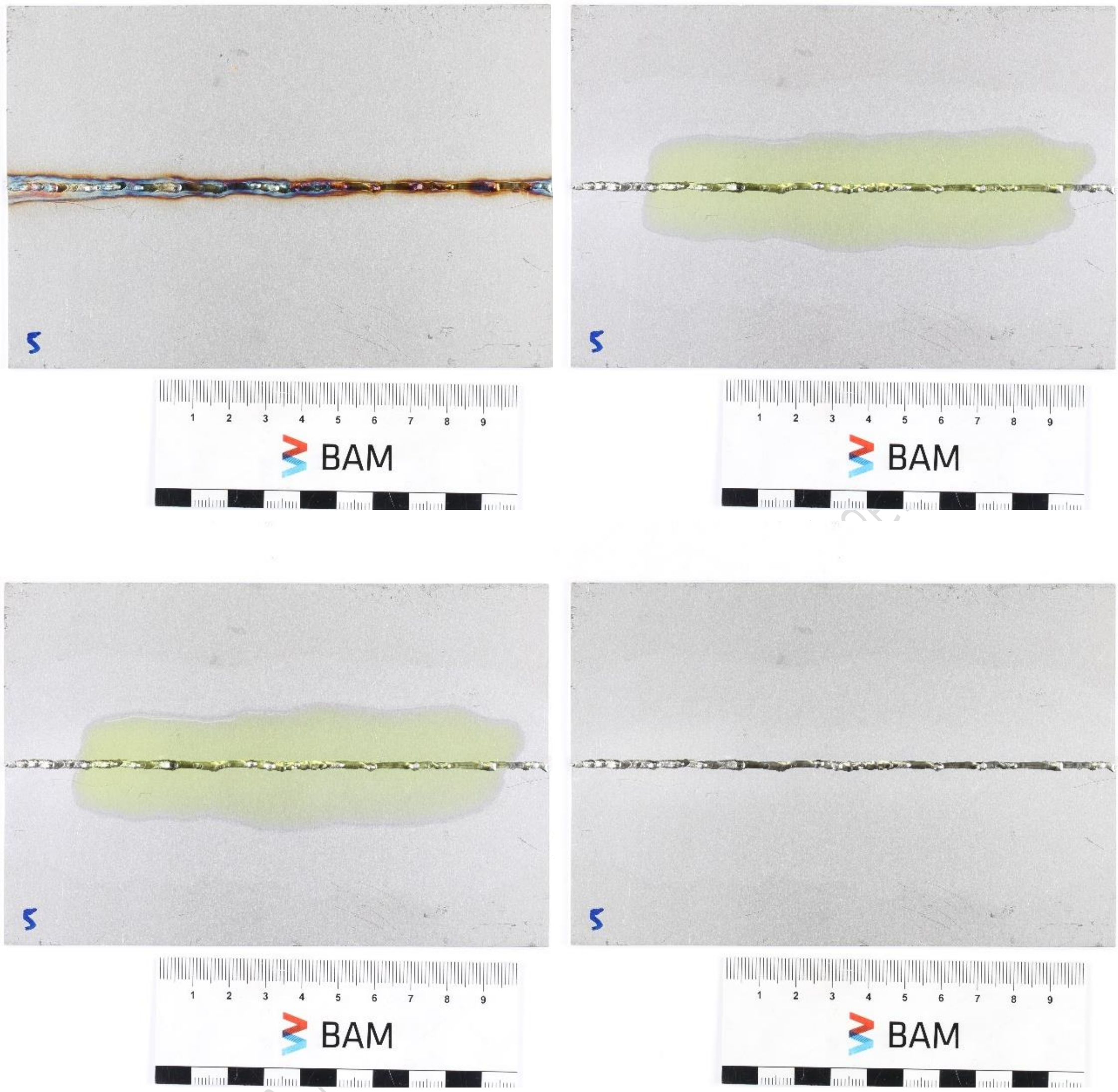


Fig. 13: ASTM A967 Practice E, sheet coupon 5, 1.4404, initial condition (top left), test solution applied after weld post-treatment (top right), test result after 30 seconds (bottom left) after rinsing with distilled water (bottom right). water (bottom right); no indications

