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D3.3 - Report on erosion-corrosion testing of samples in simulated environment

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Abstract	This deliverable describes work carried out on erosion-corrosion testing of WP2 developed coatings in simulated geothermal drilling environment.	

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PU – Public (e.g. on website, for publication etc.) / **PP** – Restricted to other programme participants (incl. Commission services) / **RE** – Restricted to a group specified by the consortium (incl. Commission services) / **CO** – confidential, only for members of the consortium (incl. Commission services)

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Summary

This deliverable describes work carried out on erosion-corrosion testing of WP2 developed coatings in simulated geothermal drilling environment, including:

- Design of an erosion-corrosion testing facility using main elements of the slurry pot equipment (ASTM G119), with in-situ electrochemical measurement capabilities.
- Define formulation of simulated geothermal drilling fluid to be used in erosion-corrosion testing.
- Preliminary erosion-corrosion testing at 24h duration in simulated drilling fluid on a total number of sixteen (16) samples, including three types of steels for benchmarking and thirteen (13) samples deposited with coatings developed in WP2. Among those, six (6) samples including Steel 1, 222Xylan/GO1, 232WC1, 234Flux1, Steel 2, and 232WC2 were down-selected for further testing.
- Further erosion-corrosion testing was carried out for 7 days to evaluate how down-selected samples perform in simulated drilling environment for longer-term. From the results, samples 222Xylan/GO1 and 234Flux1 are proved to have good protection to Steel 1 in the selected testing condition. Steel 2 performs very well itself and is recommended not to have the need to be coated in similar drilling environment.
- Ranking and recommendations from this deliverable report is based on results from erosion-corrosion testing in simulated geothermal drilling environment only. Final decisions on material ranking should be based on overall performance of all environmental testing in WP3, which will be put together in D3.5.

Objectives Met

The work described in this report contributes to the following objectives:

- To design an erosion-corrosion testing facility with in-situ electrochemical testing capability and to define drilling fluid formulation,
- To carry out erosion-corrosion testing of samples received from WP2 development in simulated drilling environment,

CONTENTS

1) INTRODUCTION

Erosion-corrosion is one of the major causes of material degradation in geothermal exploration, i.e. within pump and pipeline components that handles erosive slurry at high flow rate. This can be affected by a number of factors such as part material type, servicing environment and type of abrasive particles. Alkaline drilling fluid is normally used to maintain the stability and efficiency of the drilling process when the operating environment is harsh. The fluid also carries the rock debris produced during the drilling process, which then becomes slurry, causing erosion-corrosion behaviour. Erosion-corrosion always combines the action of mechanical wear and chemical corrosion. It can cause low mechanical efficiency, short component lifetime, and seriously affects the stability and safe operation of the drilling system [1]. In order to combat such degradation, applying surface coatings can provide protection against erosive particulates within the slurry [2]. Therefore, drilling equipment has a great demand for coatings having excellent erosion-corrosion resistance.

This deliverable report focuses on carrying out erosion-corrosion testing in a simulated geothermal drilling environment. All developed coatings in WP2 including PTFE/GO, high velocity oxygen fuel (HVOF) sprayed alloy and cermet and electroless Ni/PTFE were deposited onto steel substrates and results are compared with selected benchmarking materials to provide ranking and recommendations with regard to their erosion-corrosion performance.

2) APPROACH

2.1 Materials

2.1.1 Selection of benchmarking materials

The selection of benchmarking materials is based on a preliminary study on currently used hammer parts, which was carried out at the early stage of the project. This has been reported in detail in Geo-Drill PR1 (M18) report. Specification and nominal compositions of selected steel substrates are given in Table 1. From preliminary study, it was found that some steels used in geothermal drilling components present high hardness (Table 7 in PR1 M18 report). Therefore, quenching and tempering were applied on both 440B and 835M30 substrates to improve their hardness from their annealed condition before applying coatings.

2.1.2 Tested samples

Geometry of the test coupon is shown in Figure 1. It is designed to be fixed onto the sample holder to allow insulation for carrying out in-situ electrochemical measurement during the testing. All test coupon substrates including benchmarking steel substrates were machined to the specified dimension. Steel substrates 440B and 835M30 were then quenched and tempered to improve their hardness. Hardness evaluation on three steel substrates was done under 300g using a Duramin Vickers hardness tester in accordance with BS EN ISO 6507-1:2005. The hardness value was expressed as an average value with a standard deviation in Table 2.

All three steel substrate materials were coated with materials developed in WP2, including PTFE coatings, HVOF coatings and electroless Ni/PTFE coatings for Steel 1, selected HVOF coatings for Steel 2 and Steel 3. A full list of samples tested in this report is detailed in Table 3. All the coated samples were tested in their as-prepared condition. All three steel substrates used as benchmarking materials were grounded using 1200 grit SiC sand paper and were then being cleaned with alcohol to remove residual impurities before testing.

2.2 Testing

The testing was carried out using TWI's designed erosion-corrosion facility using main elements of the slurry pot equipment based on ASTM G119. Design of the facility is detailed in Section 3.1. Maximum of six samples

could be fixed onto the erosion-corrosion testing facility. The back face and edge of the test coupons were masked with epoxy resin. A nitride rubber O-ring against the counter bored sample well in the wall of the carousel was used to insulate the sample from the disk and from each other, with an exposing area of 11.36cm^2. The sample holder was spun at 120 rpm in a 30L mixture of simulated drilling fluid made of Clearbore, water, NaCl and 15wt.% sand (detailed in Section 3.2). The rotational speed, in RPM, was monitored at regular intervals by an Extech Digital Photo Tachometer. The sample surface was exposed to various impact angles due to cylindrical shaped flow.

In-situ electrochemical measurement was carrying out during the testing. Open circuit potential (OCP) and linear polarisation resistance (LPR) values against Sentek calomel reference electrodes via 3mm DIA 10000mm long gel-filled, chloride-doped polymers salt bridges were recorded during the testing. Tafel scan measurement was also carried out for each sample in simulated drilling fluid to define the anodic and cathodic Tafel parameters(Ba and Bc values) for calculating corrosion rate (mm/year).

Two rounds of testing were designed to study erosion-corrosion performance of developed materials in simulated drilling environment. The 1st round of testing was carried out for 24h. Results were then reviewed to down select six samples. The 2nd round of testing was then carried out for 7 days for these six down-selected samples to study their longer-term performance. Both rounds of testing were carried out under ambient temperature and pressure. Material performance at elevated temperature and pressures was evaluated in T3.2 and will be reported in D3.2 by University of Iceland.

2.3 Characterisation

Preliminary evaluation of the test with selected benching marking materials showed that material mass loss from the defined condition was very low and could not be measured using analytical balance of 0.0001g precision. Therefore, it is not recorded in this report. The erosion-corrosion performance of developed coatings systems on various steel substrates in simulated geothermal drilling environment was evaluated using a combination of the following characterisation methods:

2.3.1 Digital images

All test coupons were recorded using digital camera before and after testing to compare their difference.

2.3.2 Microstructure characterisation

Microstructural analysis was performed using scanning electron microscopy (SEM) equipped with energy dispersive X-ray spectroscopy (EDX) to check whether there was any corrosion products produced at both top surface, in the coating and at the coating-substrate interface. Inspections were also carried out to check whether there any delamination was occurred due to corrosion or whether there any scratches were formed on test coupon surface due to erosion. Images were taken mainly in backscattered mode to obtain high-resolution images that show the distribution of various elements in a sample. EDX spectroscopy was employed while imaging on SEM to obtain the elemental compositions at different areas of the sample cross-sections.

For cross-sectioned test coupons, metallography samples were prepared after the specimens were tested using cold-mounting. Mounted samples were ground using successive silicon carbide papers 600-1200-2500 grit. This was followed by polishing using diamond pastes: 3µm then 1µm.

2.3.3 Electrochemical measurement

Electrochemical measurements were carrying out during erosion-corrosion testing to determine the contributing factors of erosion, corrosion and the synergistic effects of both to the performance of test coupons.

Recordings of OCP and LPR measurements were take each 30min interval from the beginning until end of the testing. LPR can give indication of the corrosion resistance of materials in an aqueous environment. It consists of applying small voltage variations to the sample above and below its corrosion potential. Over this narrow range in the vicinity of the corrosion potential the current response obtained is linear. The polarisation resistance is defined as the slop of this current-potential curve [3].

Tafel scan measurement for each sample was carried out separately in static simulated drilling fluid to define the anodic and cathodic Tafel parameters(Ba and Bc values) to be used together with LPR readings to calculate corrosion rate (mm/year) of the sample.

3) RESULTS AND DISCUSSION

3.1 Design of erosion-corrosion testing facility

As part of this deliverable, TWI has developed a design for an erosion-corrosion test facility, using main elements of the slurry pot equipment (ASTM G119). The design has been developed to allow in-situ electrochemical measurements through the inclusion of reference and counter electrodes, and a potentiostat electronic instrument. A diagram in Figure 2 shows the design of the system.

In the design, a 200mm diameter sample holder carousel assembly can spin at 120 rpm controlled by a 24V DC motor mounted underneath the cell. The sample holder can hold up to six specimens each time, which will be submerged in the test fluid during testing. Copper leads (6-off in total) take the signal through the inside of the upper shaft to a 6-way slip ring mounted on the lid of the test cell that allowed connections to the ACM potentiostat. Slip ring can convert rotating electrical wire connections to static electrical wires connections. A photograph showing final set-up of the facility is illustrate in Figure 3, including testing cell, ACM potentiostat, and central control station.

3.2 Define of testing fluid

Alongside facility construction, TWI and Geolorn have developed the simulation slurry for the erosion-corrosion testing. Geolorn established the initial composition of the slurry using their knowledge of industrial practice. TWI then worked with suppliers to determine suitable commercial, lab scale products, which could be used to develop a simulation fluid. The simulated slurry formulation was then validated using a Marsh Funnel with a target value of 60 seconds. The slurry formulation was initially defined as below, as has been reported in Geo-Drill PR1 report:

- Liquid
  - Clear Bore Polymer diluted with DI water to obtain a Marsh Funnel Viscosity of 60 seconds. - 3.7g/L of Clearbore
  - Sodium carbonate added to obtain pH 9.
- Aggregate
  - 50:50 mix of gravel:sand, 20% w/w of slurry.
  - Gravel – 2-3mm
  - Sand – 0.85-1.7mm

After a few trials when the testing facility was assembled, the above formulation did not seem to work well. Two main problems were noticed:

- Particle size of selected gravel is too big. Gravel particles were sediment at the bottom of the pot and could not flow well during the trial.

- The fluid is not conductive enough to conduct effective electrochemical measurement.

Therefore, the formulation was reviewed again in order to maintain a good flowability of 60 seconds using a Marsh Funnel, homogeneous slurry flow during testing and good conductivity for electrochemical measurement. The final slurry formulation is presented below for carrying out erosion-corrosion testing for all received test coupons.

- Liquid
  - Clear Bore Polymer diluted with DI water to obtain a Marsh Funnel Viscosity of 60 seconds. - 3.7g/L of Clearbore
  - Sodium carbonate added to obtain pH 9.
- Aggregate
  - Sand 0.85-1.7mm, 15% w/w of slurry.
- Salt
  - Sodium Chloride (NaCl), 1.0(w/v)%

SEM and EDX analyse carried out on sand particles indicates that its particle size is in the same range with information provided by the supplier. It is mainly composed of silicon oxide, with small amount of alumina (Figure 4).

3.3 The 1st round – 24h testing

The 1st round testing was carried out for 24h at room temperature in simulated drilling environment. The coating systems involves three main categories: Xylan/GO coatings, HVOF hardfacing and alloy coatings and electroless plated Ni coatings. They were designed and developed in WP2 to provide either low friction performance or improved wear resistance or a combination of the two. This deliverable only focuses on results and discussions on their erosion-corrosion performance in simulated geothermal drilling environment.

3.3.1 Coatings on Steel 1

Steel 1 is a low alloy steel in annealed condition that is representative for a lot hammer parts such as piston bushing, sliding case, valve house etc. It was selected as the main steel substrate that Geo-Drill is focusing on. All coatings systems developed in WP2 were deposited onto this substrate. Digital images of these test coupons before and after testing are shown from Figure 5 to Figure 14. SEM and EDX analyse on their cross-sectioned surface are presented from Figure 15 to Figure 24. Corrosion rate changes vs time is shown in Figure 25.

1) Steel 1

After 24h erosion-corrosion testing in simulated geothermal drilling environment, severe corrosion happened on Steel 1 (Figure 5, Figure 15). About two thirds of the surface area is covered by a thicker and dense corrosion layer, which is about 15µm. Corrosion rate measurement shows that corrosion rate of Steel 1 was very low at the first 1.5hrs, which then dramatically increased to 0.560mm/yr after 2hrs. Corrosion rate seemed to get stabilised at 0.700mm/yr after about 18hrs.

2) Xylan/GO coating systems

After 24h erosion-corrosion testing in simulated geothermal drilling environment, no sign of corrosion product is observed on the surface for both 221Xylan and 222Xylan/GO samples (Figure 6, Figure 7). Results from SEM and EDX analyse showed that no iron oxide is found on the sample surface or in the coating (Figure 16, Figure 17). It is also noted that Xylan coating has a more porous microstructure after addition of graphene oxide (GO) particles, but there does not cause any obvious difference on their erosion-corrosion performance in the defined testing condition. Both 221Xylan and 222Xylan/GO coatings show the lowest corrosion rate compared with other samples (Figure 25). It should be noted that Xylan is not conductive material and the electrochemical

measurement may not be accurate for such sample. Nevertheless, microstructure analyse is enough to prove that both samples have protected Steel 1 very well during the 24h testing in simulated geothermal drilling environment.

3) HVOF coating systems

The tested HVOF coating systems include both cermet coatings such as WC-CoCr, CrC-NiCr, WC-CrC-Ni and alloy coatings such as self-fluxing Ni alloy, amorphous Fe alloy, high entropy alloy (HEA) coatings. These coatings performed very differently in erosion-corrosion testing in simulated geothermal drilling environment.

After 24h erosion-corrosion testing, their digital images show that there is hardly any corrosion product can be observed on the surface of samples 232WC1, 234Flux1 (Figure 8, Figure 10). There is very little amount of corrosion product presented on the surface of samples 233CrC1 and 283HEA1 (Figure 9, Figure 14). It seems that Samples 235Amor1 and 282WCr1 had severe corrosion during testing (Figure 11, Figure 13). When the sample is not corroded on the surface, some minor scratch caused by erosion can be observed on its surface, such as sample 233CrC (Figure 9). However, this is not seen on test coupons with harder coatings, such as 232WC1 (Figure 8).

In order to further study how these samples performed under erosion-corrosion and whether there was any delamination between coating-substrate interface after 24h testing, metallography samples were prepared for microstructural and elemental analysis. From SEM and EDX analyses, there is no strong sign of iron oxide observed either in the coating or at the coating-substrate interface for samples 232WC1, 233CrC1, 234Flux1 and 283HEA1 (Figure 18, Figure 19, Figure 20, Figure 24). A corrosion layer of about 20µm is observed on the top surface of sample 235Amor1, moreover, its coating is found to be entirely delaminated from the substrate (Figure 21). Some corrosion product was also found on the top surface of sample 282WCr1. EDX analyse on its coating-substrate interface shows strong peak of oxide (O) (Figure 23). This indicates that corrosive media had penetrated onto the substrate during this 24h testing and caused corrosion, which could eventually lead to coating delamination.

From corrosion rate measurement, all the HVOF coatings have considerably lower corrosion rate compared with Steel 1 under erosion-corrosion conditions in simulated geothermal drilling environment except sample 232WC1 (Figure 25). Among these, sample 234Flux1 has the lowest corrosion rate. Though corrosion rate for sample 232WC1 is not very stable and is of comparable level with Steel 1, it can be proved from SEM and EDX result that WC-CoCr coating can protect the substrate well during this 24h erosion-corrosion testing. One of the reasons may be ascribed to that WC-CoCr coating are not purely conductive material compared with alloy coatings, which can affect accuracy of electrochemical measurement. Further testing need to be done in the future to explore this.

In overall, samples 232WC1 and 234Flux1 have superior performance against erosion-corrosion in simulated geothermal drilling environment. Samples 232CrC1 and 283HEA1 can effectively protect the steel substrate in the 24h testing duration but some sign of corrosion was observed on their sample surface. Coating failure happened for sample 235Amor1 and heavy corrosion was noticed for sample 282WCr1, therefore, both coatings are not recommended for the application in the simulated drilling environment.

4) Electroless Ni/PTFE system

One electroless plated coating 271Ni that has the best performance from various parameter combinations in WP2 was selected and was tested in this report. After 24h erosion-corrosion testing, some minor sign of scratch from erosion was observed and there is hardly any corrosion product can be seen on its surface (Figure 12). SEM and EDX analyse on its cross-sectioned surface also indicates that both top Ni/PTFE and Ni interlayer

adhere well with each other and no sign of corrosion is found in the coating (Figure 22). Corrosion rate measurement indicates that sample 271Ni had a slightly higher corrosion rate of about 0.200mm/year at the beginning of the test. This was then gradually decreased to 0.050mm/year during the testing. In overall, the selected electroless Ni/PTFE test coupon can effectively protect Steel 1 in the simulated geothermal drilling condition though some minor scratch is found on its surface (Figure 25).

3.3.2 Coatings on Steel 2

Steel 2 is a stainless steel being heat-treated, with a final hardness of over 600$HV_{0.3}$. From preliminary results of coatings tested on Steel 1 and material properties previously reported in D2.4 and D2.8, coating WC-CoCr (232WC) can offer higher hardness than Steel 2 and self-fluxing Ni alloy (234Flux) has good corrosion-resistance with good toughness, they were selected to be coated and tested on Steel 2.

Digital images were taken before and after the 24h erosion-corrosion testing, from which it can be seen that both Steel 2 and 234Flux2 have no sign of damage from either corrosion or erosion (Figure 26, Figure 28). However, some corrosion product is found on the surface of sample 232WC2 (Figure 27). SEM and EDX analyse on their cross-sectioned surface was then carried out to further check their erosion-corrosion performance. No sign of corrosion is noticed for Steel 2 and there is also not any iron oxide is found in the coating and at coating-substrate interface for both samples 232WC2 and 234Flux2 (Figure 29-Figure 31). Corrosion rate measurement shows that Steel 2 has the lowest corrosion rate among all three samples. Sample 234Flux2 has slightly higher corrosion rate than Steel 2 and Sample 232WC2 has the highest corrosion rate (Figure 32).

Therefore, it can be concluded from results above that Steel 2 has very good performance against erosion-corrosion and both types of HVOF coatings selected do not provide any improved performance of this substrate when served in simulated geothermal drilling environment. However, this does not exclude that other coating technology may still have potential to improve its performance.

3.3.3 Coatings on Steel 3

Steel 3 is a low alloy steel type being heat-treated, with a final hardness of over 500$HV_{0.3}$. From preliminary results of coatings tested on Steel 1 and material properties previously reported in D2.4 and D2.8, WC-CoCr and WC-Ni HVOF coatings were selected to be applied on this substrate due to their high hardness, which are expected to be able to provide sufficient protection to Steel 3 against erosion and corrosion.

After 24h testing in simulated geothermal drilling environment, it can be seen from digital images that severe pitting and corrosion happened on the surface of Steel 3 (Figure 33). There is very few corrosion product on the surface of sample 232WC3 and nothing on the surface of sample 281WC3 (Figure 34, Figure 35). SEM and EDX analyse on the cross-sectioned surface of Steel 3 shows that the substrate surface is covered by a dense layer of iron oxide (Figure 36). Some brittle damage is found near the top surface of sample 232WC3 and EDX spectrum indicates appearance of iron oxide at its top surface. However, there is no corrosion being found in the coating or at the coating-substrate interface (Figure 37). As WC-CoCr coating has high hardness of around 1100$HV_{0.3}$, such brittle damage should not come from erosion. This is most likely to be caused during the metallography preparation due to brittleness of such coating. Such damage would speed up penetration of corrosion substances into the substrate. Sample 281WC3 presented a uniform coating, with no sign of corrosion or brittle damage being observed on its cross-sectioned surface (Figure 38). The main difference between these two WC carbide coatings is that Sample 281WC3 uses Ni as binder while 232WC3 uses Co-Cr as binding phase. Ni can provide better toughness than Co-Cr, which, therefore, offer better coating integrity from metallography preparation. Corrosion rate measurement shows that 232WC3 has lower corrosion rate than Steel 3 though

some brittle damage from metallography preparation is observed on its surface after 24h testing (Figure 39). Sample 281WC3 has higher corrosion rate than Steel 3, however, there is no corrosion product formed as proved from microstructural and chemical analyse.

Therefore, it can be concluded that Steel 3 is not resistant to erosion-corrosion attack and both coatings could be applied for protection in similar geothermal drilling environment. However, WC-CoCr coating (sample 232WC3) has very high hardness but is a brittle material, special attention should be paid if any post treatment is required for applications against corrosion to avoid any coating damage.

3.4 The 2nd round – 7day testing

From results obtained in the 1st round of erosion-corrosion testing and preliminary testing results (pull-off test, slurry test, pin-on-disc test) shared by partners in T3.1 (to be reported in D3.1), six samples were selected to be tested for 7-day testing to study how they perform under longer time of erosion-corrosion testing in simulated drilling environment, including:

a) Steel 1: low alloy steel benchmarking material, similar erosion-corrosion behaviour as Steel 3
b) 222Xylan/GO1: good erosion-corrosion performance in 1st round testing and low friction coefficient
c) 232WC1: good erosion-corrosion performance in 1st round testing and high adhesion strength
d) 234Flux1: good erosion-corrosion performance in 1st round testing and cost-effective
e) Steel 2: stainless steel benchmarking material
f) 232WC2: effective erosion-corrosion performance in 1st round testing

Detailed information of these samples can be found in Table 4. Digital images of those samples before and after testing are shown in Figure 40-Figure 45. Their SEM and EDX analyse results on top surface are presented from Figure 46 to Figure 51 and on polished cross-sectioned images are presented from Figure 52 to Figure 57. Corrosion rate changes versus testing time is shown in Figure 58.

a) Steel 1

After 7-day testing in simulated drilling environment, Steel 1 has a thick layer of corrosion product on its surface (Figure 40). SEM and EDX analyse confirms that its surface is mainly covered by iron oxide and some areas is covered with adhered substances from the simulated drilling fluid such as silicon oxide. Iron oxide layer formed due to corrosion has a very porous top surface and no sign of scratch from erosion on the surface is observed (Figure 46). This possibly indicates that corrosion takes more predominant role in the selected erosion-corrosion testing condition for Steel 1 and formation of corrosion product is faster than generation of scratch if any. SEM and EDX analyse on its cross-sectioned surface indicates that the formed corrosion layer is about 15µm thick (Figure 52). Though the corrosion layer formed during the 7-day testing is of similar thickness as when compared with the one being tested under 24h, it is much denser (Figure 15). From its corrosion rate measurement, Steel 1 has increased corrosion rate up to about 1.00mm/year at the first 5h and then gradually decreases and becomes stabilised (Figure 58).

b) 222Xylan/GO1

After 7-day testing in simulated drilling environment, there is no any obvious damage can be observed on the surface of sample 222Xylan/GO1 (Figure 41). SEM and EDX analyse on its top surface presents some very fine adhered sand particles that are uniformly distributed across its surface, but there is no sign of corrosion product or any scratch from erosion-corrosion can be observed (Figure 47). From SEM and EDX analyse on its cross-sectioned surface, some alumina oxide grits indicated the surface was previously grit-blasted for improved adhesion strength of the coating but no sign of formation of iron oxide can be found (Figure 53). Though corrosion rate for this sample may not be accurate due to its non-conductivity, above analyse results can prove

that Xylan/GO coating protected the substrate very well during the 7-day erosion-corrosion testing in simulated drilling environment.

c) 232WC1

After 7-day testing in simulated drilling environment, the surface of sample 232WC1 is fully covered by corrosion product (Figure 42). Some sand particles from testing fluid are found adhered on its surface from SEM and EDX analyse. The sample is mainly covered by porous iron oxide and no sign of scratch is seen on its surface (Figure 48). SEM and EDX analyse on its cross-sectioned surface confirms that its corrosion layer built-up is about 50µm, but no appearance of iron oxide is detected in the coating. EDX analyse carried out at coating-substrate interface shows strong peaks of iron and oxide. This confirms that corrosive liquid had penetrated onto the substrate and caused corrosion during the 7-day testing. Corrosion rate measurement indicates that corrosion rate of this sample was seldom stabilised during the 7-day testing duration (Figure 58). Corrosion rate increased the fastest at the 10 hours, then gradually increased to maximum corrosion rate of about 6.250mm/year until 135 hours and started to decrease afterwards until testing finishes.