5.5 Sheet coupon 6 – 1.4404

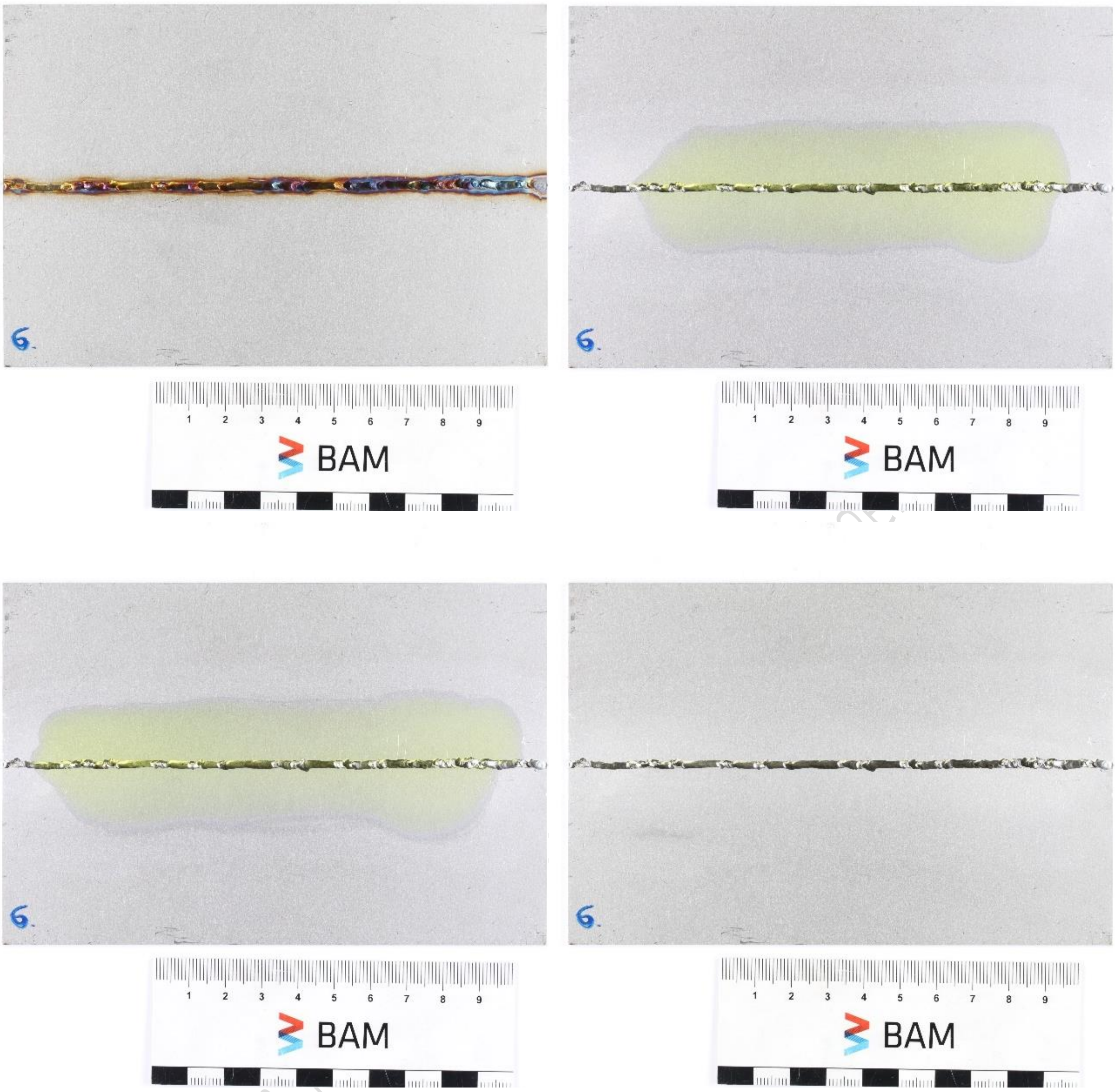


Fig. 14: ASTM A967 Practice E, sheet coupon 6, 1.4404, initial condition (top left), test solution applied after weld post-treatment (top right), test result after 30 seconds (bottom left) after rinsing with distilled water (bottom right). water (bottom right); no indications

5.6 Sheet coupon 23 – 1.4301

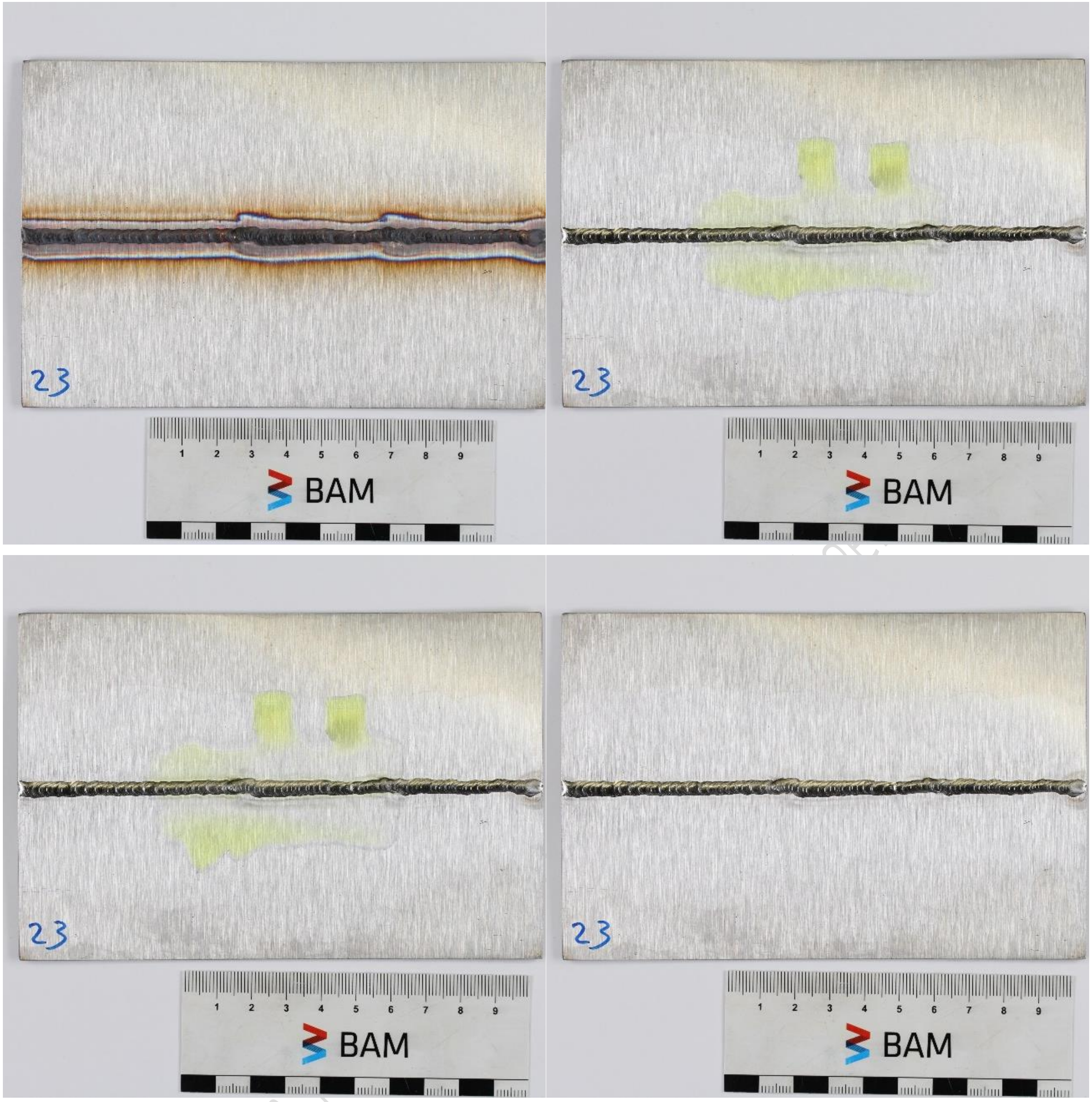


Fig. 15: ASTM A967 Practice E, sheet coupon 23, 1.4301, initial condition (top left), test solution applied after weld post-treatment (top right), test result after 30 seconds (bottom left) after rinsing with distilled water (bottom right). water (bottom right); no indications

6 KorroPad®

Non-destructive indicator tests using KorroPad® were carried out to investigate the passive layer stability. This is a test pad with an indicator solution to which an activator is added and which is kept in a gel-like state using a binder. At points where the passive layer of the stainless steel is disturbed, the passage of iron ions is indicated by a color change to "Berliner blue". The test principle is shown in Fig. 16. Iron particles that have already been completely oxidized (e.g. corrosion products) are not indicated, as the iron ions are already present there in a reacted state.

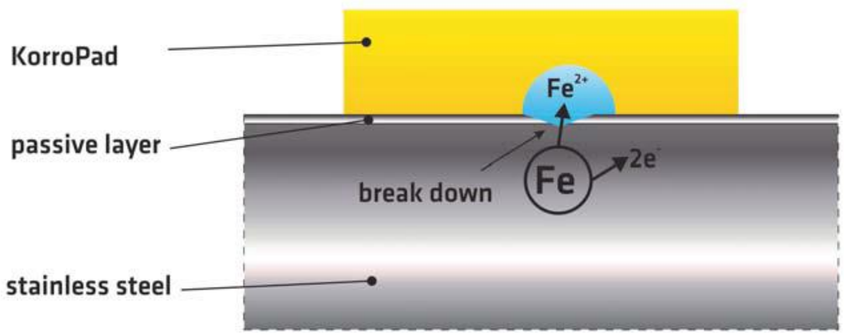


Fig. 16: Test principle of the KorroPad® test, blue coloration of the indicator due to contact with iron ions at passive layer defects

The KorroPads used had a diameter of 22 mm, and the test area was thus approx. 380 mm² and had an ingredient concentration of 1.0 mM potassium ferricyanide and 0.1 M NaCl. This set a redox potential in the test medium of E_{Redox} = +280 mVAg/AgCl.

A temperature of 22 °C could be measured for the ambient air in the laboratory. On the tested surfaces, surface temperatures of 23-25 °C were determined by IR measurement at the actual time of testing. The measurements were carried out with a thermo-hygrometer GANN Hydromette BL Compact TF-IR 2.