It seems that sample 232WC1 had more severe corrosion than Steel 1 during the 7-day testing, though it protected the substrate well in the 24h testing. It is assumed that one of the reasons can be ascribed to the porous nature of HVOF coatings, which could enable penetration of corrosive liquid onto the substrate for longer-term testing. The penetrated liquid may cause a high concentrated corrosive environment between coating and substrate, which then accelerate corrosive reactions. For industrial applications, sealant is normally applied onto as-deposited thermal sprayed coatings if used in corrosive environment to fill their pores to prevent or retard corrosion. As this WC-CoCr coating offers very high hardness and low porosity, it is recommended that further study to be done on this to explore how sealant can improve its performance in corrosive environment.

d) 234Flux1

After 7-day testing in simulated drilling environment, self-fluxing Ni alloy coating (234Flux1) seems to protest the substrate well with only little corrosion product can be seen on its surface (Figure 43). SEM and EDX analyse on the top surface shows that some adhered liquid substances such as sand was scattered across the sample surface, but no sign of scratch from erosion can be found (Figure 49). Further examination on its polished cross-sectioned surface indicates that some iron oxide was formed at the coating top surface during testing but no corrosion product is found in the coating (Figure 55). EDX analyse at the coating-substrate interface shows a strong peak for oxygen, indicating that corrosive liquid had penetrated onto the substrate and some corrosion reaction at the interface had happened. However, the coating integrity still seems good (Figure 55). Corrosion rate measurement shows that sample 234Flux1 was quite stable during the 7 days and its value is always lower than Steel 1. Therefore, the selected self-fluxing Ni alloy coating could provide effective protection on Steel 1 in simulated drilling environment. But attention should be paid if it is used for longer-term protect in its as-deposited condition.

e) Steel 2

After 7-day testing in simulated drilling environment, Steel 2 had no corrosion or scratch formed from all analyse on both its surface and cross-sectioned surface (Figure 44, Figure 50, Figure 56). Its corrosion rate was the lowest during the testing duration (Figure 58). It seems that Steel 2 can resist erosion-corrosion very well for the simulated drilling environment.

f) 232WC2

After 7-day testing in simulated drilling environment, some corrosion product was formed on its surface (Figure 45). SEM and EDX analyse on its top surface and cross-sectioned surface shows that iron oxide was found both

on its top surface and at coating-substrate interface, but not in the coating (Figure 51, Figure 57). This means that corrosive liquid had penetrated onto the substrate. Similarly with 232WC1, the formation of high concentrated corrosive environment at the coating-substrate interface accelerated corrosion of the substrate. Therefore, this sample could not resist erosion-corrosion as well as Steel 2 when there is not coating applied. Corrosion rate measurement also confirms that 232WC2 has higher level of corrosion rate than substrate material Steel 2 (Figure 58).

From results obtained in 7-day testing, it can be concluded that samples 222Xylan/GO1 and 234Flux1 can rovide good protection to the substrate material in a similar drilling environment. Though Sample 232WC1 received in the as-deposited condition does not have enough protection to the substrate in the selected environment, this may be improved when an effective sealant layer being applied on its surface to prevent penetration of corrosive media. Steel 2 survives well in the simulated drilling fluid and it is not necessary to apply any coating on it if to be serviced in a similar environment.

4) CONCLUSIONS

This report focuses on carrying out erosion-corrosion testing for WP2 developed coatings in simulated geothermal drilling environment using designed slurry pot equipment. Main conclusions of the report include the following:

- An erosion-corrosion testing facility was designed using main elements of the slurry pot equipment (ASTM G119), which allows in-situ electrochemical measurement.
- Simulated geothermal drilling fluid for carrying out erosion-corrosion testing was defined, which comprises a mixture of clearbore, water, sand and sodium chloride, with a pH value of about 9 adjusted using sodium carbonate.
- In the 24h erosion-corrosion testing, a total number of sixteen (16) samples were tested including: three (3) substrate materials for benchmarking and thirteen (13) coated samples. Among those, six (6) samples were down-selected including Steel 1, 222Xylan/GO, 232WC1, 234Flux1, Steel 2, and 232WC2.
- From 7-day erosion-corrosion testing, samples 222Xylan/GO and 234Flux1 are proved to have good protection for Steel 1. Steel 2 performs very well itself and is recommended not to have the need to be coated in the simulated geothermal drilling environment.
- Ranking and recommendations from this deliverable report is based on results from erosion-corrosion testing in simulated geothermal drilling environment only. Final decisions on material ranking should be based on their overall performance from all environmental testing in WP3, which will be put together in D3.5.

REFERENCES

[1] Fei Huang, Jia-jie Kang, Wen Yue, Xiao-bin Liu, Zhi-qiang Fu, Li-na Zhu, Ding-shun She, Guo-zheng Ma, Hai-dou Wang, Jian Liang, Wei Weng, Cheng-biao Wang. Effect of heat treatment on erosion–corrosion of Fe-based amorphous alloy coating under slurry impingement. Journal of Alloys and Compounds. 820 (2020) 153132,

[2] Tom Peat, Alexander M. Galloway, Athanasios I. Toumpis, David Harvey. Evaluation of the synergistic erosion-corrosion behaviour of HVOF thermal spray coatings, Surface and Coatings Technology. 299 (2016) 37-48.

[3] F. Ropital. 15 - Environmental degradation in hydrocarbon fuel processing plant: issues and mitigation. Editor(s): M. Rashid Khan, In Woodhead Publishing Series in Energy, Advances in Clean Hydrocarbon Fuel Processing, Woodhead Publishing. (2011) 437-462.

LIST OF TABLES

Table 1 Specifications and nominal compositions of benchmarking materials

ID	Steels	Composition
Steel 1	34CrNiMo6 (EN24T)	C 0.430, Si 0.310, Mn 0.590, P 0.009, S 0.033, Cr 1.200, Mo 0.220, Ni 1.400, Pb 0.0007, Cu 0.200, Sn 0.014, Ca 0.0029, N 0.012, Nb 0.005, Ti 0.0048, V 0.010
Steel 2	440B	C 0.83, Si 0.43, Mn 0.40, P 0.029, S 0.001, Cr 0.001, Cr 16.7, Mo 0.59
Steel 3	835M30 (EN 30B)	C 0.310, Si 0.260, Mn 0.550, P 0.008, S 0.0047, Cr 1.190, Mo 0.330, Ni 4.100, Cu 0.150, Sn 0.008,Al 0.024, Ti 0.0014, N 0.006, Ca 0.005, V 0.003

Table 2 Micro-hardness values of three steel substrates

Micro-hardness	Steel 1	Steel 2	Steel 3
Average, $HV_{0.3}$	341.3	657.7	515.5
Deviation, $HV_{0.3}$	11.6	18.3	1.9

Table 3 List of samples received from partners for erosion-corrosion testing

Number	ID	Coating materials	Substrates	Number	Supplier
1	Steel 1	-	34CrNiMo6	6	TWI
2	221Xylan	PTFE	34CrNiMo6	8	GRAPHENEA
3	222Xylan/GO	PTFE/Graphene oxide	34CrNiMo6	8	GRAPHENEA
4	232WC1	WC10Co4Cr	34CrNiMo6	8	TWI
5	233CrC1	75CrC-25NiCr	34CrNiMo6	8	TWI
6	234Flux1	NiCrFeBSi	34CrNiMo6	8	TWI
7	235Amor1	Amorphous/Nano-crystalline C3Cr25B5Mo20Mn5Si2W10Fe balance	34CrNiMo6	8	TWI
8	271Ni1	Electroless Ni/PTFE, HP/LP+PTFE	34CrNiMo6	8	TWI
9	282WCr1	WC-CrC-Ni	34CrNiMo6	8	TWI
10	283HEA1	CoCrFeNiMo0.85 (HEA)	34CrNiMo6	8	TWI
11	Steel 2		440B	6	TWI
12	232WC2	WC10Co4Cr	440B	8	TWI
13	234Flux2	NiCrFeBSi	440B	8	TWI
14	Steel 3		835M30	8	TWI
15	232WC3	WC10Co4Cr	835M30	8	TWI
16	281WC	90WC10Ni	835M30	4	TWI

Table 4 List of samples down selected for 7-day erosion-corrosion testing in simulated drilling fluid

Number	Sample ID	Coating materials	Substrates
1	Steel 1	-	34CrNiMo6
2	222Xylan/GO	PTFE/Graphene oxide	34CrNiMo6
3	232WC1	WCCoCr	34CrNiMo6
4	234Flux1	NiCrFeBSi	34CrNiMo6
5	Steel 2	-	440B
6	232WC2	WC-CoCr	440B

LIST OF IMAGES

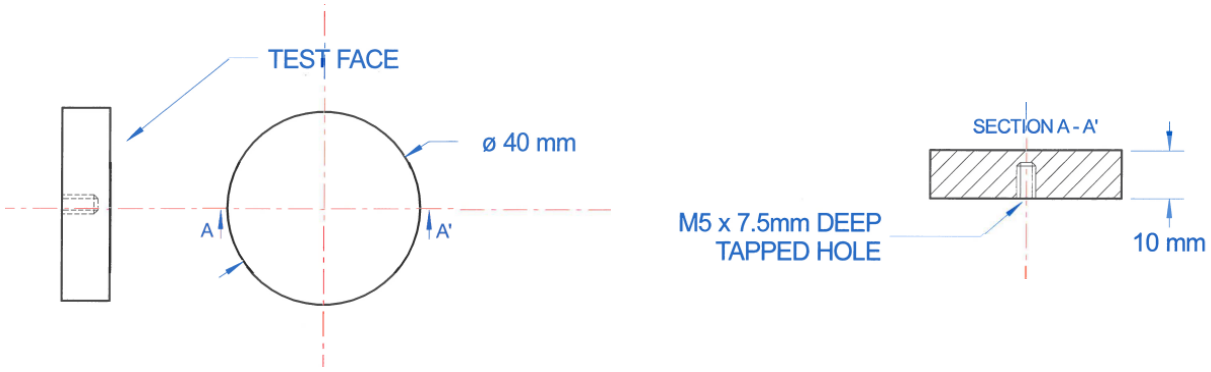


Figure 1 Design of test coupon for Geo-Drill erosion-corrosion testing

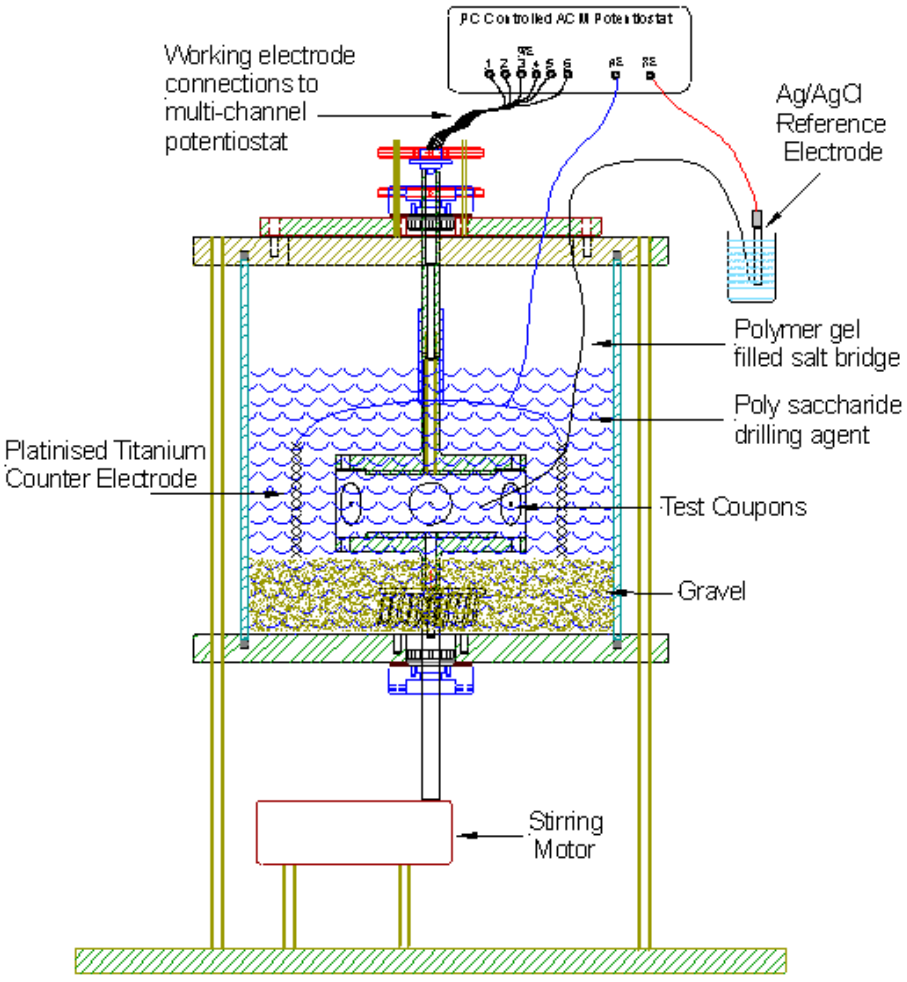


Figure 2 The design of TWI's erosion-corrosion facility

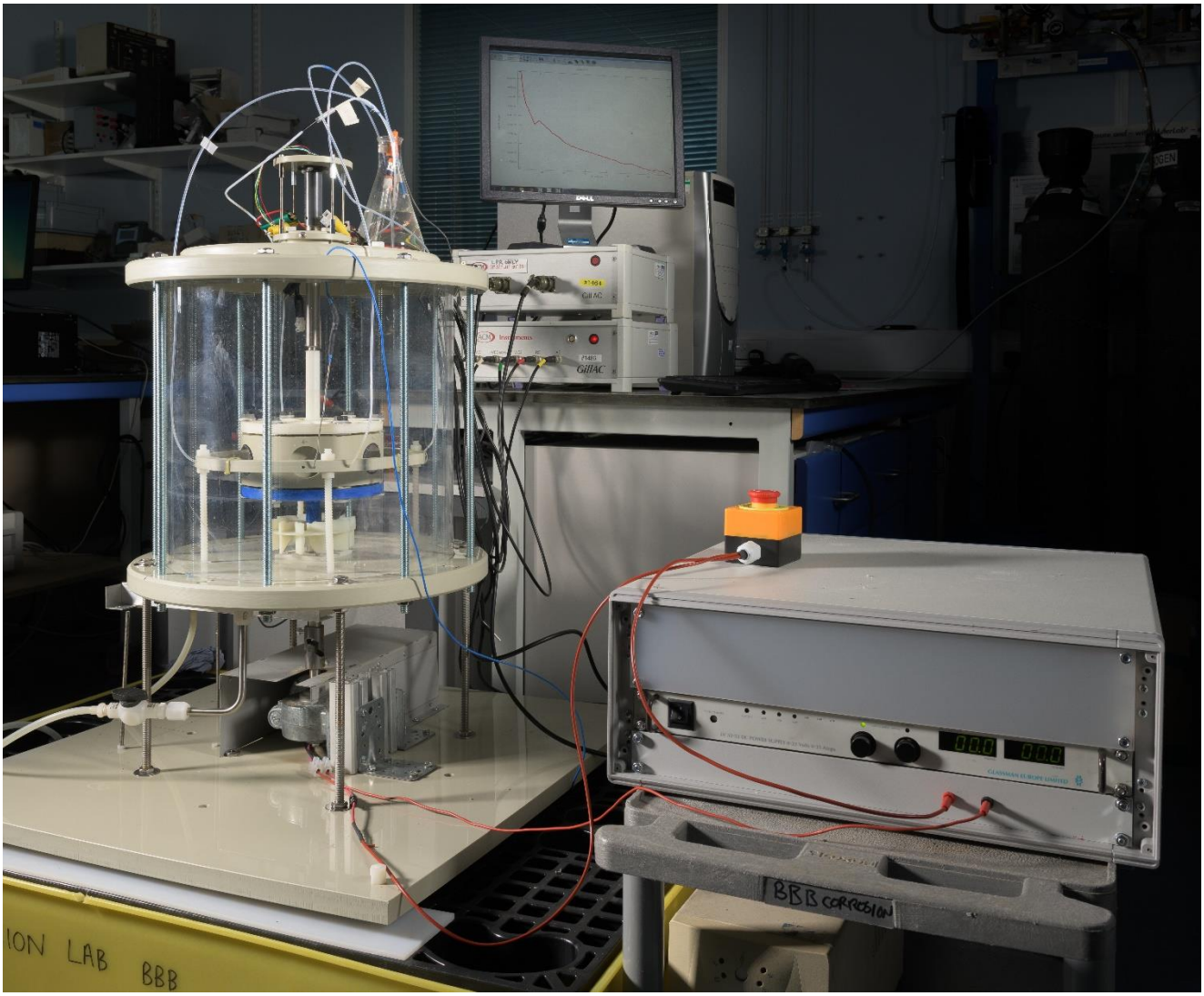


Figure 3 A photograph of TWI's erosion-corrosion facility

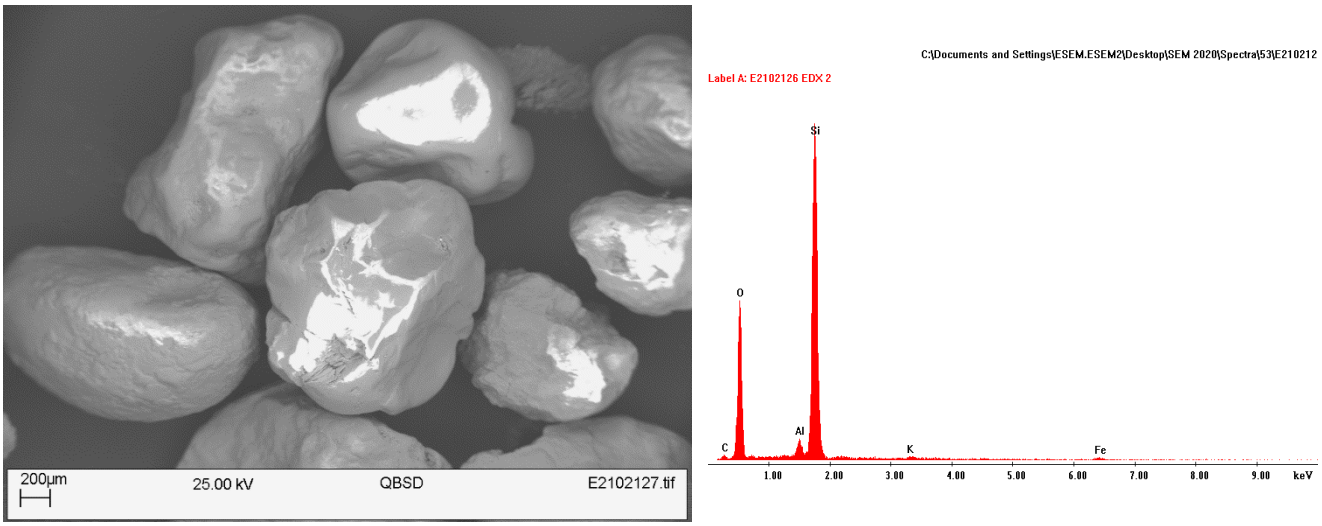


Figure 4 SEM and EDX analyse of sand particles added into simulated drilling fluid

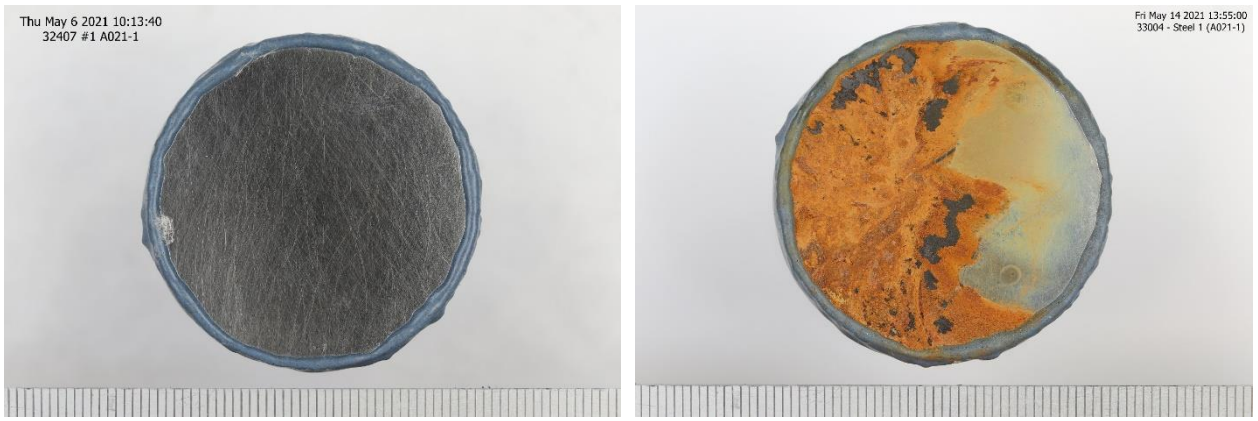


Figure 5 Digital images of Steel 1 after 24h testing: (left) before testing, (right) after testing

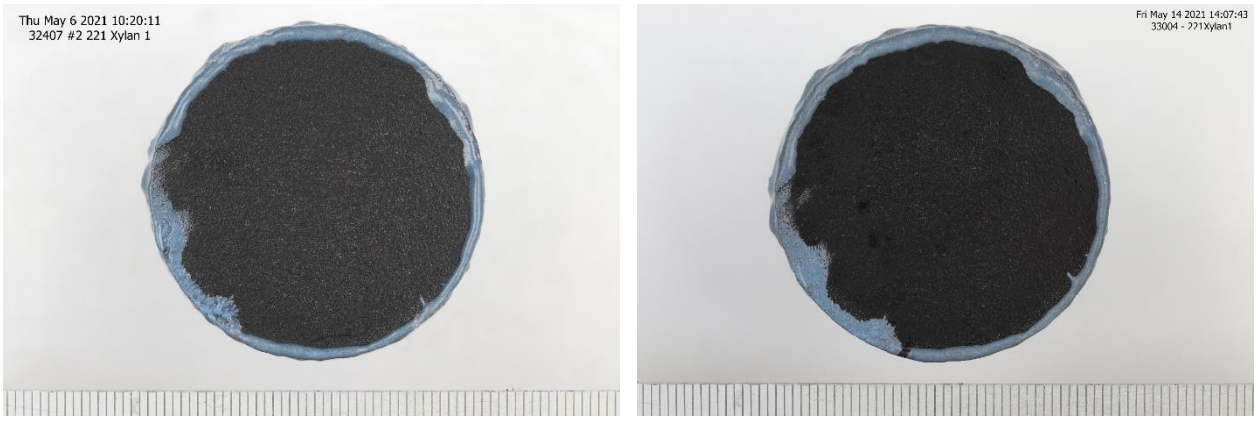


Figure 6 Digital images of 221Xylan after 24h testing: (left) before testing, (right) after testing

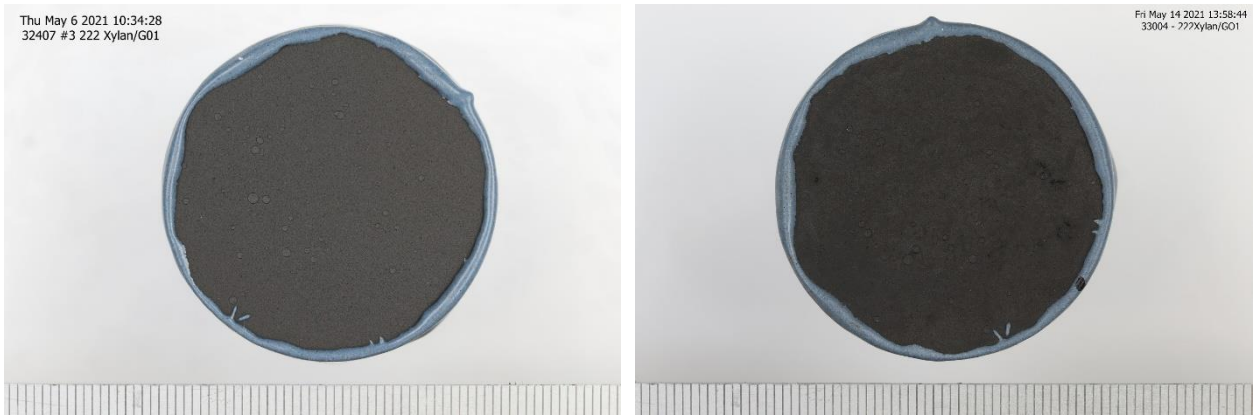


Figure 7 Digital images of 222Xylan/GO after 24h testing: (left) before testing, (right) after testing

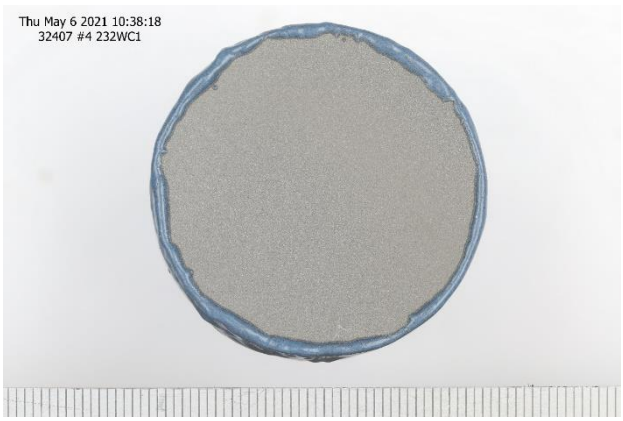
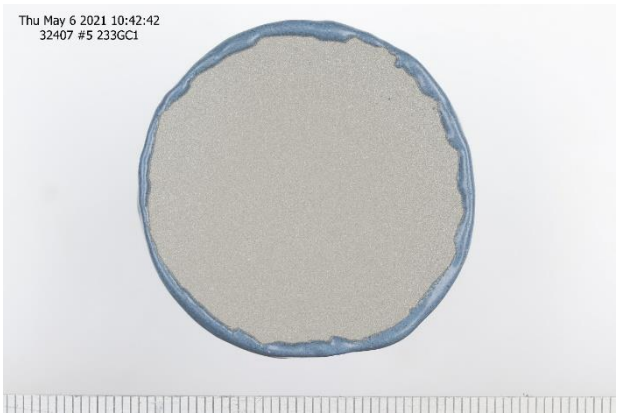


Figure 8 Digital images of 232WC1 after 24h testing: (left) before testing, (right) after testing



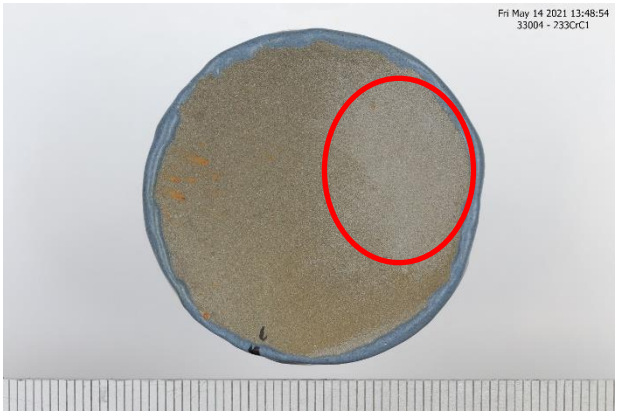


Figure 9 Digital images of 233CrC1 after 24h testing: (left) before testing, (right) after testing

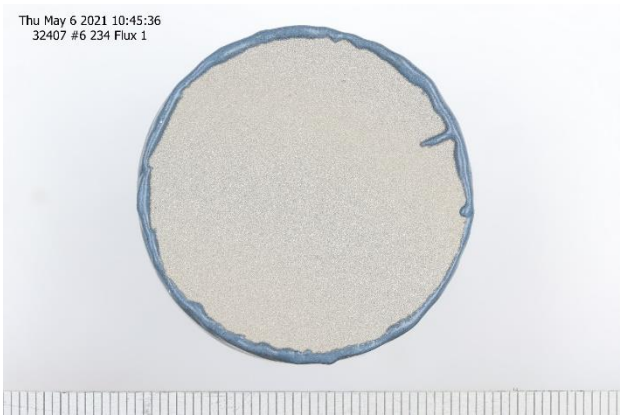


Figure 10 Digital images of 234Flux1 after 24h testing: (left) before testing, (right) after testing

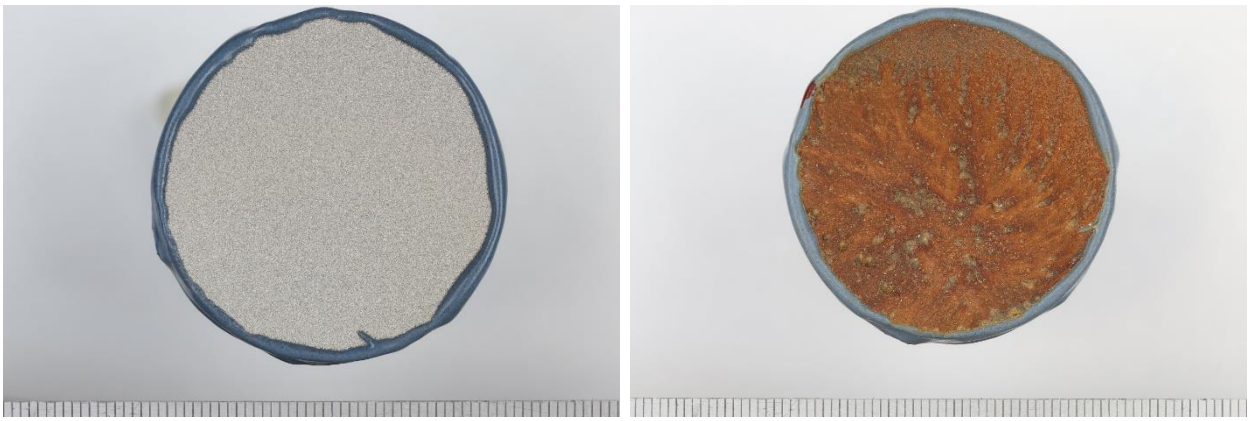


Figure 11 Digital images of 235Amor1 after 24h testing: (left) before testing, (right) after testing

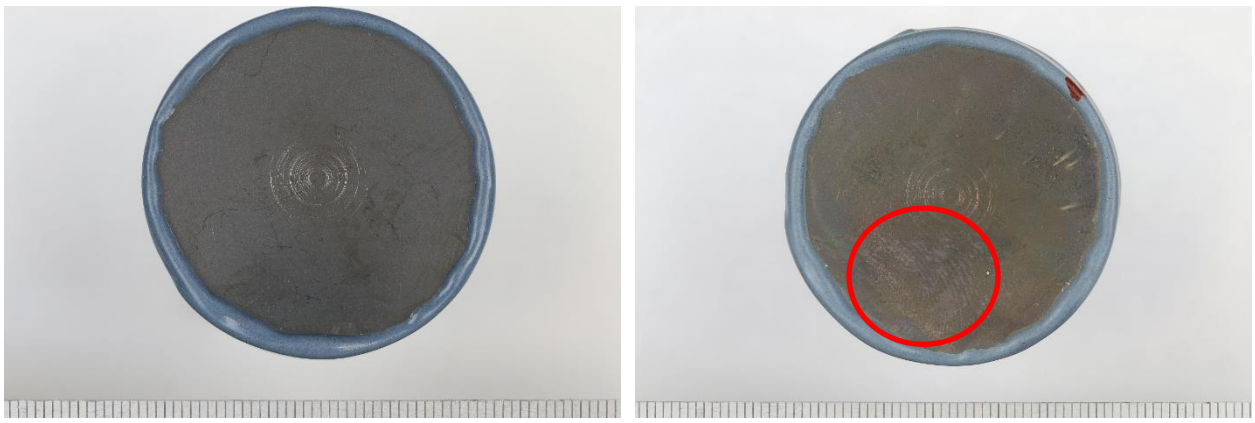


Figure 12 Digital images of 271Ni after 24h testing: (left) before testing, (right) after testing

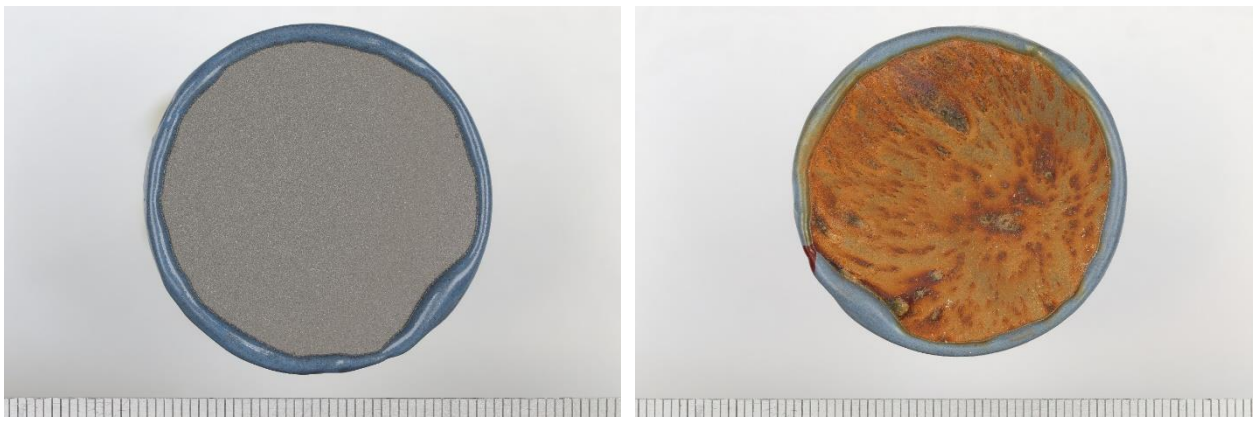


Figure 13 Digital images of 282WCr1 after 24h testing: (left) before testing, (right) after testing

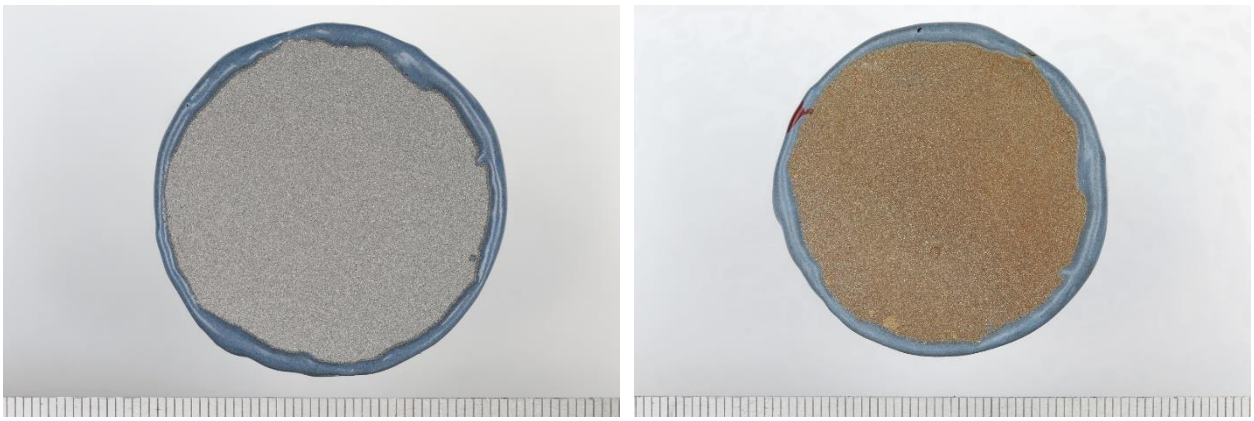


Figure 14 Digital images of 283HEA1 after 24h testing: (left) before testing, (right) after testing

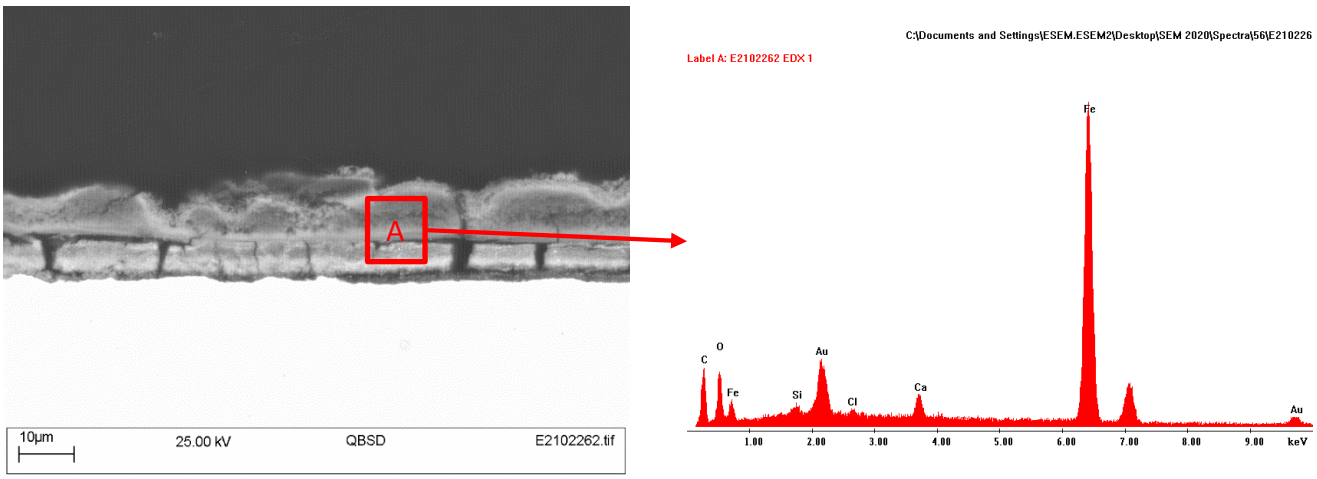


Figure 15 SEM and EDX analyse on polished cross-sectional surface of Steel 1 after 24h testing

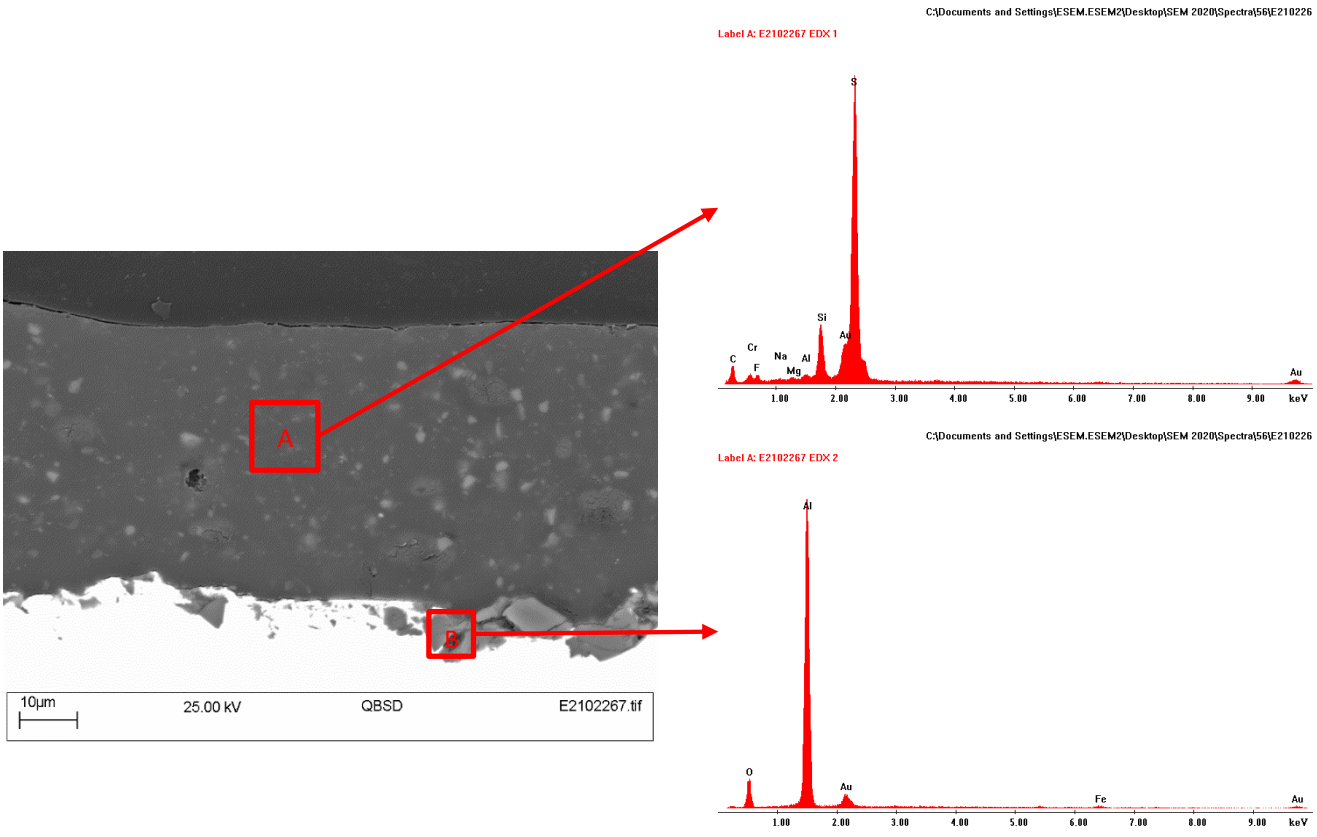


Figure 16 SEM and EDX analyse on polished cross-sectional surface of 221Xylan after 24h testing

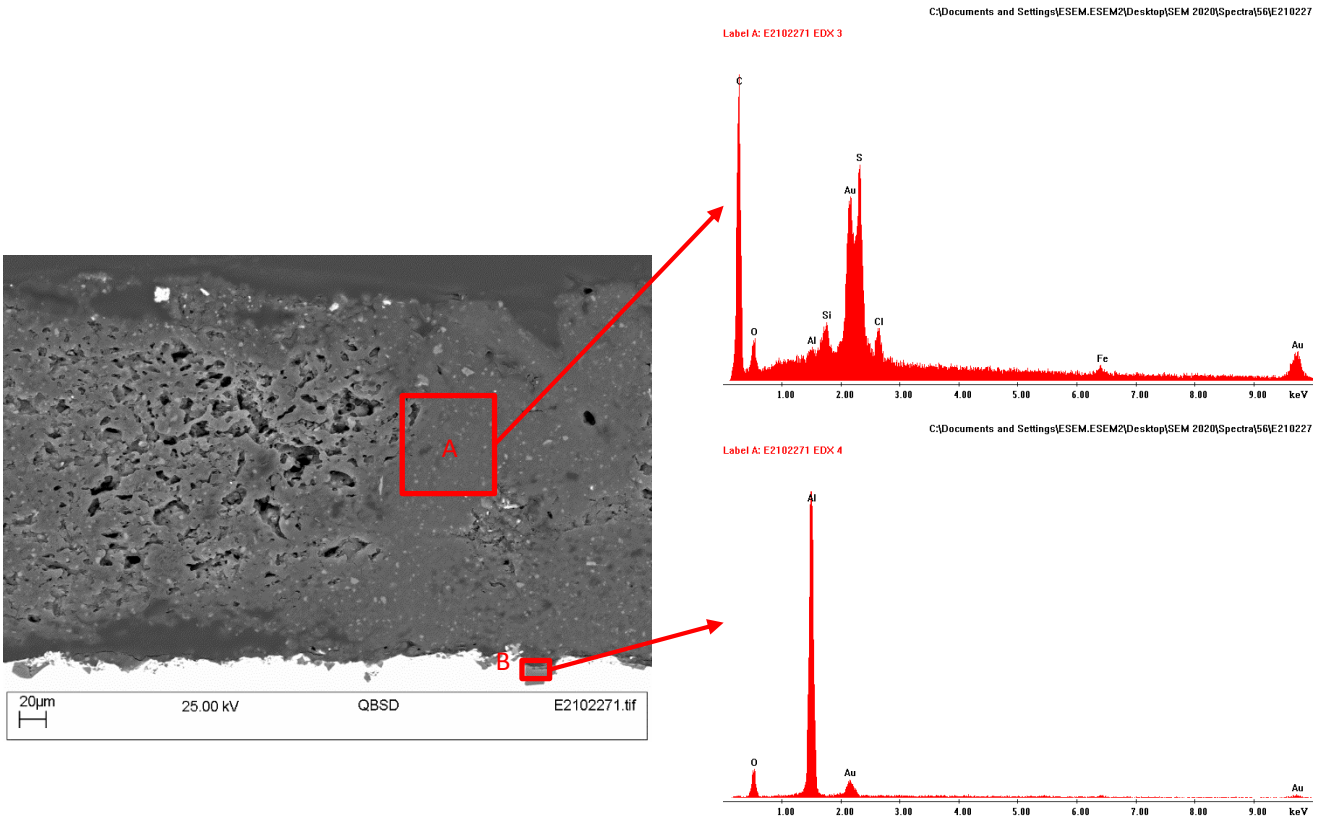


Figure 17 SEM and EDX analyse on polished cross-sectional surface of 222Xylan/GO after 24h testing