The duration of the weld seam post-processing (SPP) was varied in two steps (10 + 20 seconds) for the materials 1.4404 and 1.4301 to check the influence of the exposure time. On the one hand, the KorroPad® test was performed directly after the post-weld treatment to check the condition of the passive layer after the post-weld treatment process. On the other hand, the same sheet coupons were stored for 96 hours in a humidity chamber at 30 °C/100 %rH before a new KorroPad® test, which specifically supports the natural formation of the passive layer. The tested variants are listed in Tab. 3.

The KorroPads were removed 15 minutes after application and high-resolution scanned. The test surfaces were cleaned with distilled water after the test.

Tab. 3: Parameter matrix for KorroPad® testing

Duration weld seam post-treatment (SPP)	Test time	Material 1.4404	Material 1.4301
10 Sekunden	**a)** directly after SPP	5 sheets	1 sheet
	b) SPP + 96 h Exposition @ 30 °C/100%rH		
20 Sekunden	**a)** directly after SPP	1 sheet	1 sheet
	b) SPP + 96 h Exposition @ 30 °C/100%rH	Not executed	

Fig. 17 to Fig. 21 show the sheet coupons made of material 1.4404 after 10 seconds of post-weld treatment with the applied KorroPads and the KorroPad test results after 15 minutes of testing. It can be seen that indications appeared in the KorroPads of all 5 sheets compared directly after the post-weld treatment. These were predominantly small, indicating metastable corrosion processes that were able to repassivate in the course of the test and indicating that passive layer formation was not yet complete. Humid storage (30 °C/100 %rh) of the same sheet coupons in a climatic chamber left all surfaces free of indications in the KorroPad tests, indicating an improvement in the passive layer stability.

In contrast, when the weld post-treatment time was extended to 20 seconds in a proof test, no indications were visible in the KorroPads immediately after the process (Fig. 22), indicating a more stable passive layer. Moisture storage was therefore not used for this test coupon.

For material 1.4301, KorroPad tests analogous to those performed on material 1.4404 were also carried out in proof tests. Fig. 23 shows the test on a sheet metal coupon after 10 seconds of weld post-treatment time, as well as the KorroPad test results after 15 minutes of testing in each case. Many small indicators can be detected in the KorroPads, indicating metastable corrosion processes. Metastable pitting corrosion is defined by repassivation in the course of the test. Passive layer formation was not yet complete in this case. Humid storage of the same sheet coupon in a climatic chamber resulted in a significant improvement of the passive layer condition and the KorroPads remained free of indications.

The extension of the weld post-treatment time to 20 seconds had a rather detrimental effect on the passive layer formation for the 1.4301 sheet metal coupon (Fig. 24) and produced many and sometimes larger indications in the KorroPads. Here, the passive layer is not intact and only humid storage can level out these imperfections.

6.1 Sheet coupons material 1.4404

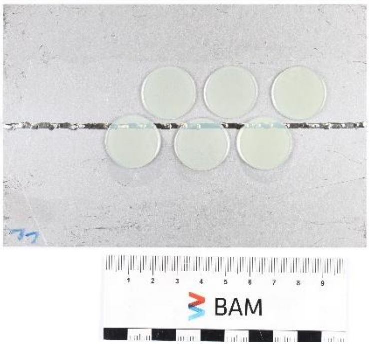
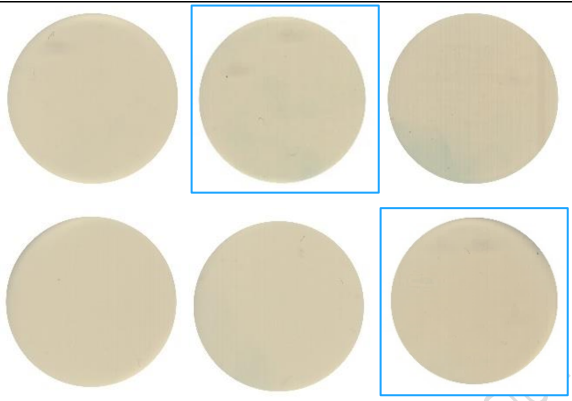
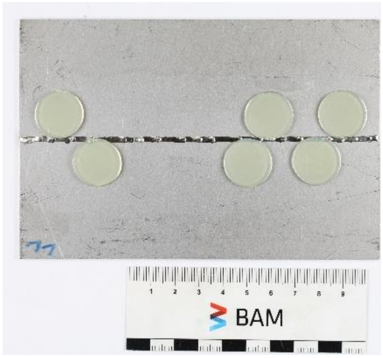
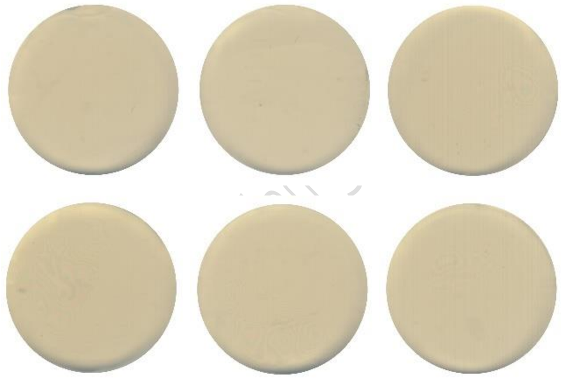
a) directly after SPP 	
b) After 96 h humid storage 	