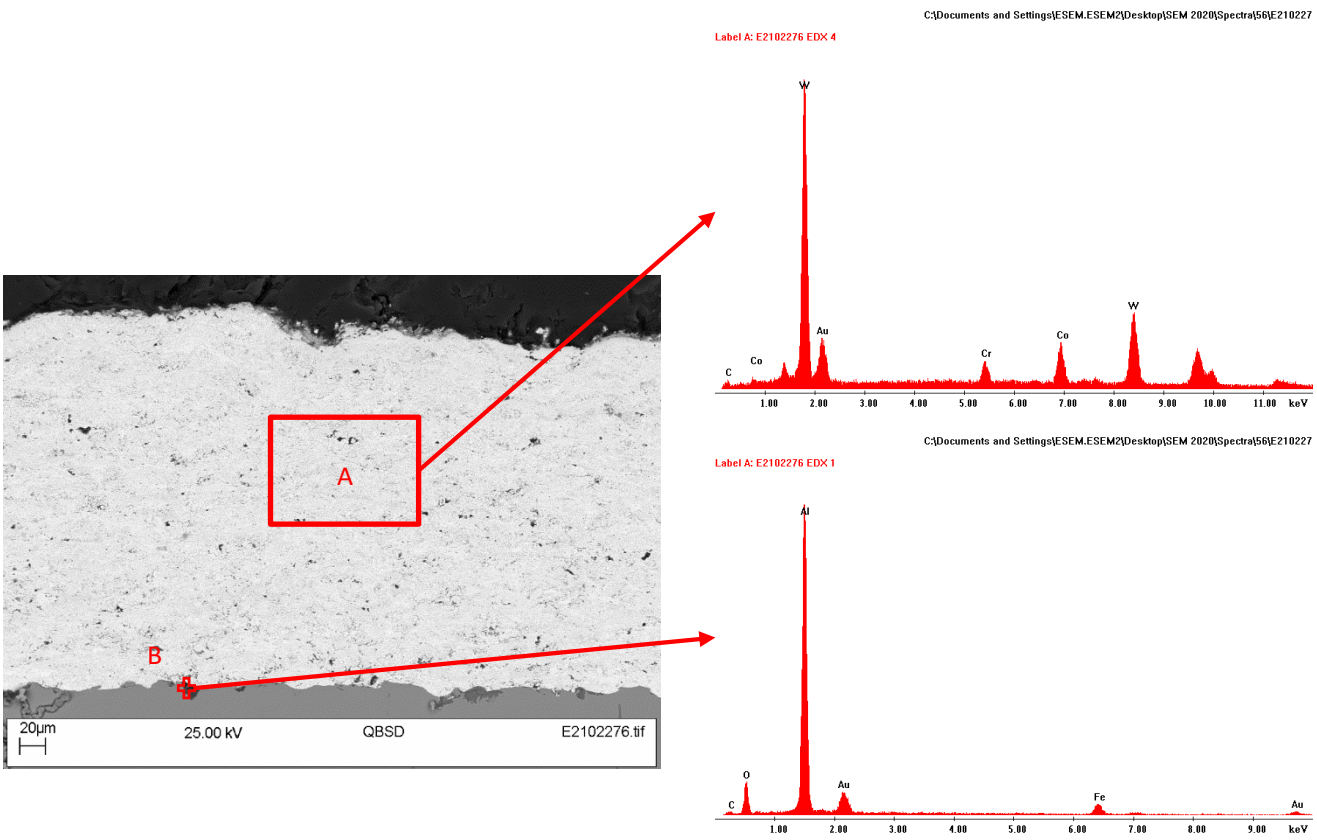


Figure 18 SEM and EDX analyse on polished cross-sectional surface of 232WC1 after 24h testing

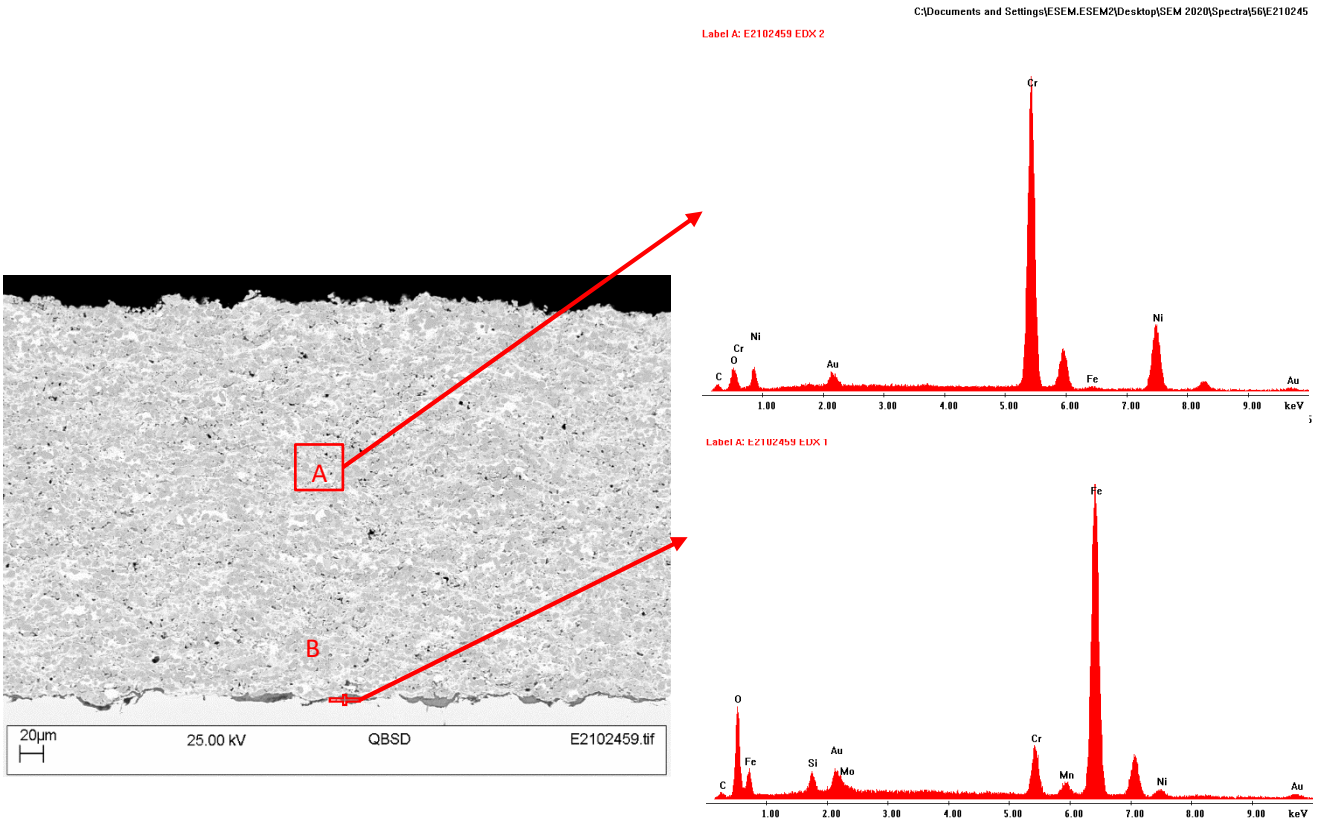


Figure 19 SEM and EDX analyse on polished cross-sectional surface of 233CrC1 after 24h testing

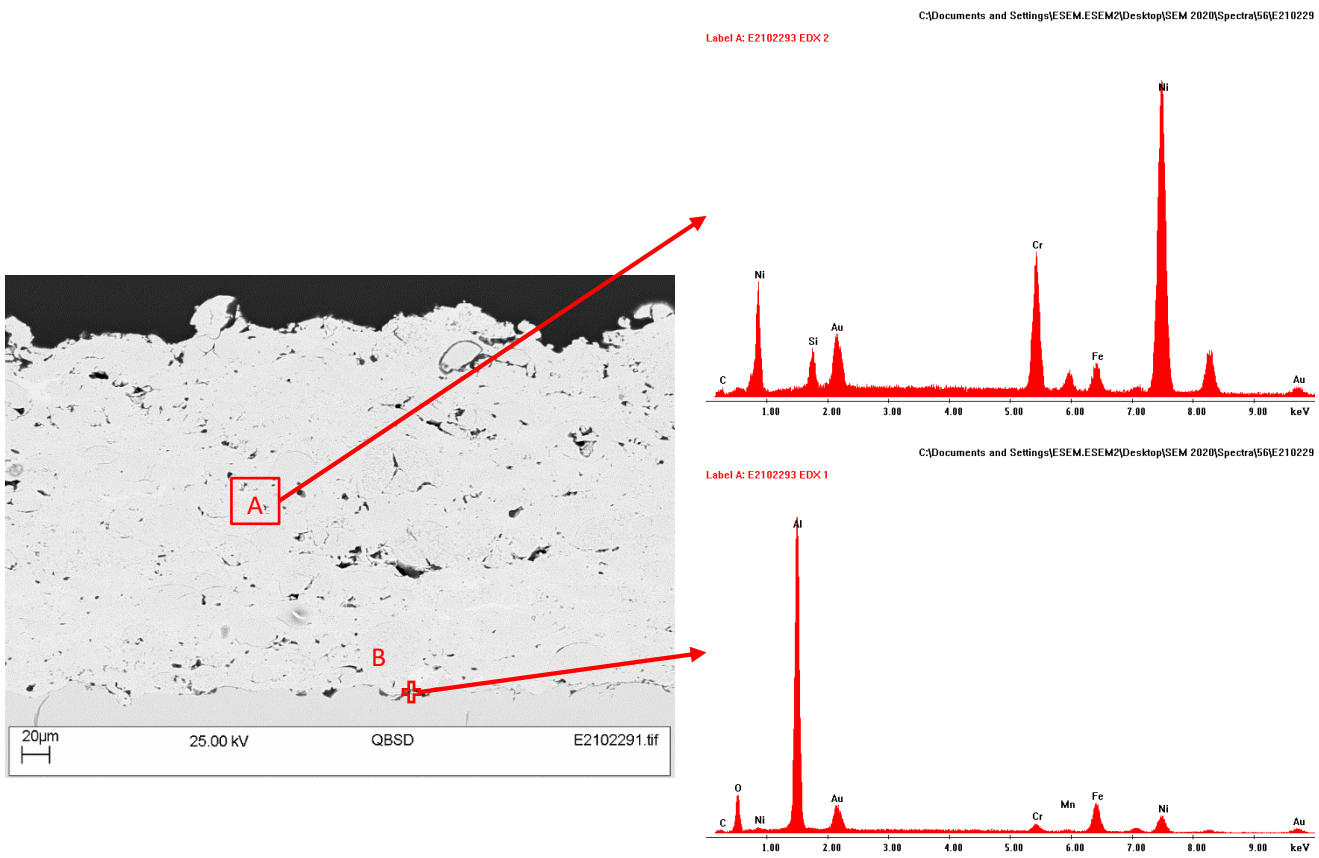


Figure 20 SEM and EDX analyse on polished cross-sectional surface of 234Flux1 after 24h testing

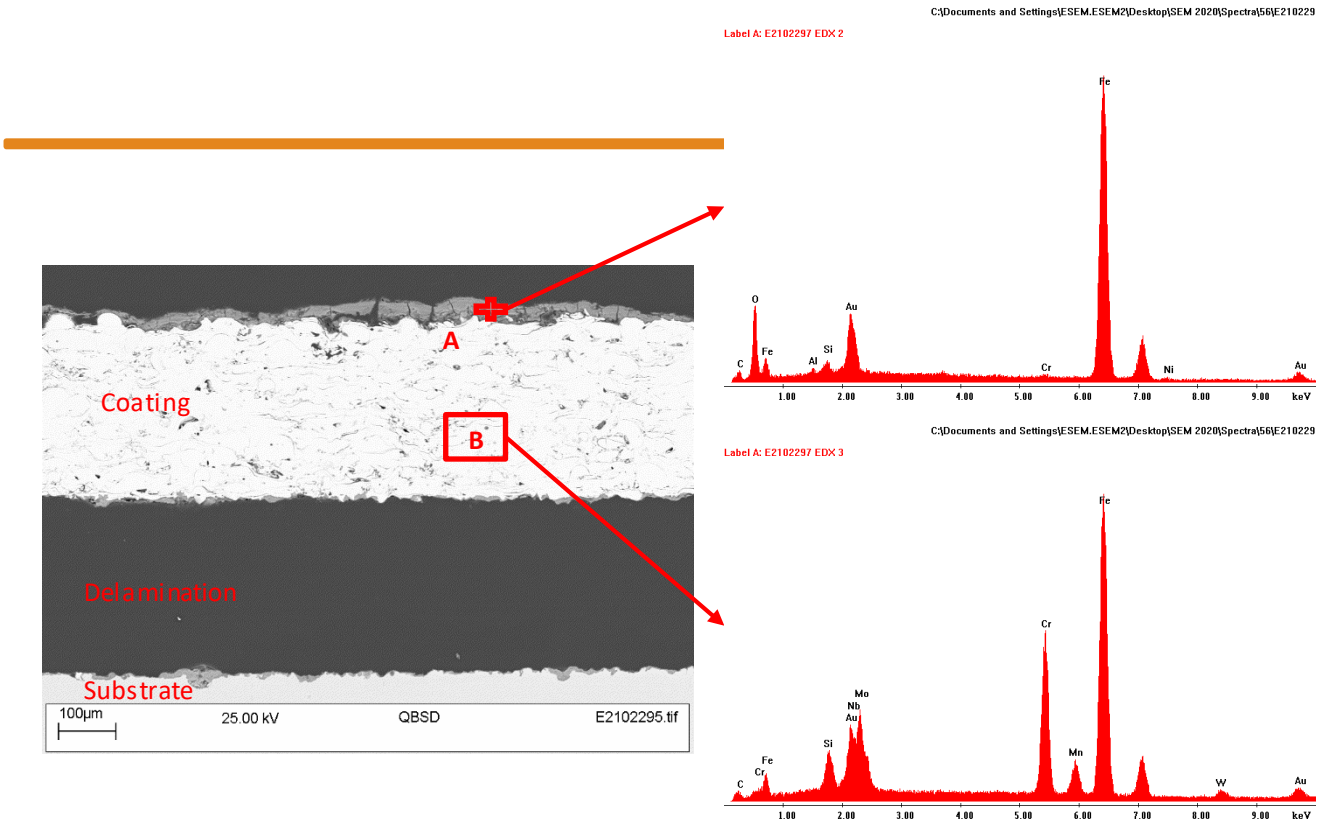


Figure 21 SEM and EDX analyse on polished cross-sectional surface of 235Amor1 after 24h testing

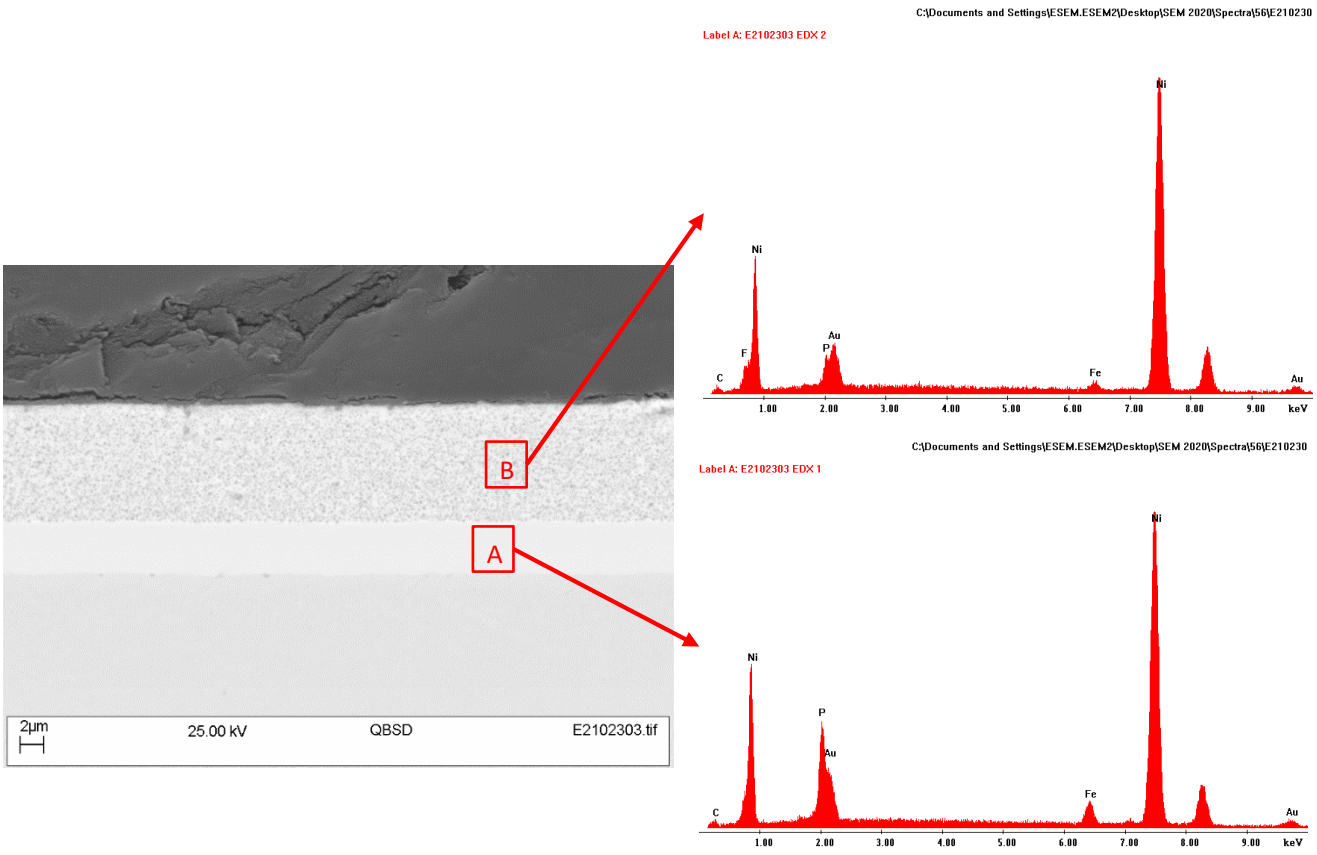


Figure 22 SEM and EDX analyse on polished cross-sectional surface of 271Ni after 24h testing

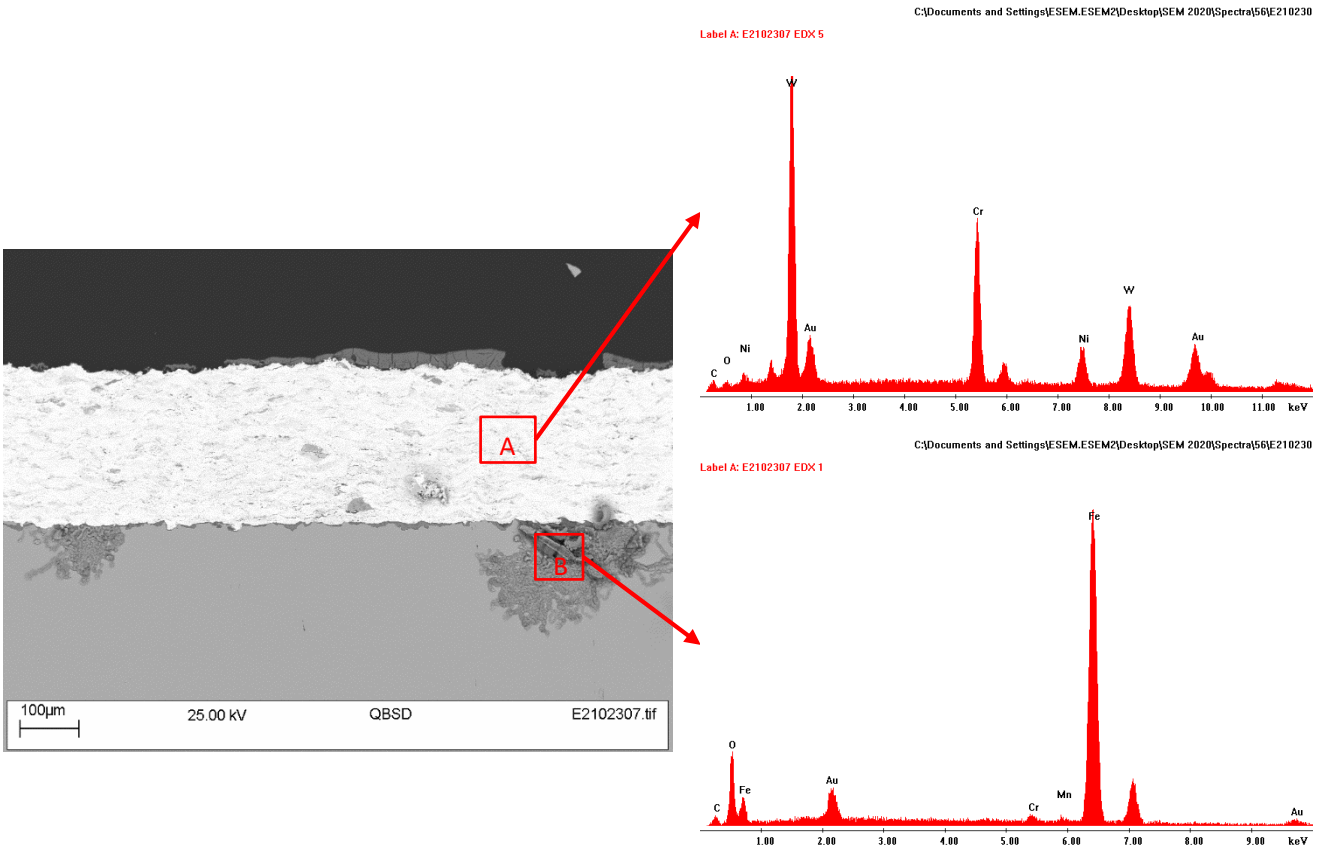


Figure 23 SEM and EDX analyse on polished cross-sectional surface of 282WCr1 after 24h testing

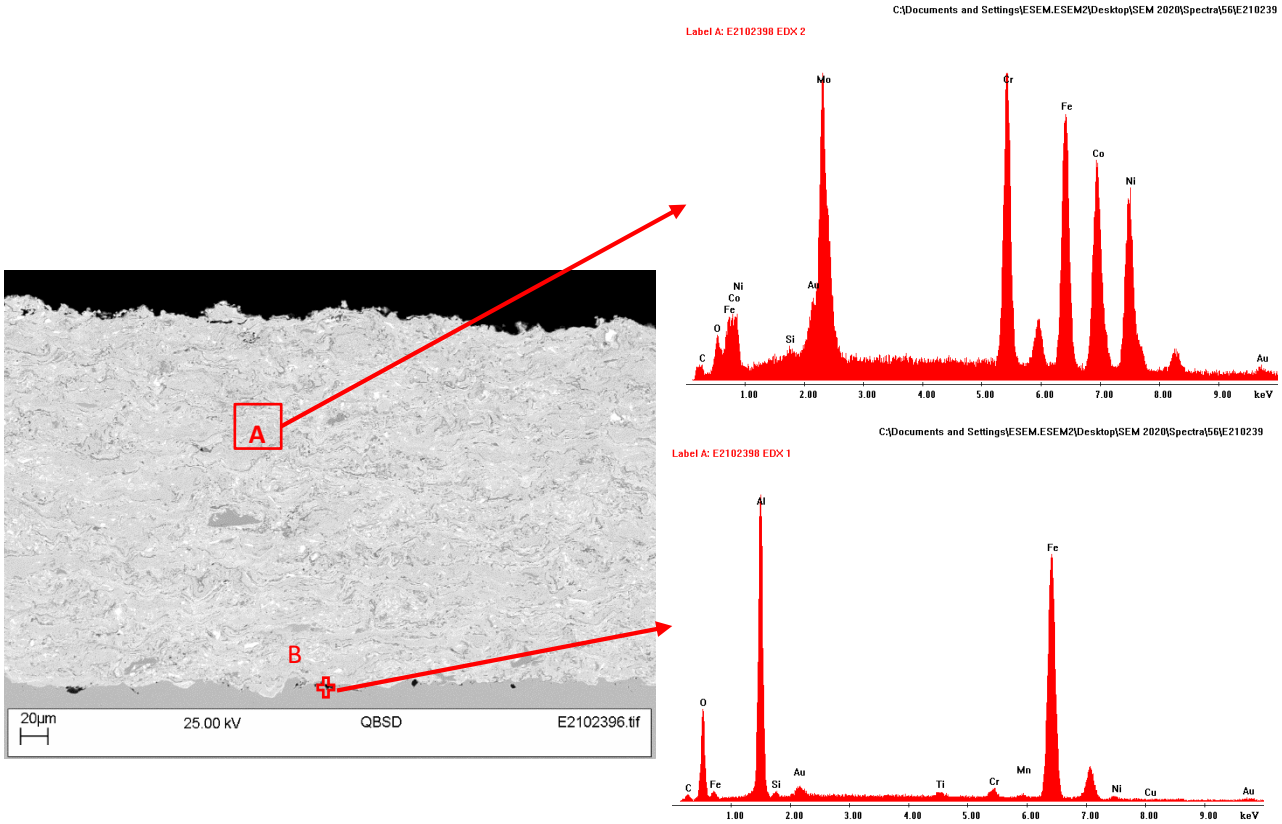


Figure 24 SEM and EDX analyse on polished cross-sectional surface of 283HEA1 after 24h testing R

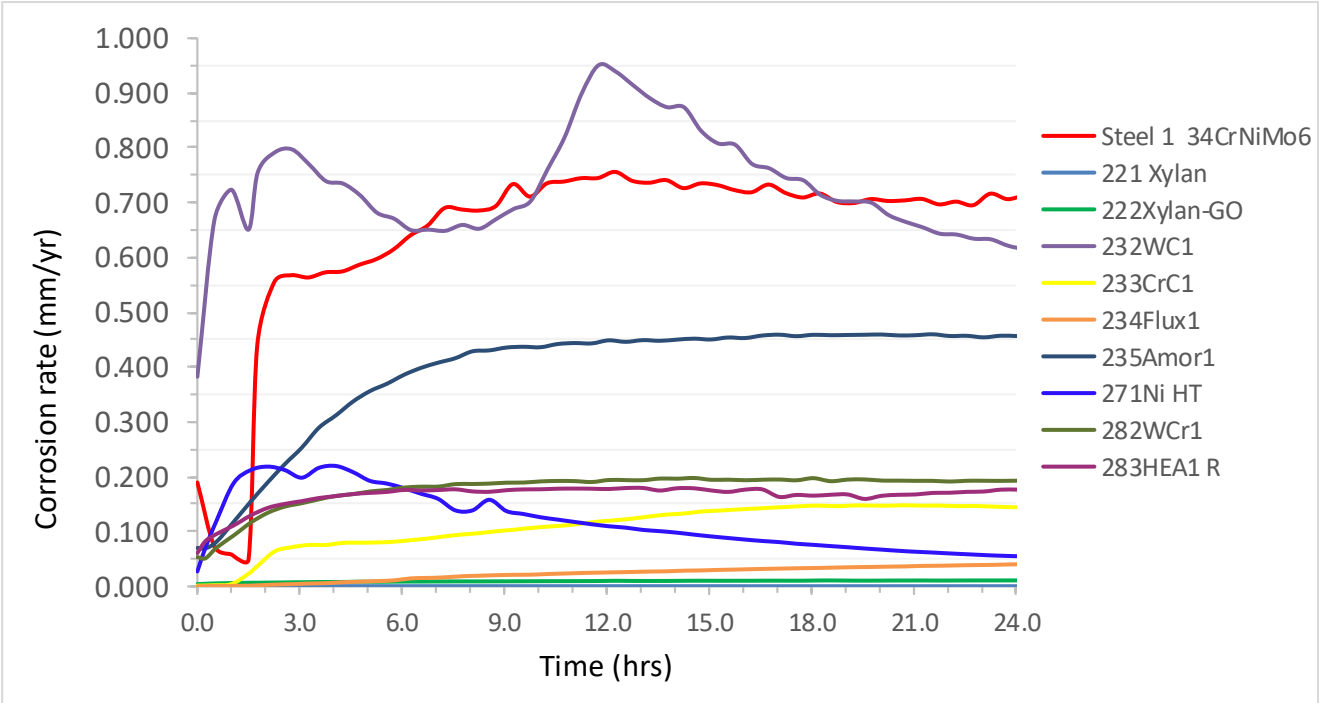


Figure 25 Corrosion rate for Coatings on Steel 1 after 24h testing in simulated drilling fluid

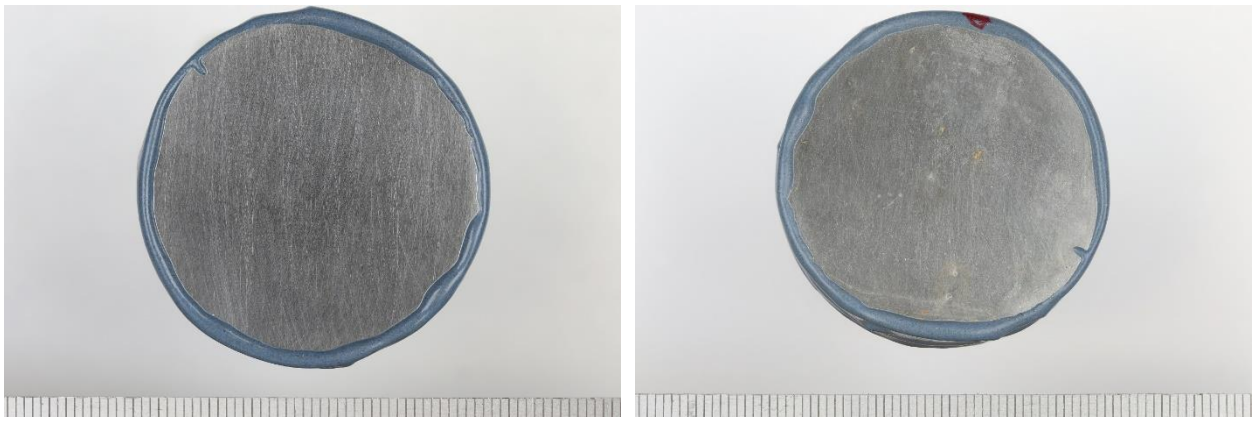


Figure 26 Digital images of Steel 2 after 24h testing: (left) before testing, (right) after testing

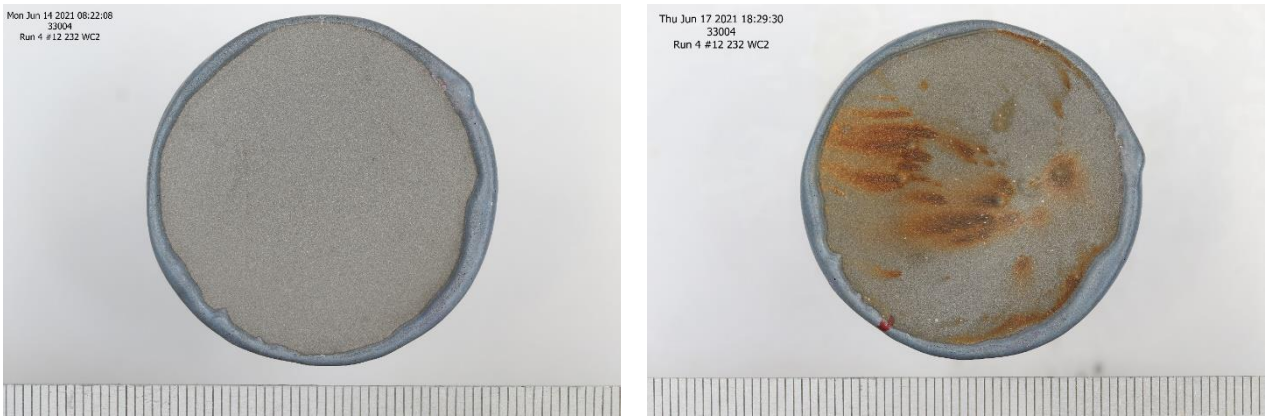


Figure 27 Digital images of 232WC2 after 24h testing: (left) before testing, (right) after testing

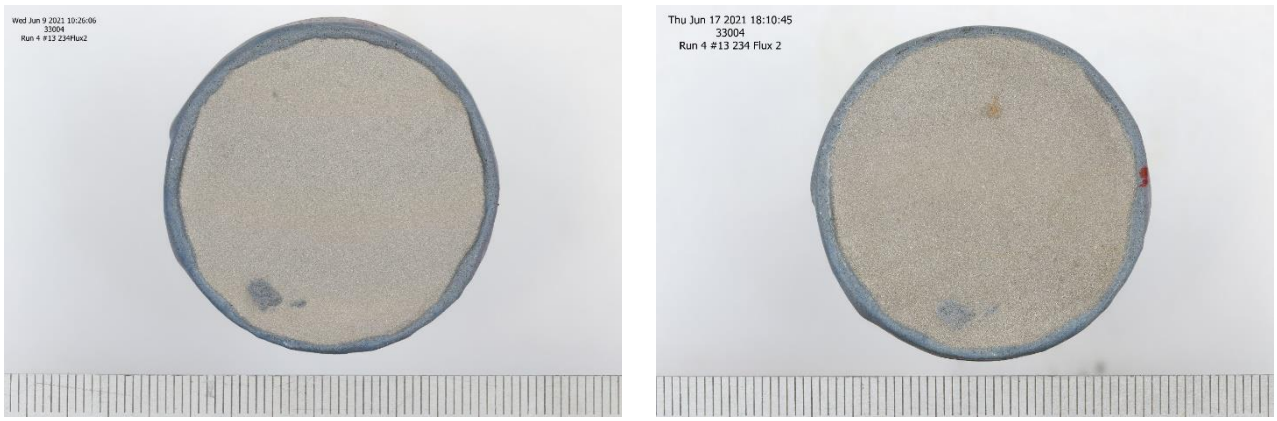


Figure 28 Digital images of 234Flux2 after 24h testing: (left) before testing, (right) after testing

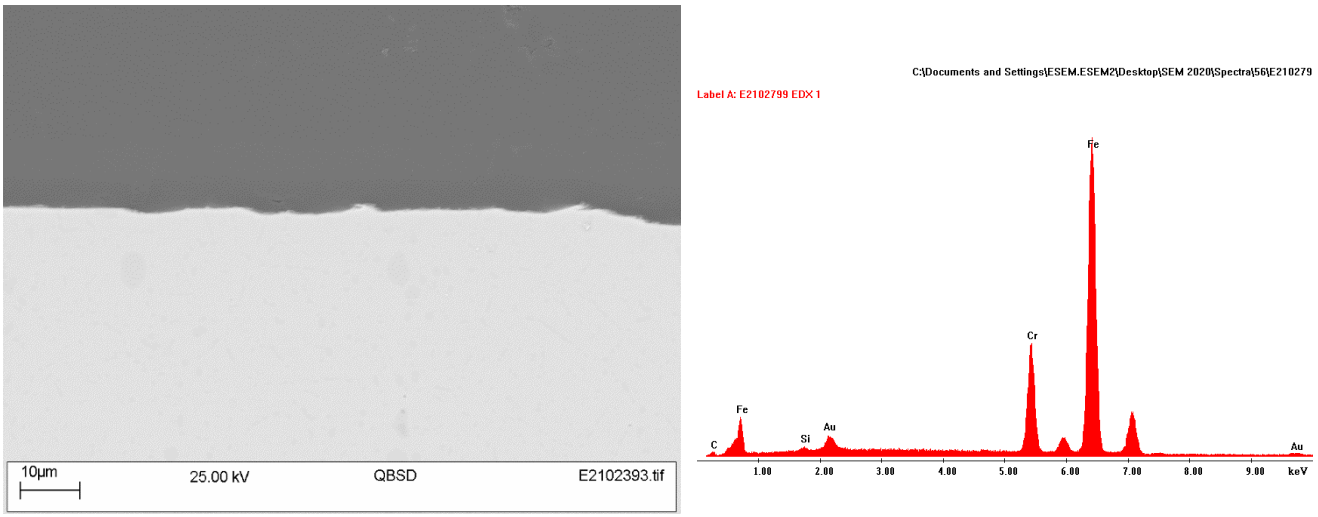


Figure 29 SEM and EDX analyse on polished cross-sectional surface of Steel 2 after 24h testing

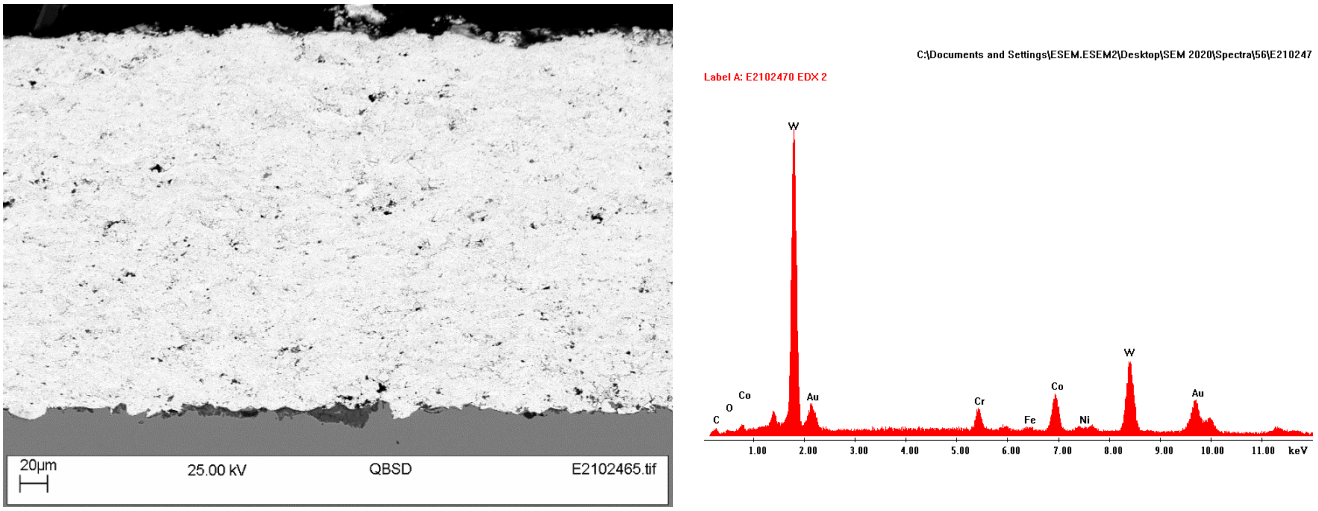


Figure 30 SEM and EDX analyse on polished cross-sectional surface of 232WC2 after 24h testing

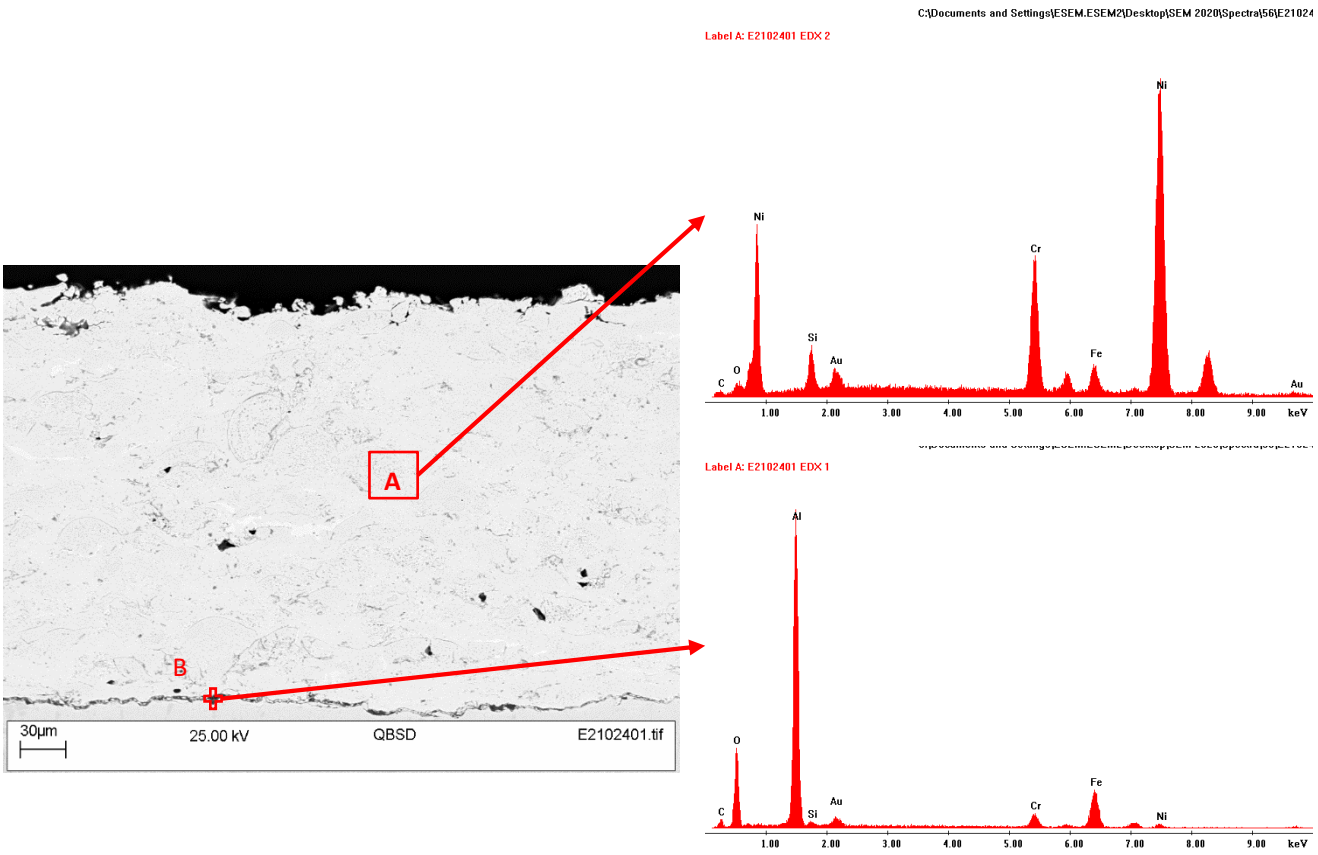


Figure 31 SEM and EDX analyse on polished cross-sectional surface of 234Flux2 after 24h testing

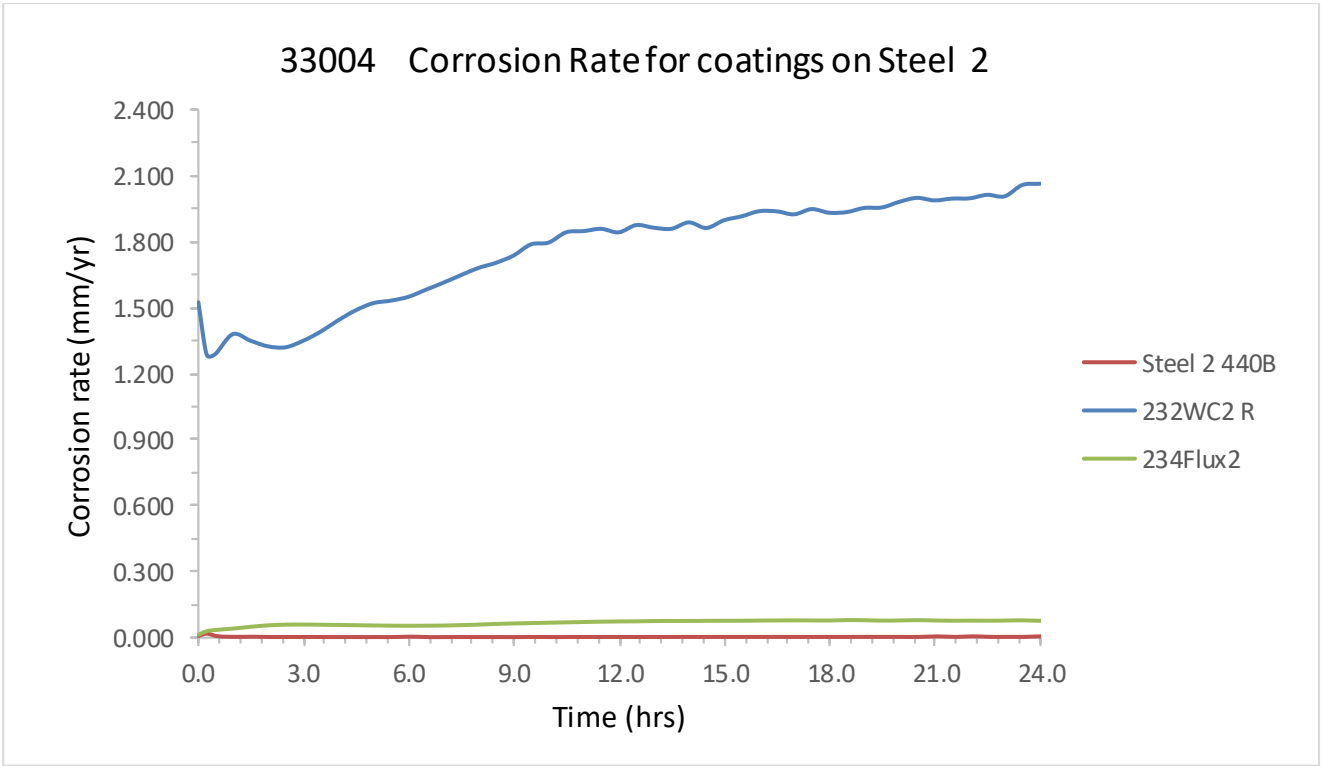


Figure 32 Corrosion rate for Coatings on Steel 2 after 24h testing in simulated drilling fluid