Fig. 17: KorroPad® test on sheet metal coupon 11 (l.), 1.4404, 10 s weld post-processing; test result after 15 min (r.); KorroPads with indication blue framed

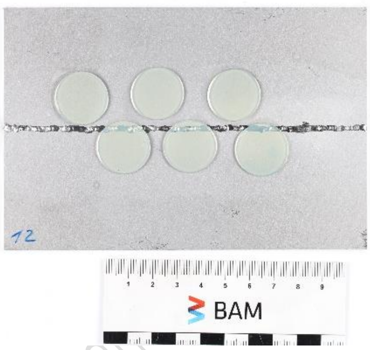
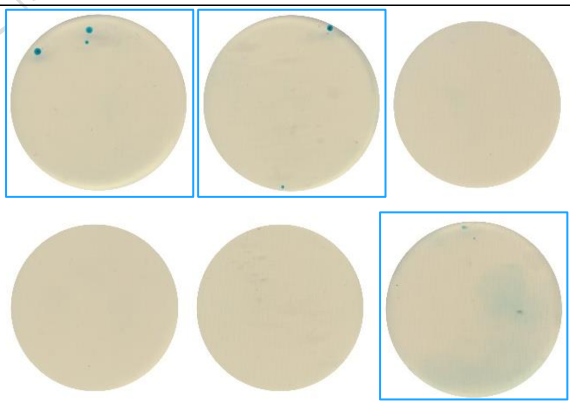
a) directly after SPP 	
b) After 96 h humid storage 	

Fig. 18: KorroPad® test on sheet metal coupon 12 (l.), 1.4404, 10 s weld post-processing; test result after 15 min (r.); KorroPads with display framed in blue

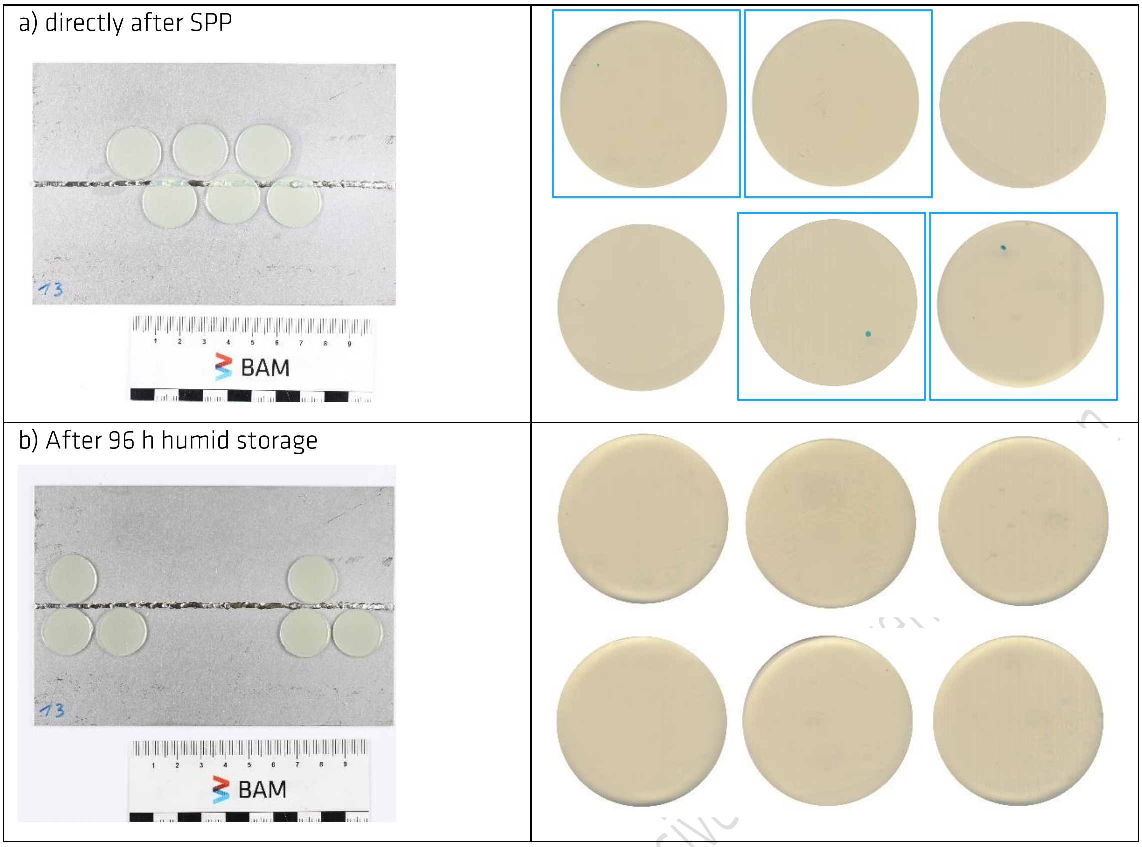


Fig. 19: KorroPad® test on sheet metal coupon 13 (l.), 1.4404, 10 s weld post-processing; test result after 15 min (r.); KorroPads with indication framed in blue