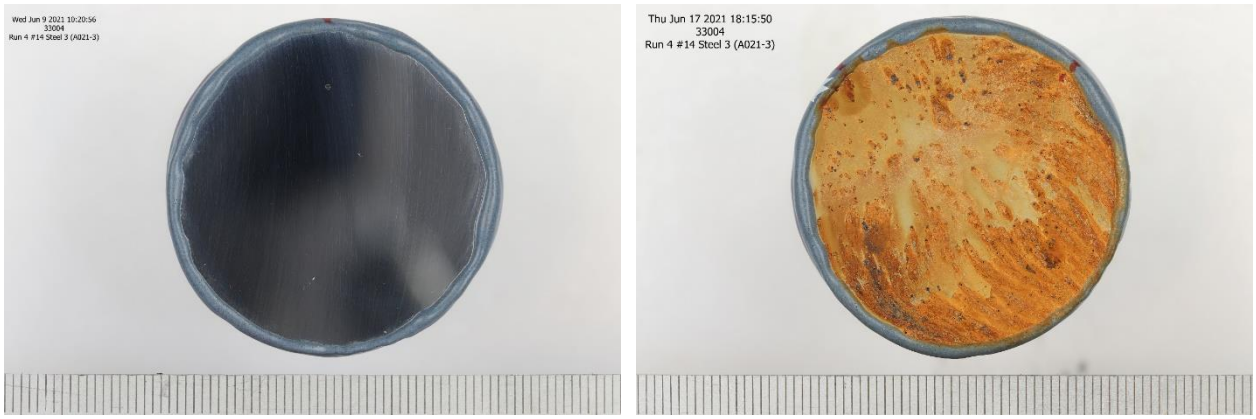


Figure 33 Digital images of Steel 3 after 24h testing: (left) before testing, (right) after testing

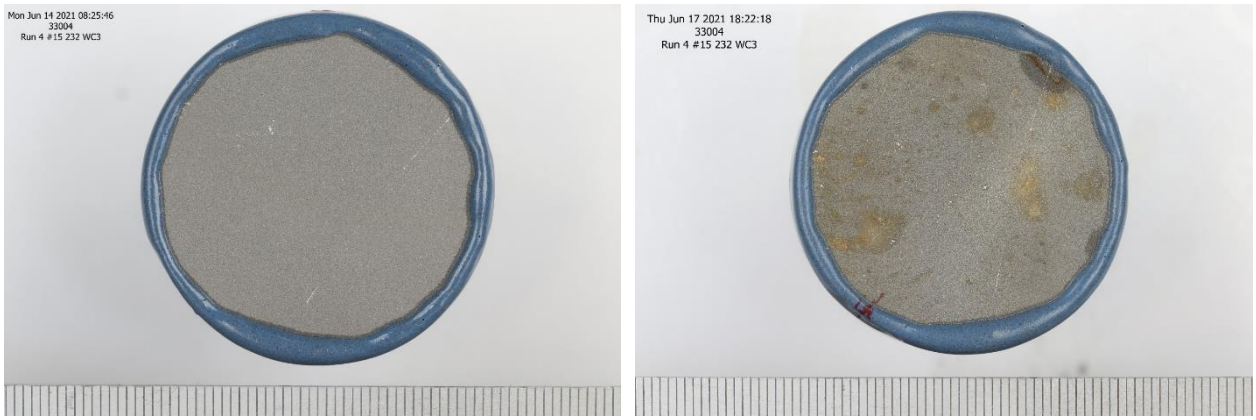


Figure 34 Digital images of 232WC3 after 24h testing: (left) before testing, (right) after testing

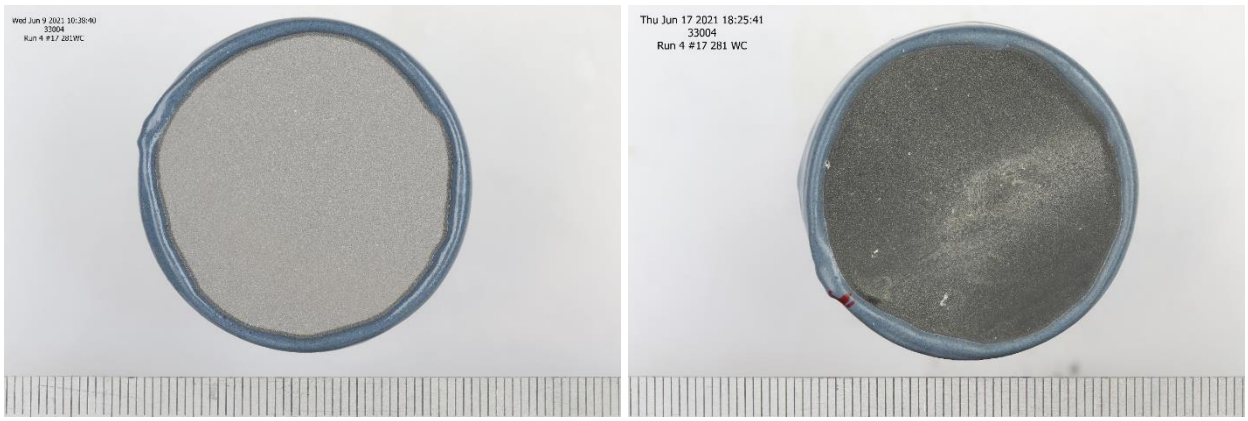


Figure 35 Digital images of 281WC3 after 24h testing: (left) before testing, (right) after testing

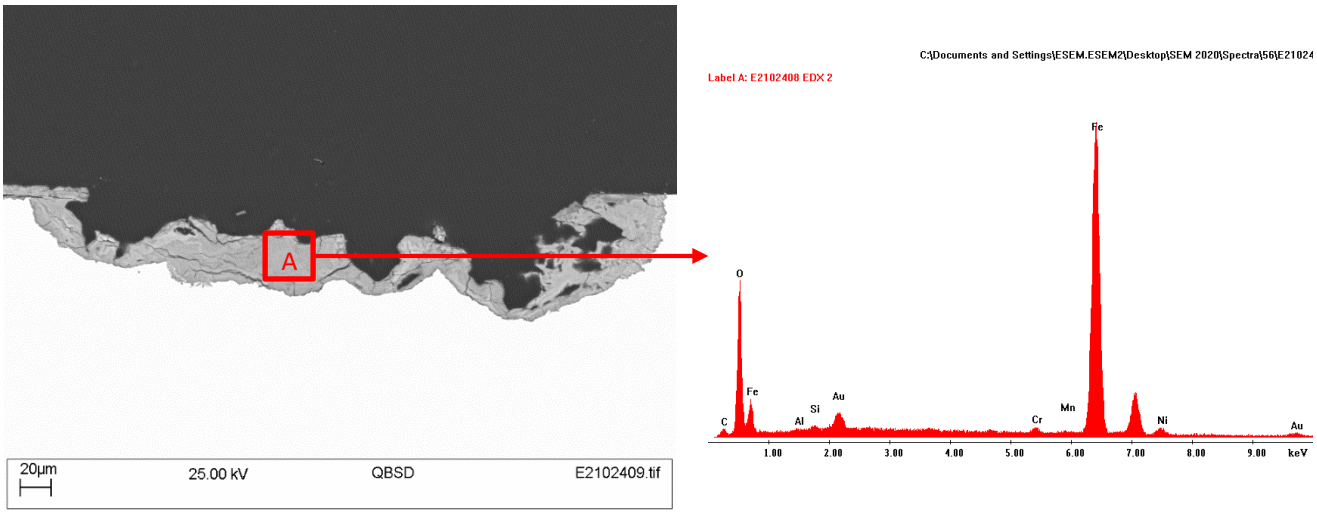


Figure 36 SEM and EDX analyse on polished cross-sectional surface of Steel 3 after 24h testing

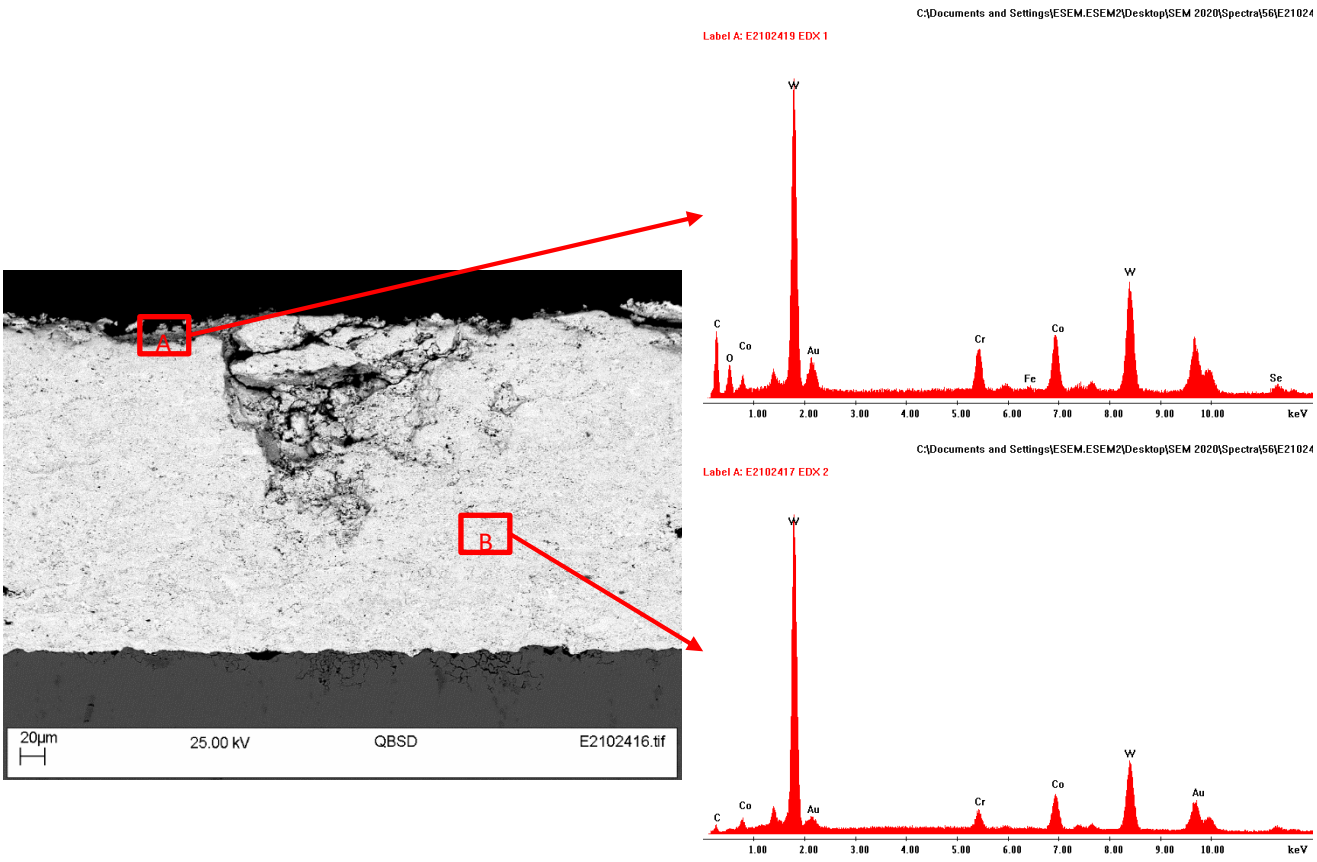


Figure 37 SEM and EDX analyse on polished cross-sectional surface of 232WC3 after 24h testing

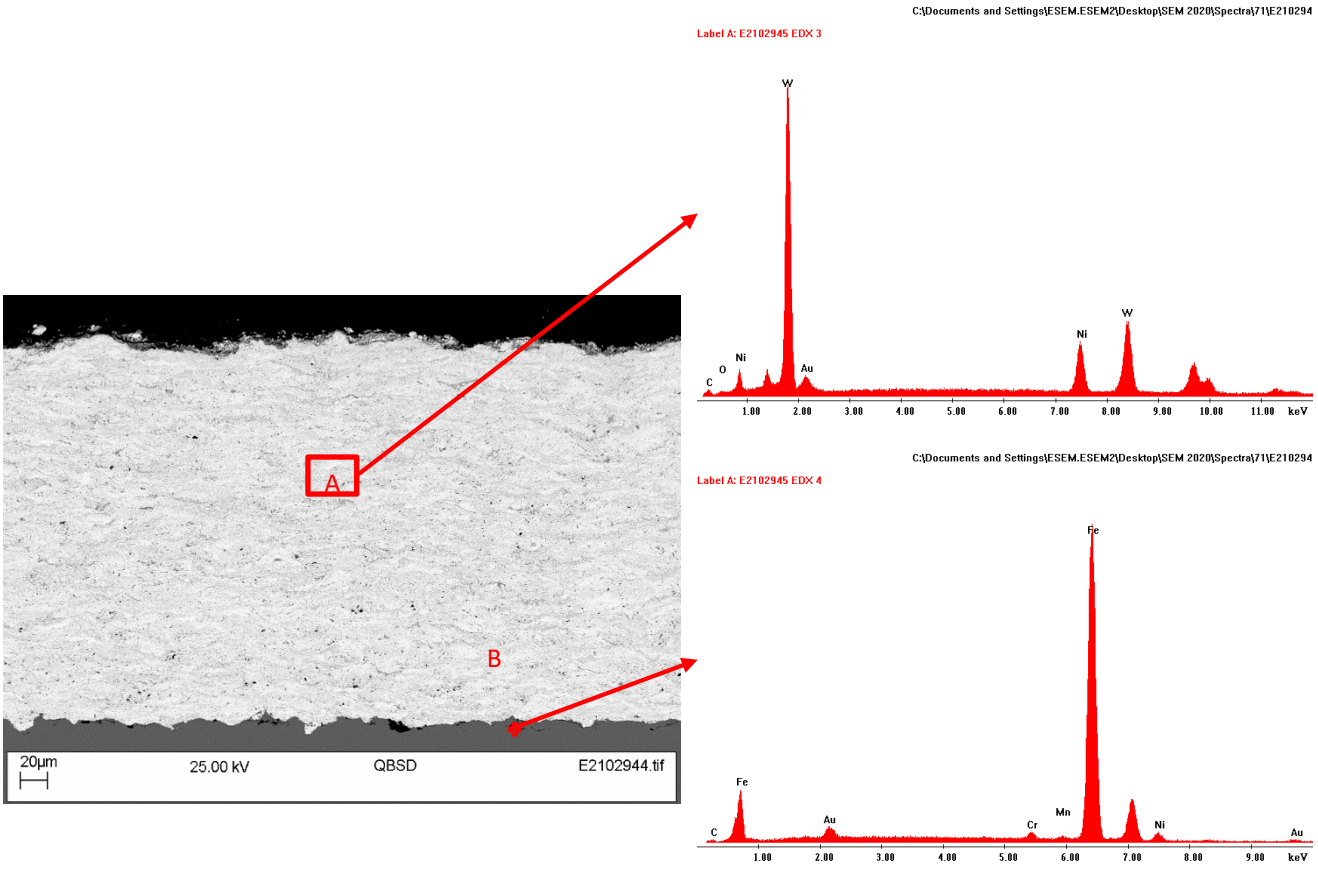


Figure 38 SEM and EDX analyse on polished cross-sectional surface of 281WC3 after 24h testing

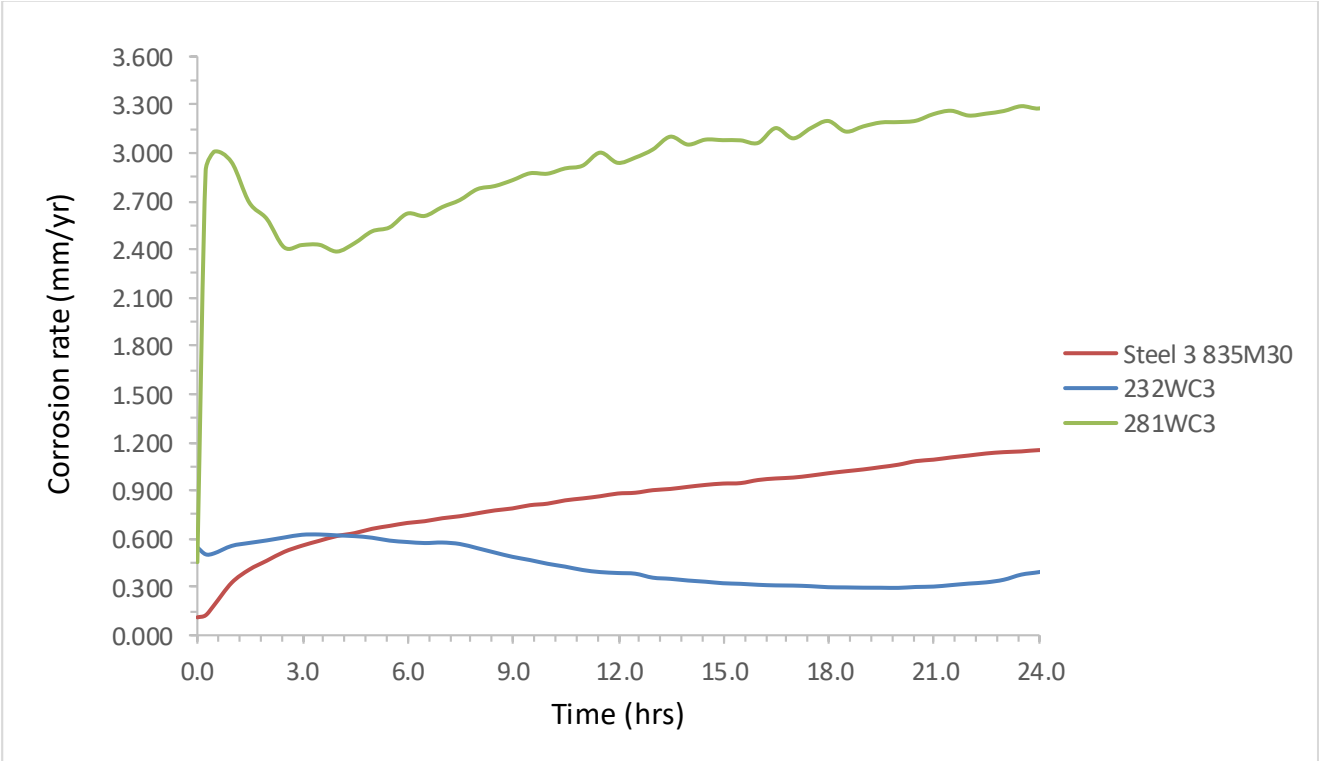


Figure 39 Corrosion rate for Coatings on Steel 3 after 24h testing in simulated drilling fluid

Figure 40 Digital images of Steel 1 after 7-day testing: (left) before testing, (right) after testing

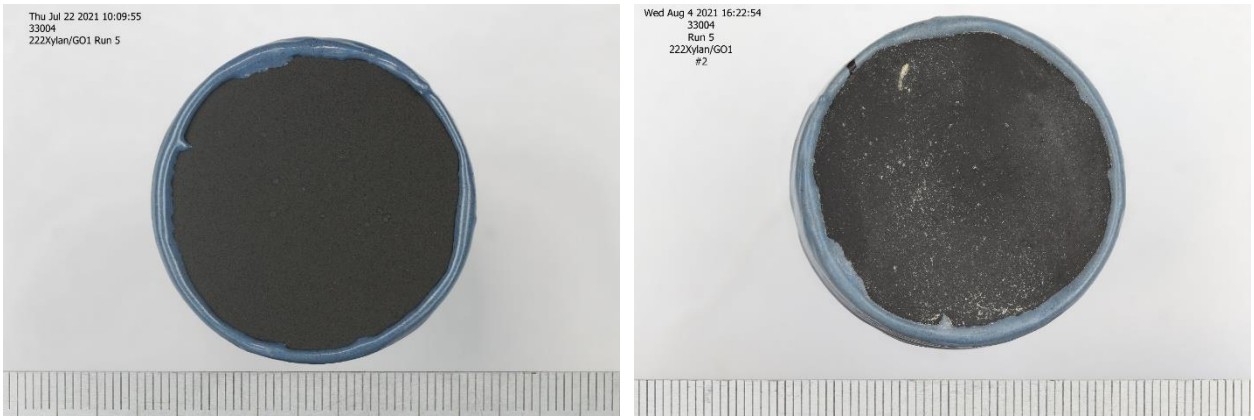


Figure 41 Digital images of 222Xylan/GO after 7-day testing: (left) before testing, (right) after testing

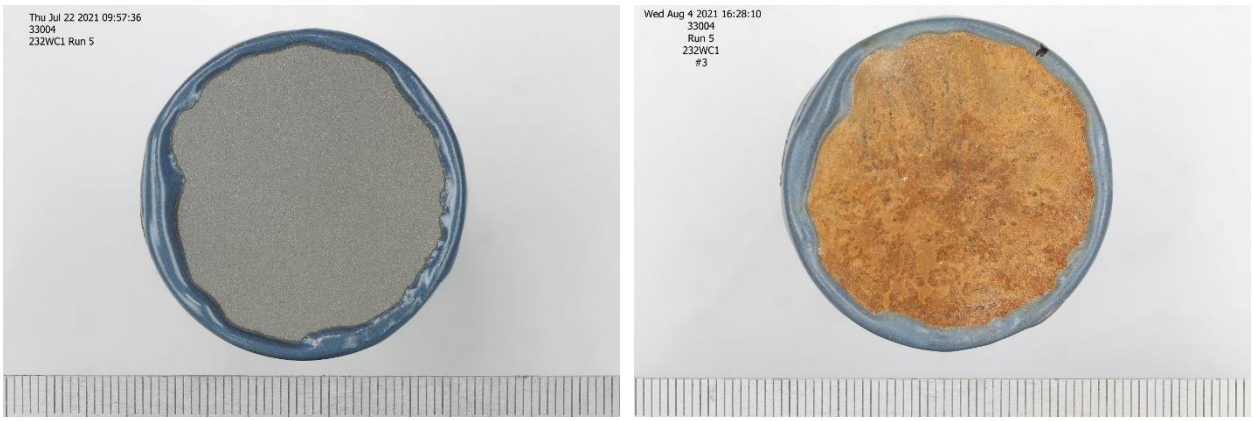


Figure 42 Digital images of 232WC1 after 7-day testing: (left) before testing, (right) after testing