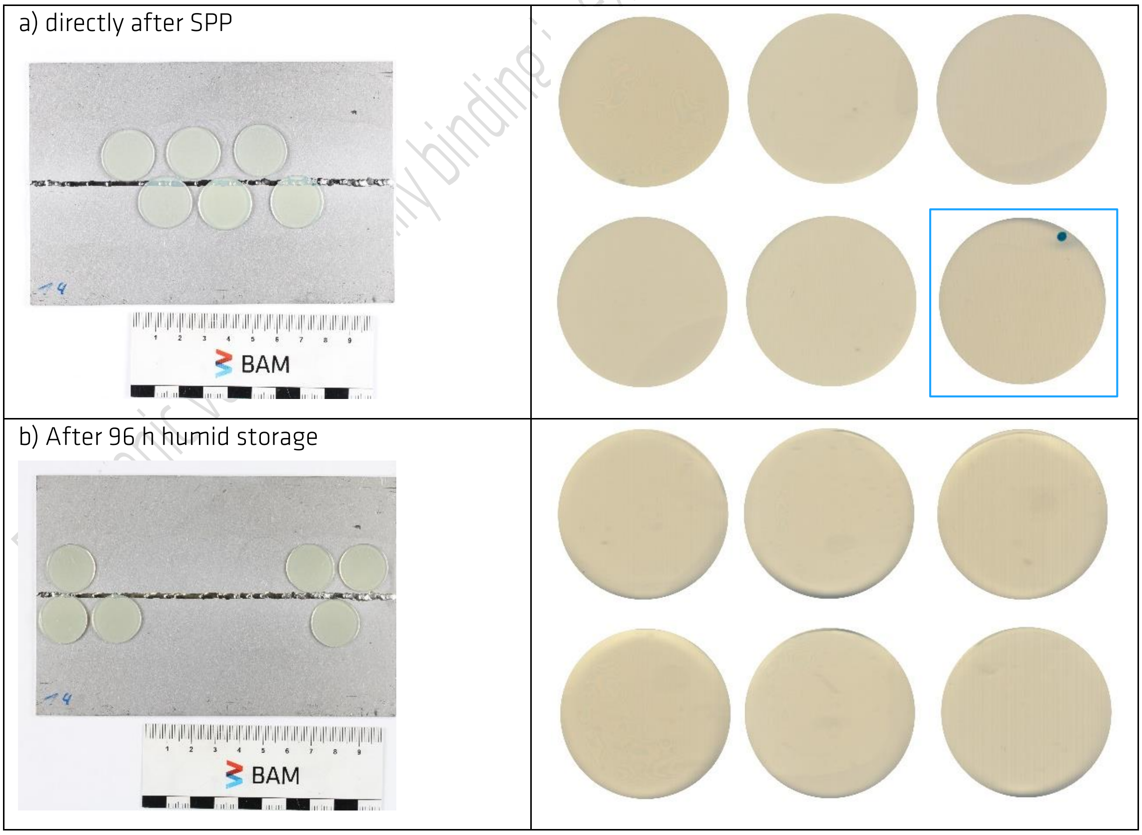


Fig. 20: KorroPad® test on sheet metal coupon 14 (l.), 1.4404, 10 s weld post-processing; test result after 15 min (r.); KorroPads with indication blue framed

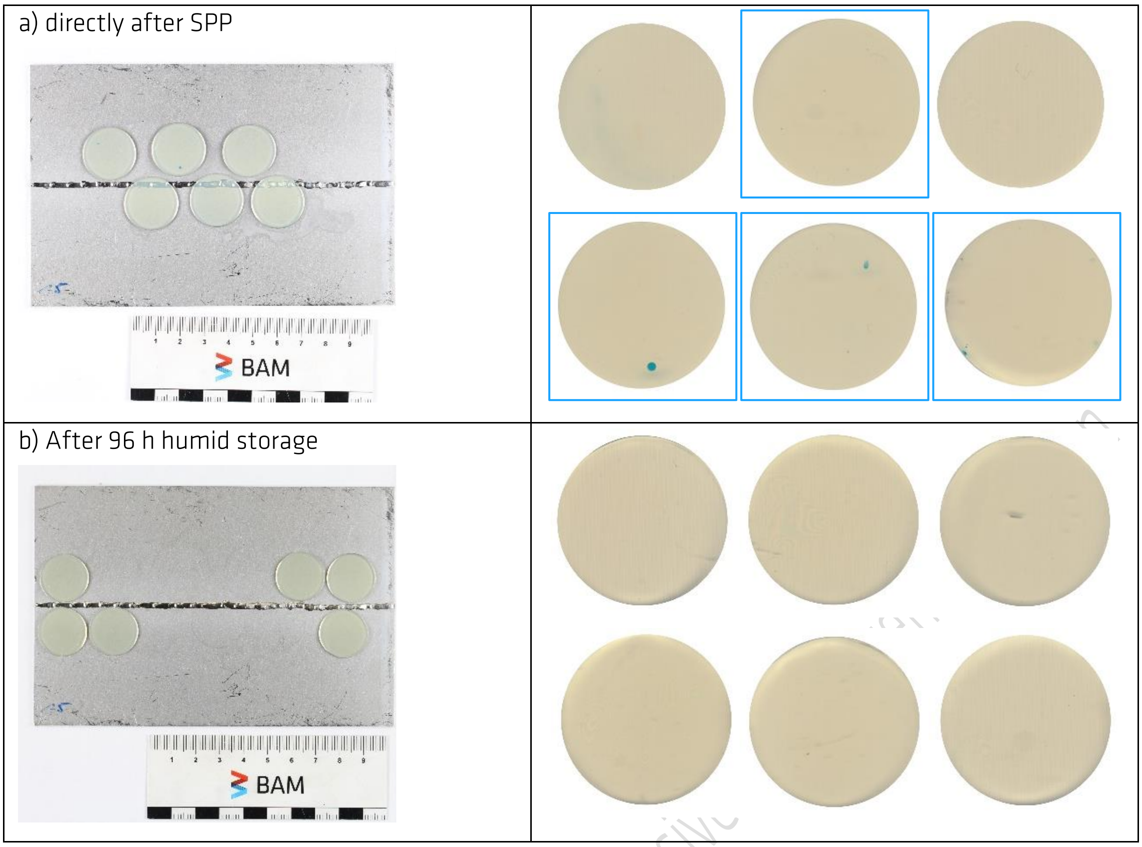


Fig. 21: KorroPad® test on sheet metal coupon 15 (l.), 1.4404, 10 s weld post-processing; test result after 15 min (r.); KorroPads with indication blue framed

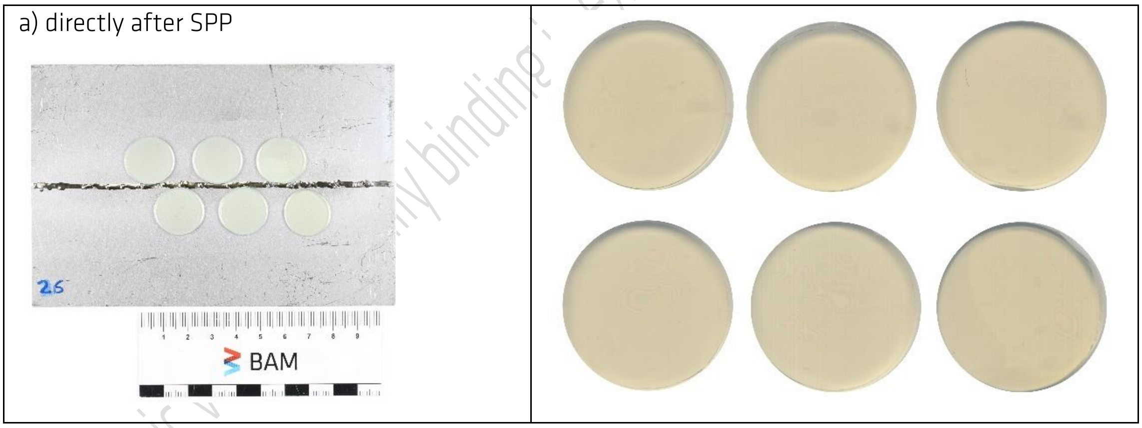
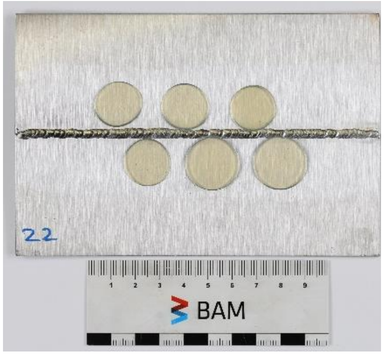
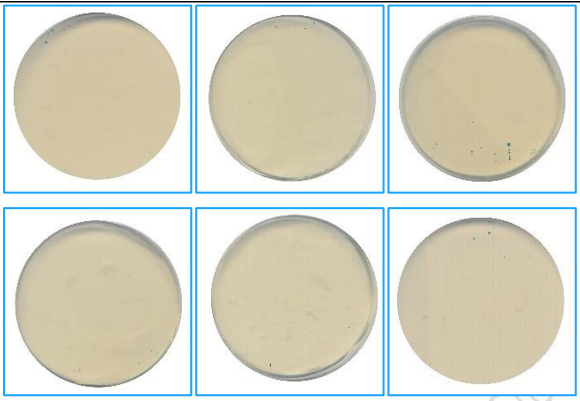


Fig. 22: KorroPad® test on sheet metal coupon 26 (l.), 1.4404, 20 s weld post-processing; test result after 15 min (r.); KorroPads with indication blue framed