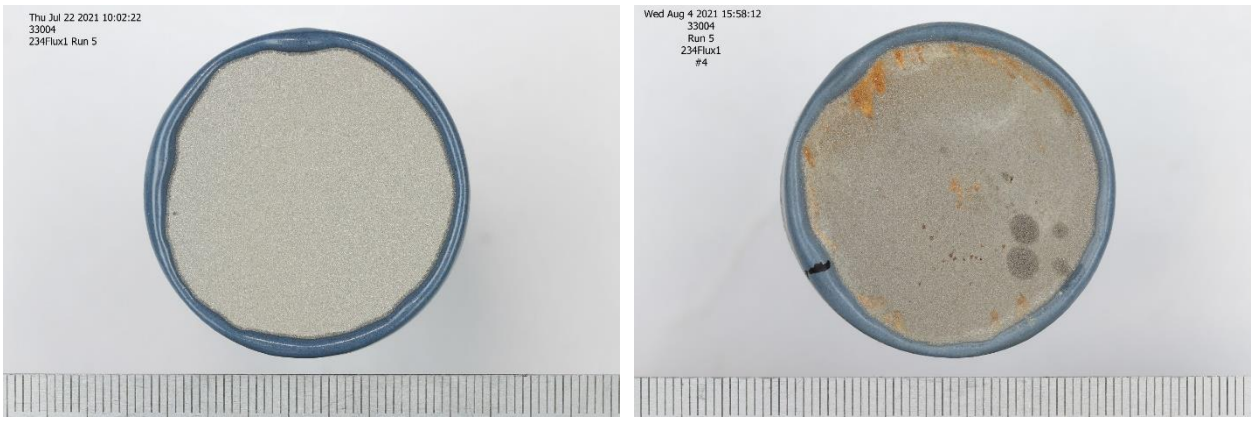


Figure 43 Digital images of 234Flux1 after 7-day testing: (left) before testing, (right) after testing

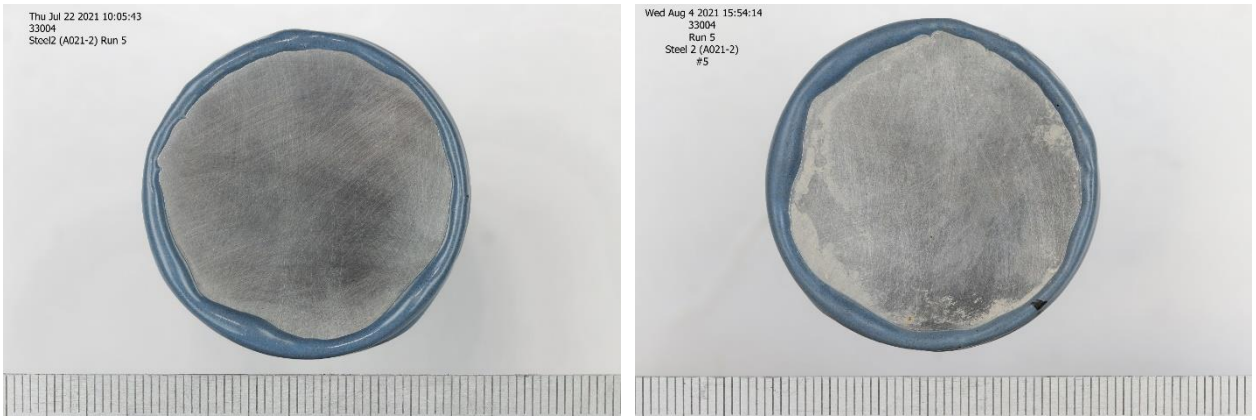


Figure 44 Digital images of Steel 2 after 7-day testing: (left) before testing, (right) after testing

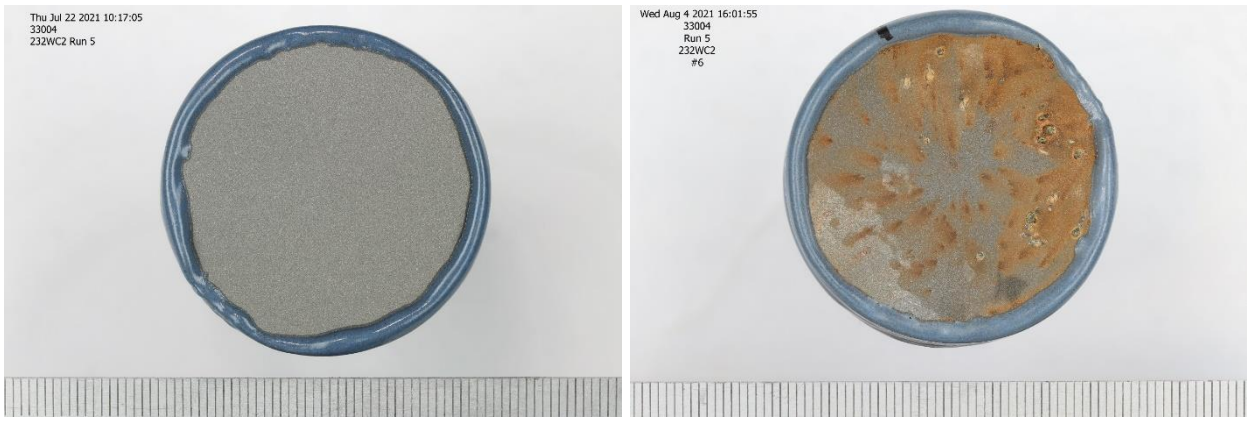


Figure 45 Digital images of 232WC2 after 7-day testing: (left) before testing, (right) after testing

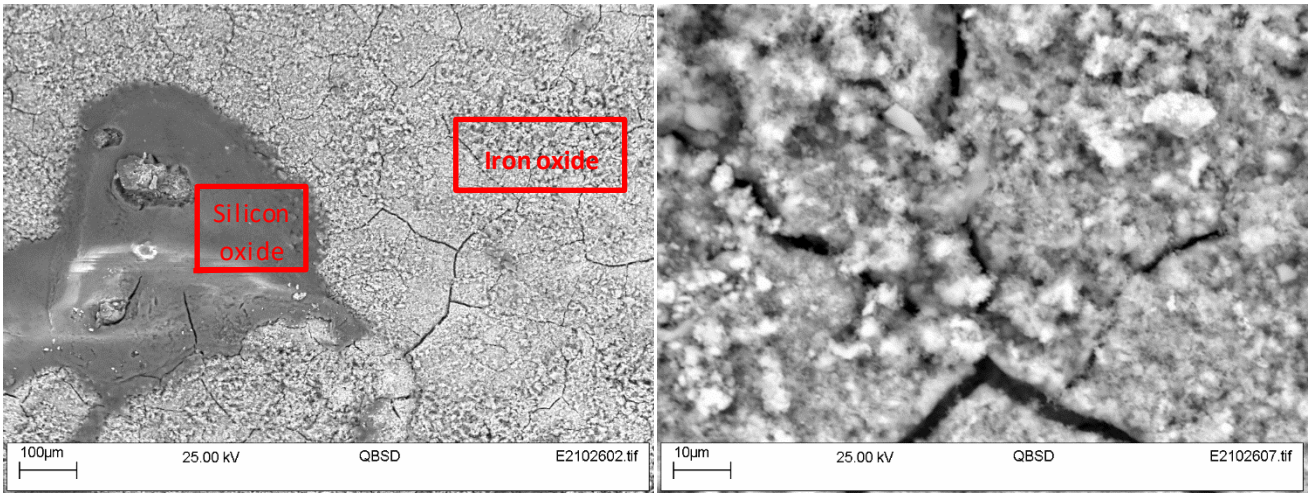


Figure 46 SEM and EDX analyse on top surface of Steel 1 after 7-day testing

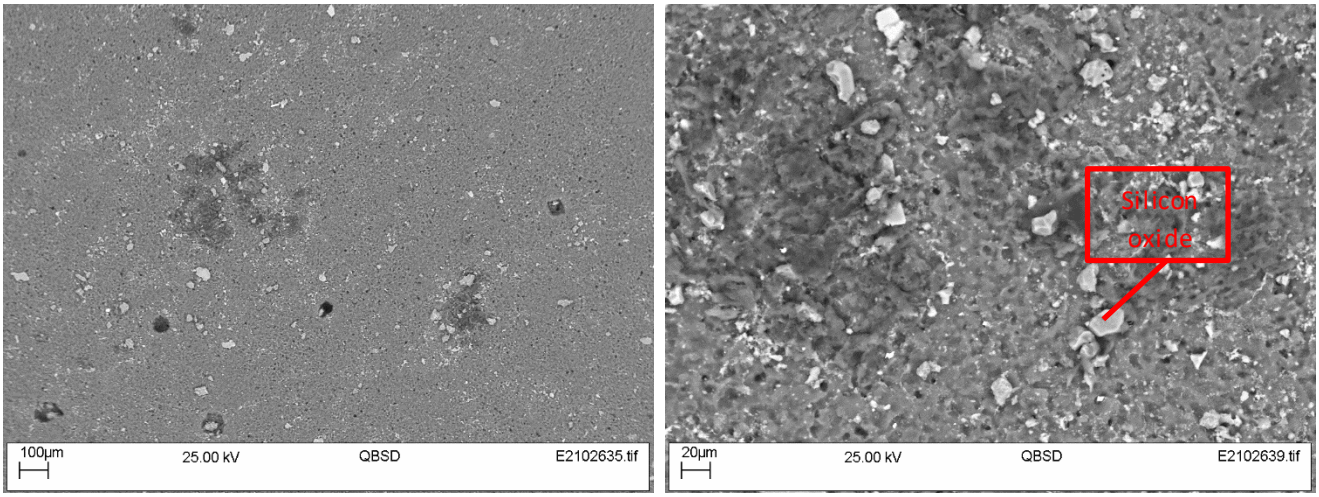


Figure 47 SEM and EDX analyse on top surface of 222Xylan/GO1 after 7-day testing

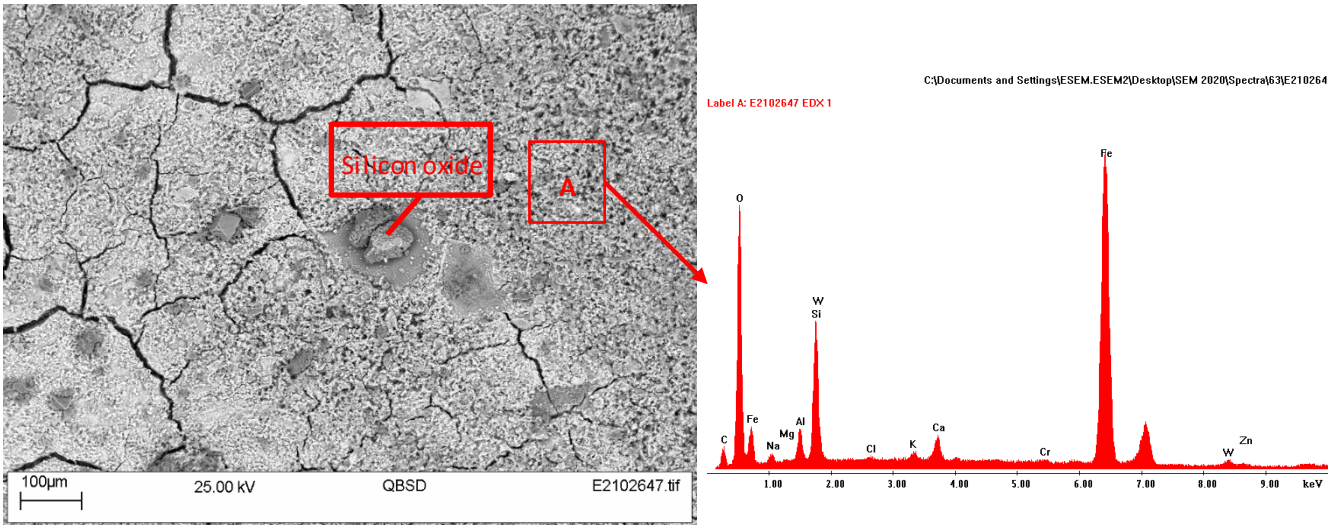


Figure 48 SEM and EDX analyse on top surface of 232WC1 after 7-day testing

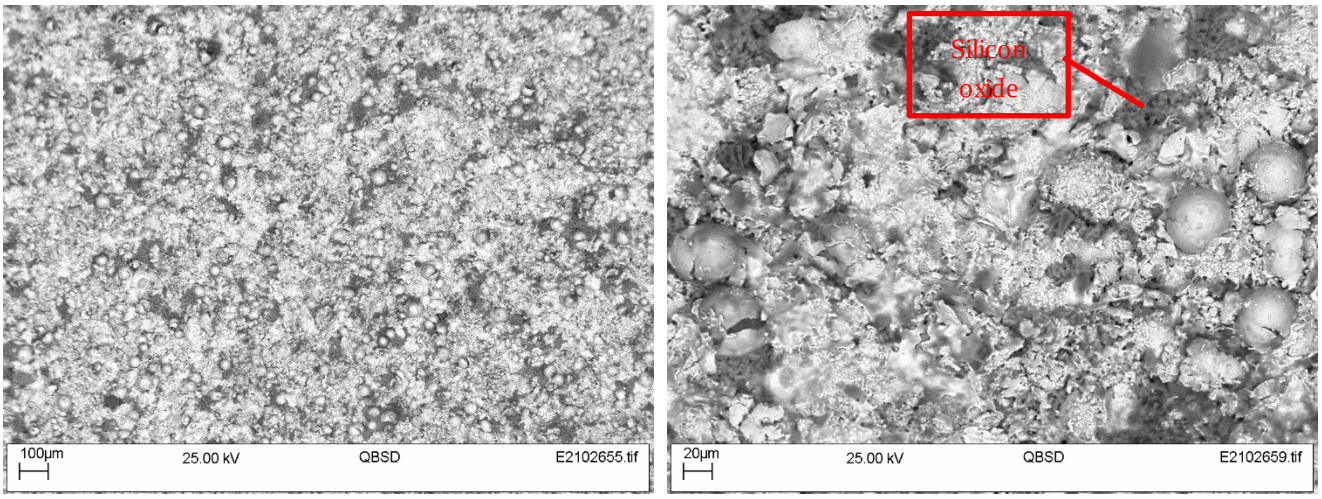


Figure 49 SEM and EDX analyse on top surface of 234Flux1 after 7-day testing

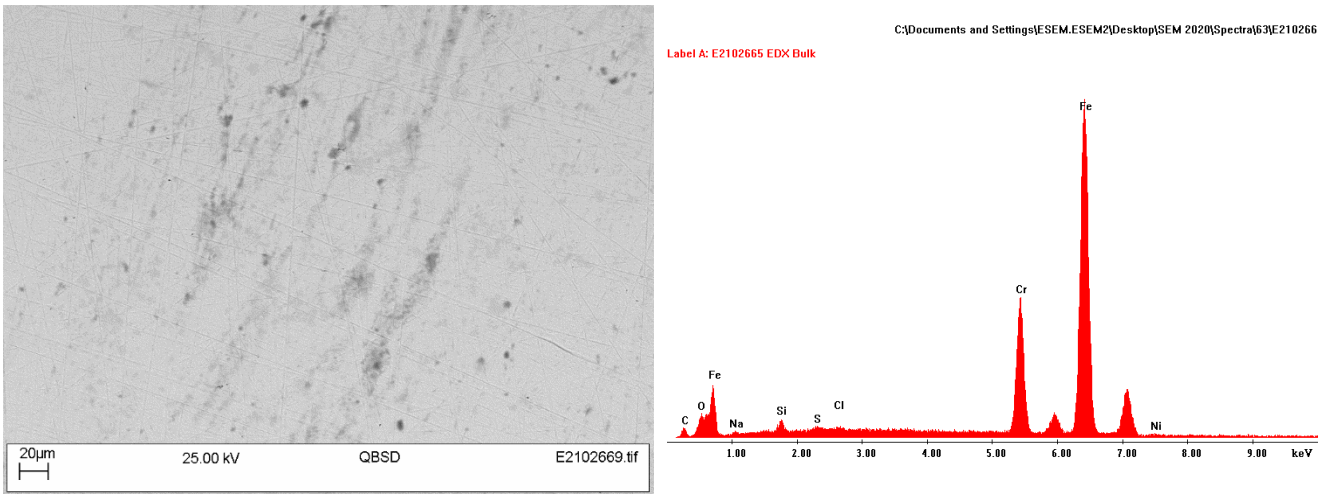


Figure 50 SEM and EDX analyse on top surface of Steel 2 after 7-day testing

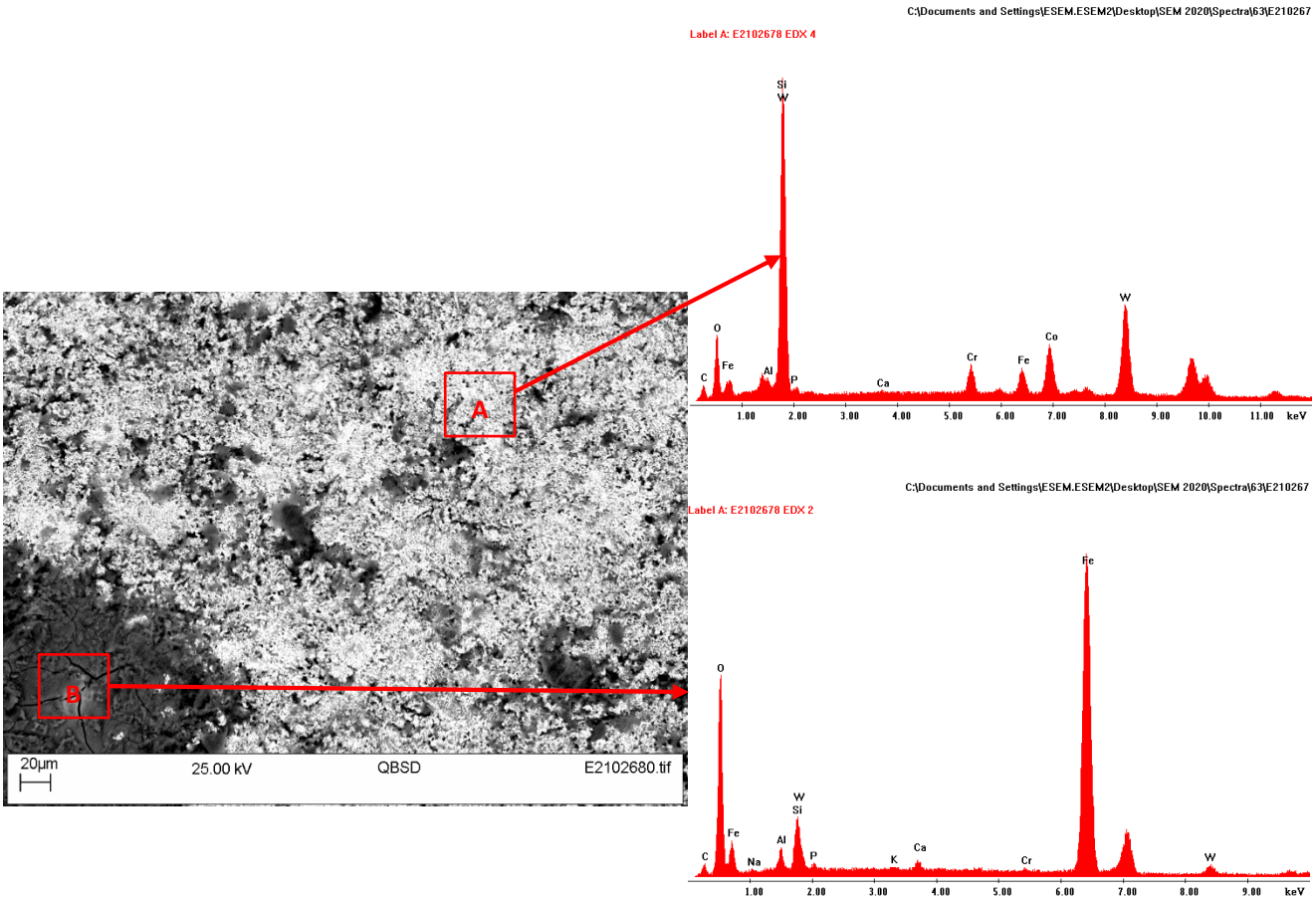


Figure 51 SEM and EDX analyse on top surface of 232WC2 after 7-day testing

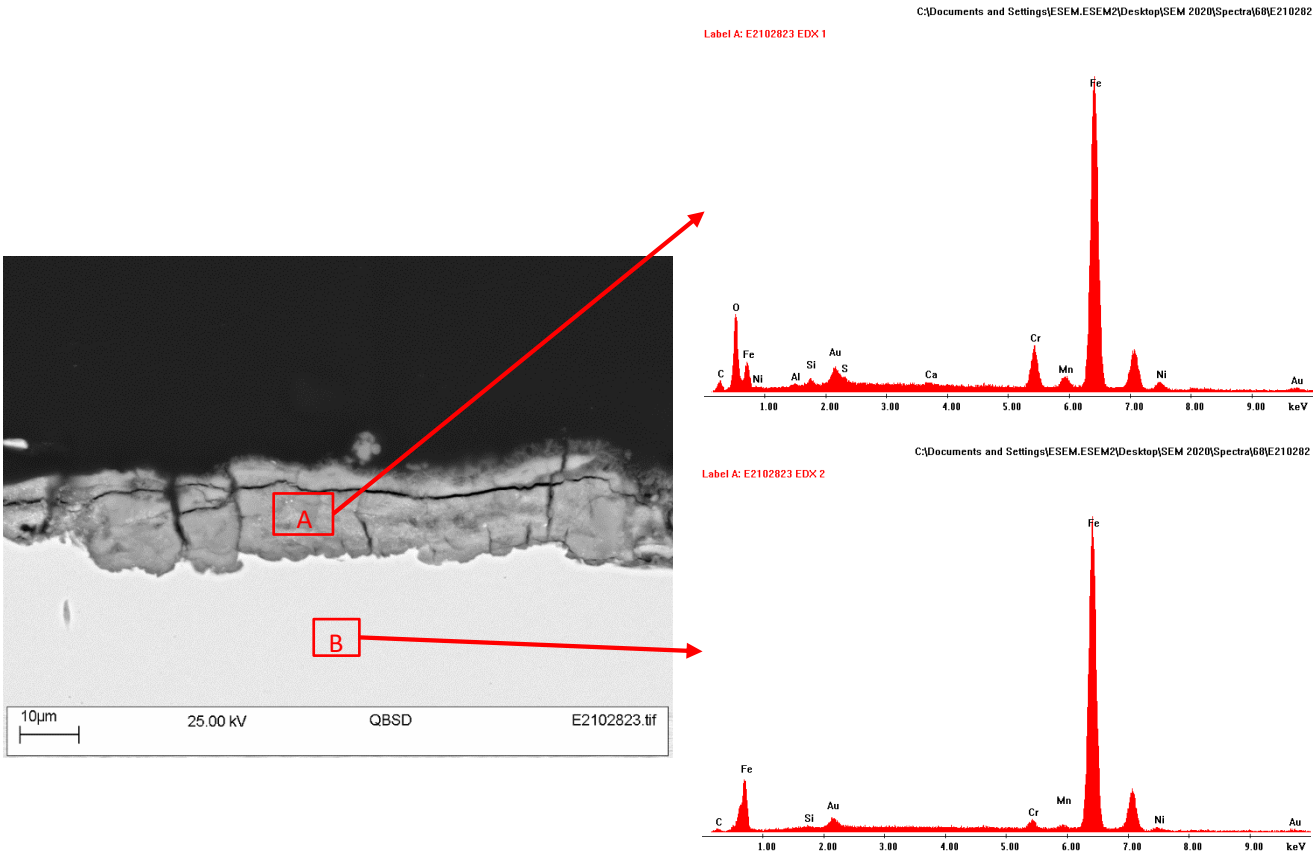


Figure 52 SEM and EDX analyse on polished cross-sectional surface of Steel 1 after 7-day testing

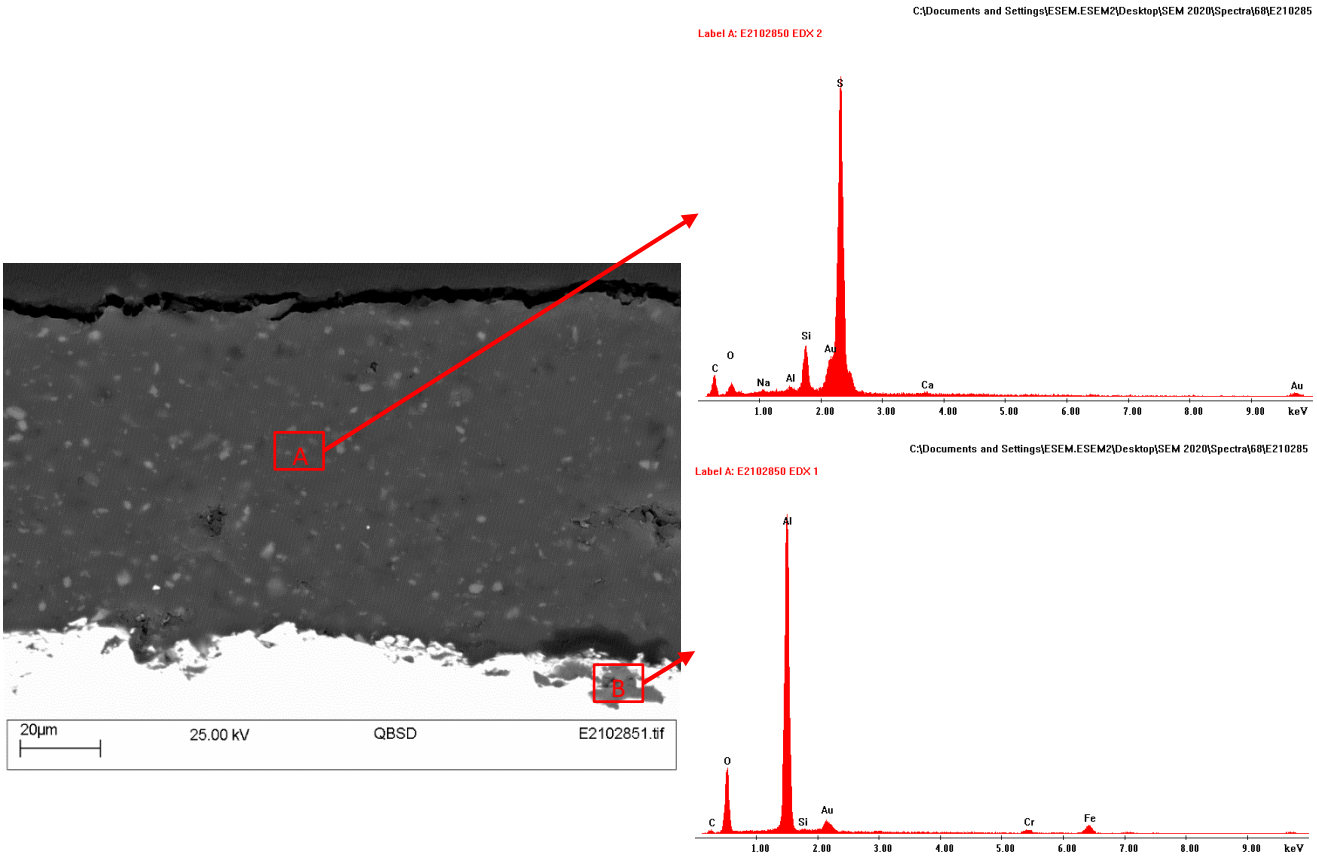


Figure 53 SEM and EDX analyse on polished cross-sectional surface of 222Xylan/GO1 after 7-day testing

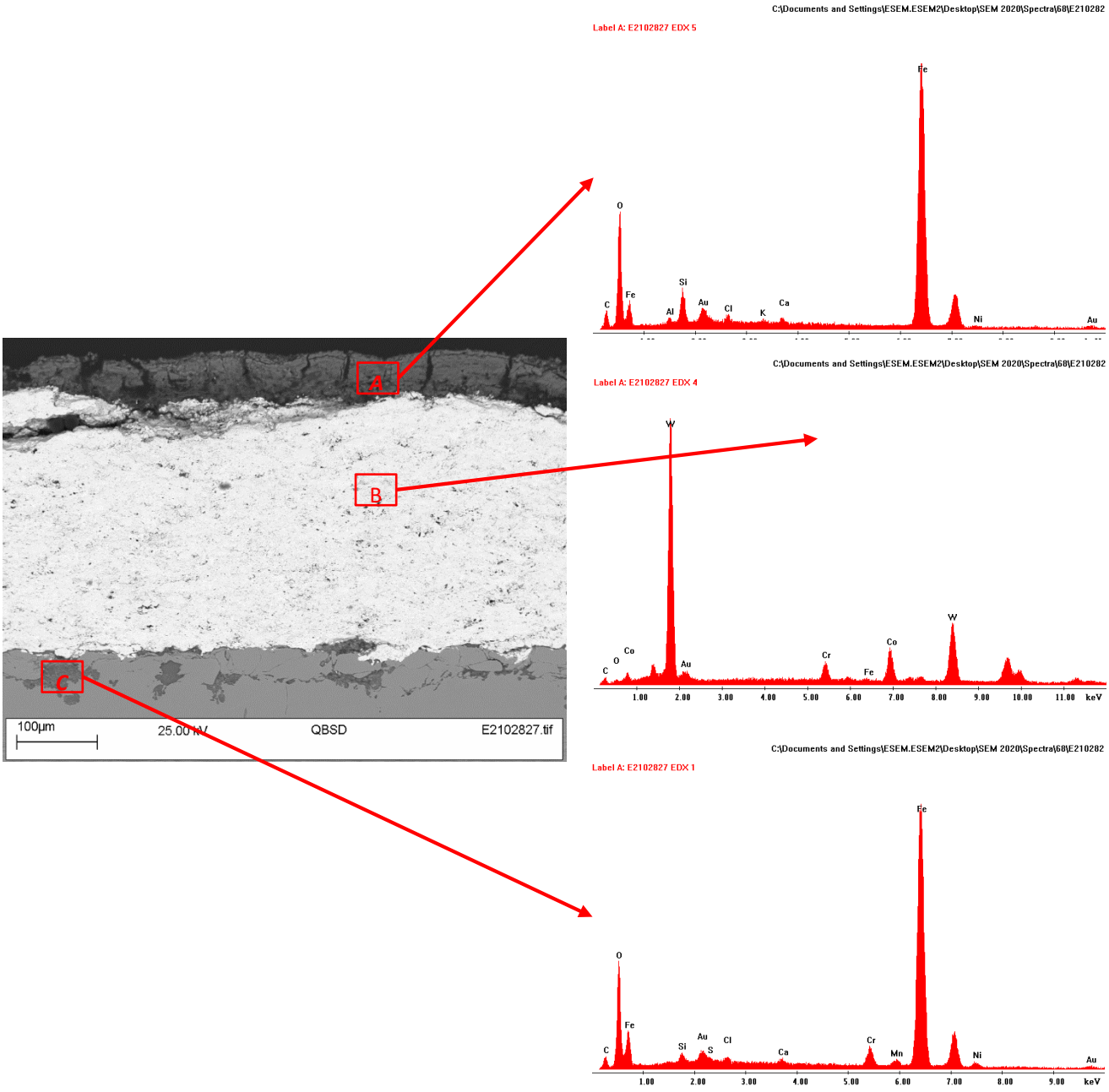


Figure 54 SEM and EDX analyse on polished cross-sectional surface of 232WC1 after 7-day testing

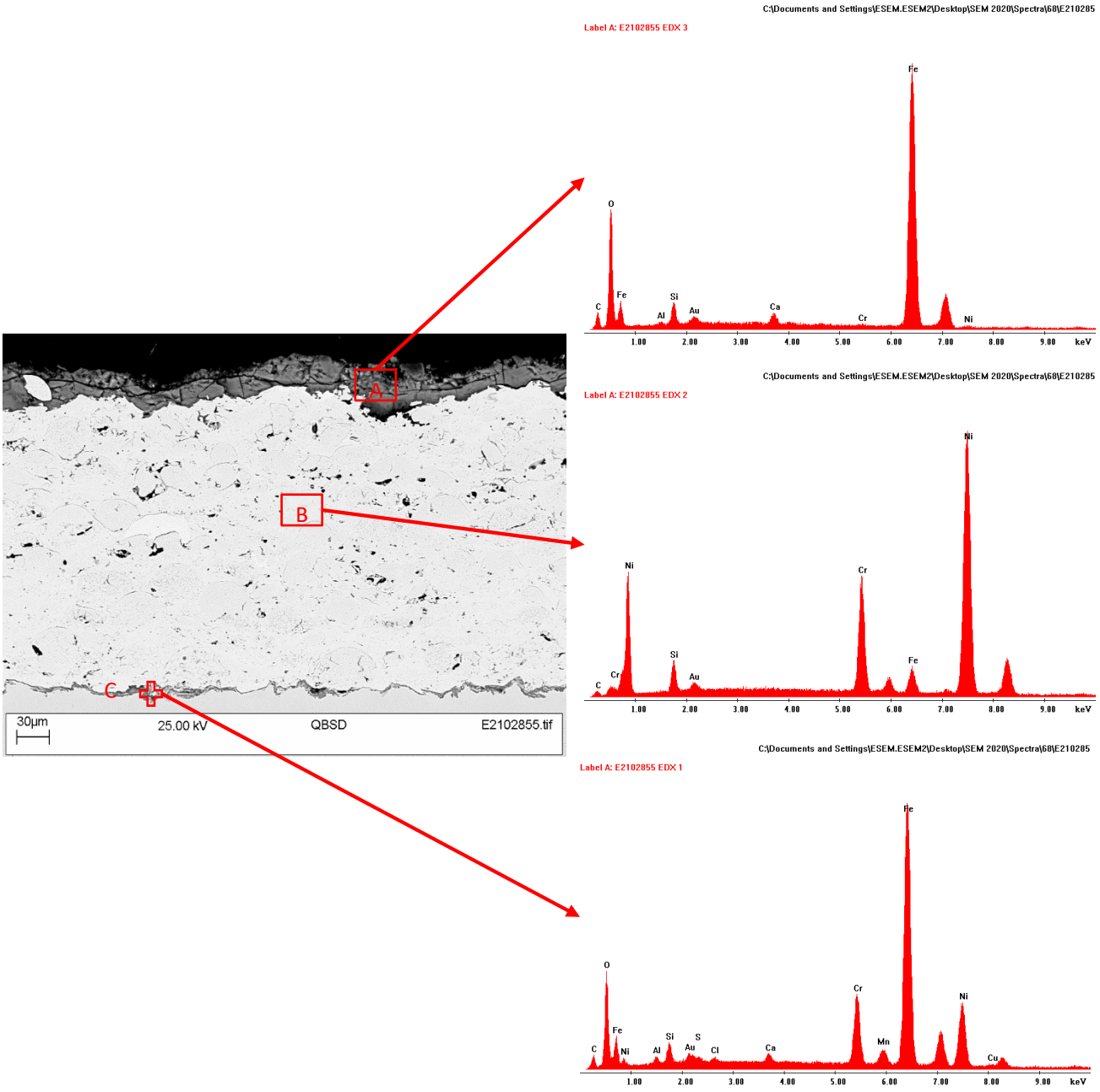


Figure 55 SEM and EDX analyse on polished cross-sectional surface of 234Flux1 after 7-day testing

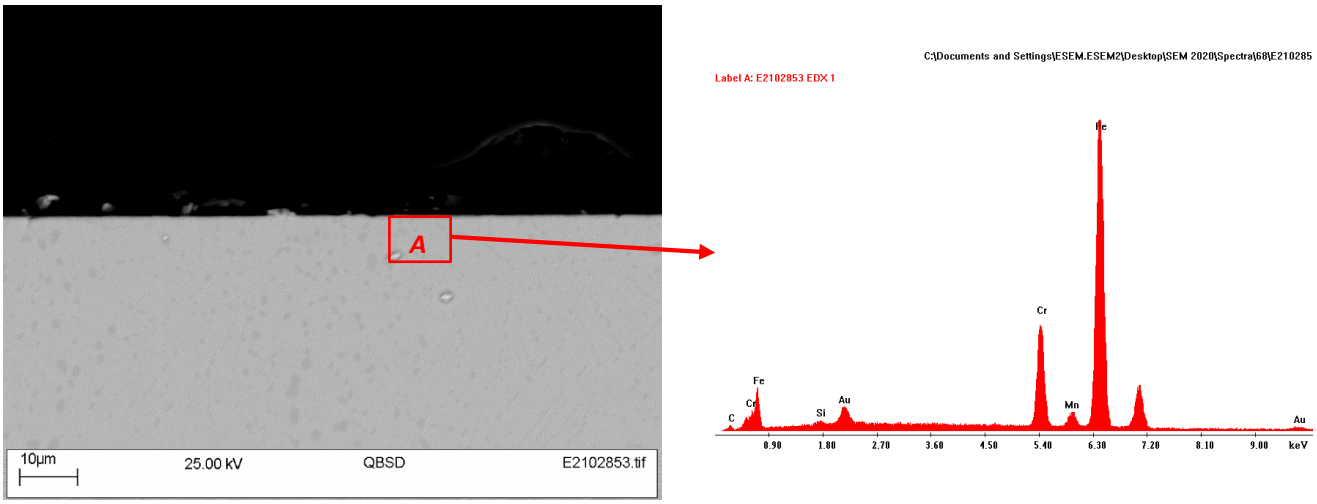


Figure 56 SEM and EDX analyse on polished cross-sectional surface of Steel 2 after 7-day testing

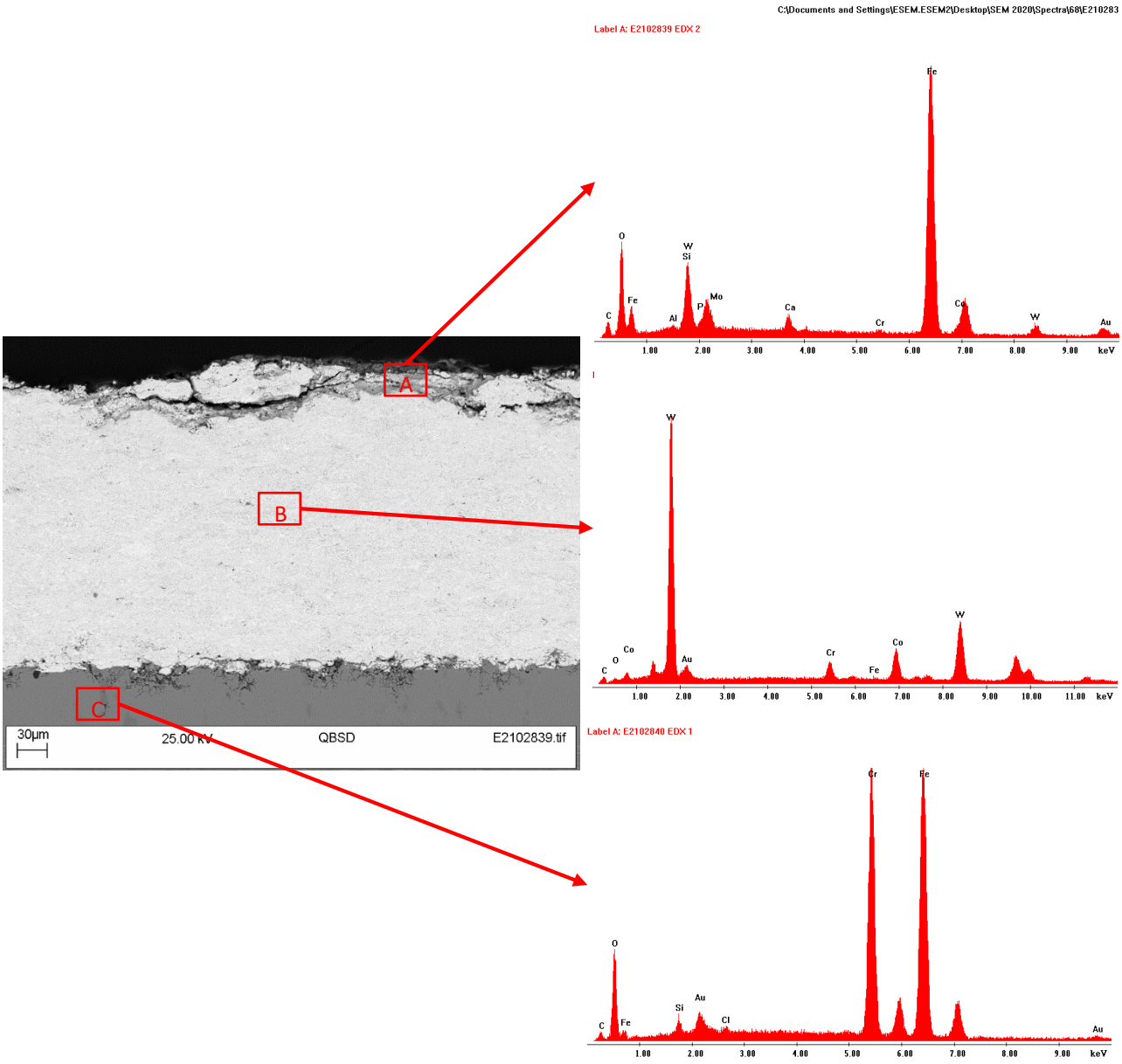


Figure 57 SEM and EDX analyse on polished cross-sectional surface of 232WC2 after 7-day testing

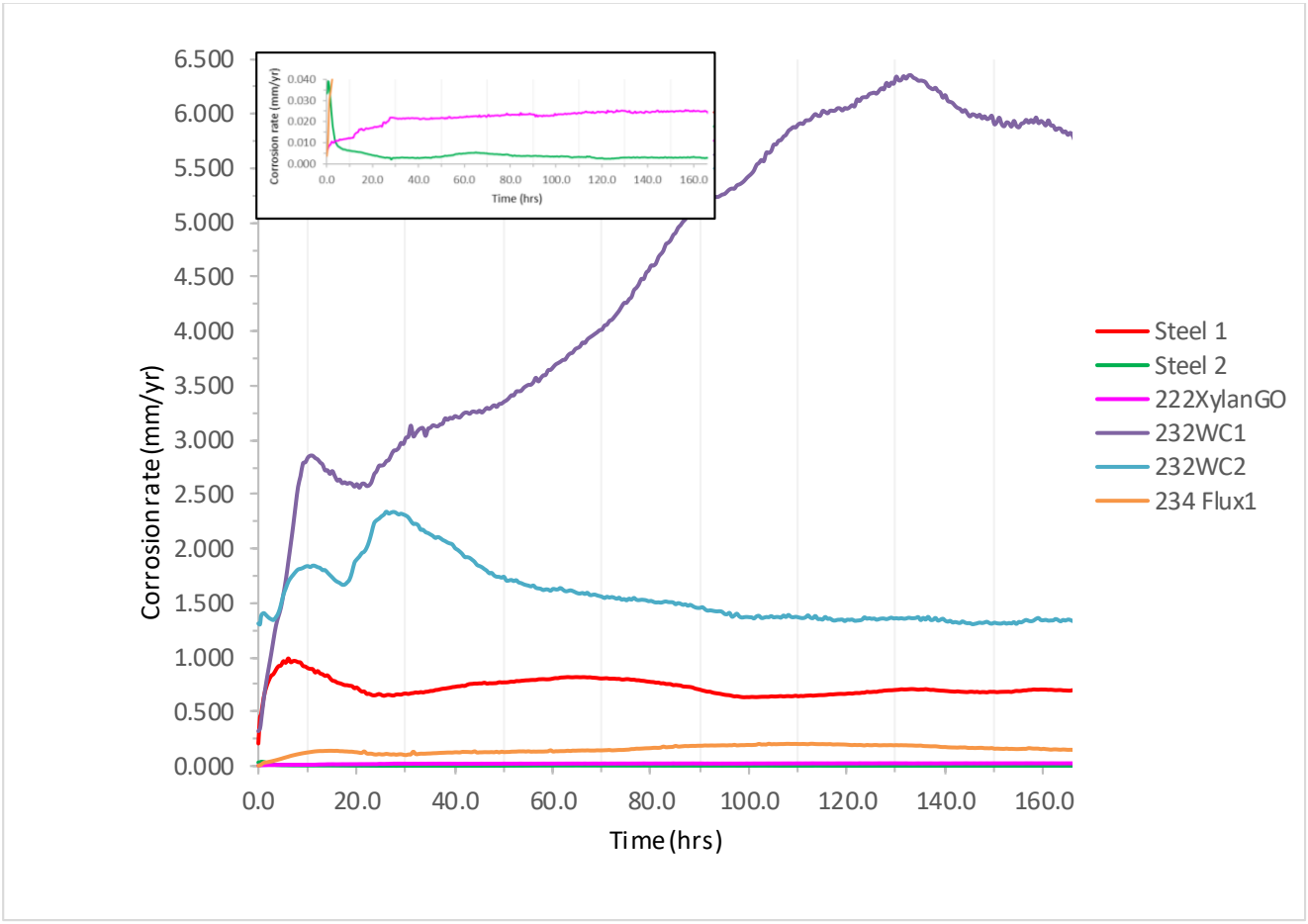


Figure 58 Corrosion rate for Coatings on Steel 3 after 24h testing in simulated drilling fluid