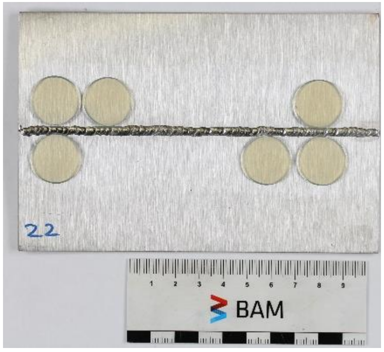
6.2 Sheet coupons material 1.4301

a) directly after SPP





b) After 96 h humid storage



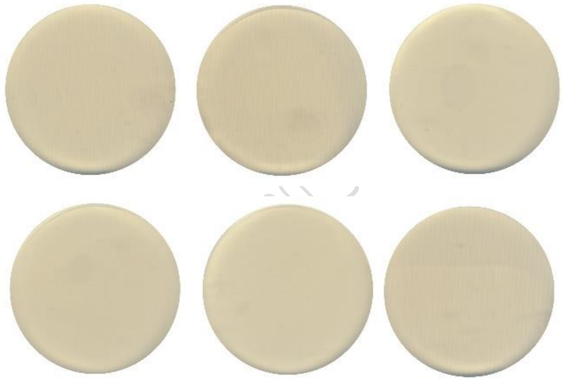
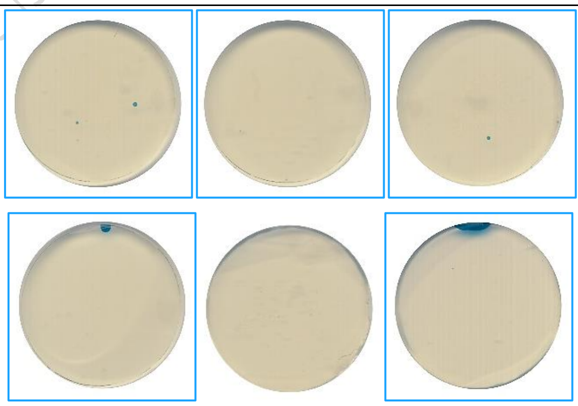
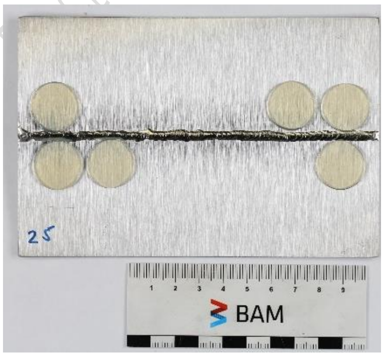


Fig. 23: KorroPad® test on sheet metal coupon 11 (l.), 1.4301, 10 s weld post-processing; test result after 15 min (r.); KorroPads with indication blue framed

a) directly after SPP



b) After 96 h humid storage



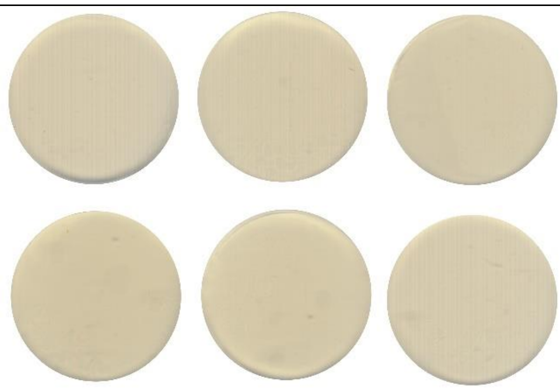


Fig. 24: KorroPad® test on sheet metal coupon 11 (l.), 1.4301, 20 s weld post-processing; test result after 15 min (r.); KorroPads with indication framed in blue

7 Summary

The chemical compositions of the 1.4404 and 1.4301 materials used could be confirmed by a material analysis. The weld cleaning equipment used was set up and used according to the customer's specifications and was intuitive to operate.

The ASTM-A 967 tests according to Practice D and Practice E were passed.

Testing of the effectiveness of the weld post-treatment process concerning the passive film stability of the surface left behind was performed using KorroPad® testing. It was found that the surfaces immediately after weld post-treatment were in an incomplete but acceptable passive film condition. On all test coupons, small indications were visible in the KorroPads in the area of the cleaning zone, which were due to metastable corrosion processes and which were able to repassivate in the course of the test. The exception was the material 1.4404 with a 20-second post-weld treatment. Here, stable passivation could already be demonstrated directly after the post-weld treatment. After the humid storage of the test coupons, all surfaces exhibited a stable passivation state.

Literature

[1] DIN EN 10088-2:2014 Nichtrostende Stähle - Teil 2: Technische Lieferbedingungen für Blech und Band aus korrosionsbeständigen Stählen für allgemeine Verwendung

[2] ASTM A 967/A 967M:2017 Standard Specification for Chemical Passivation Treatments for Stainless Steel Parts

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Division 7.6 Corrosion and Corrosion Protection

By order	By order
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