

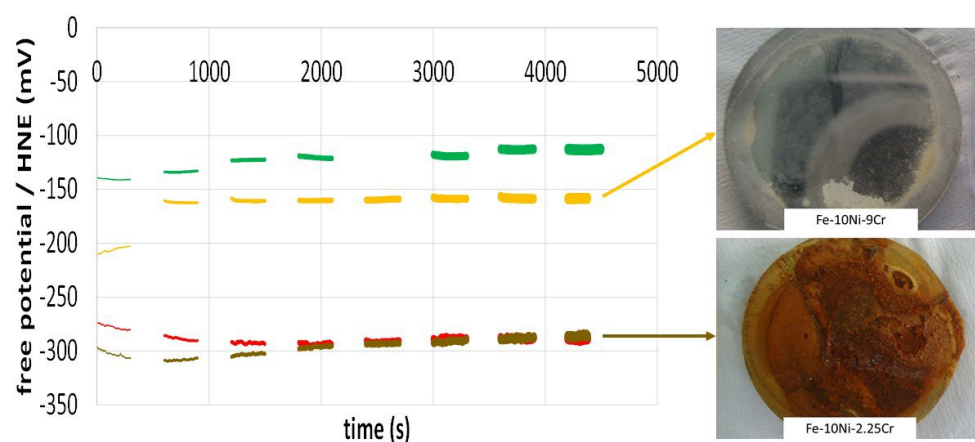
Teaching wet corrosion of metals and alloys: An example of enrichment of the basis practical works with the case of orthodontic steels in artificial saliva

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Abstract: To consolidate the knowledge in corrosion acquired by students during courses and theoretic exercises, practical works are highly important. This is already true when the corrosion behavior of pure metals is explored in a broad variety of simple solutions. This can be strengthened further by applying characterizing methods and sense of observation to more complex problems to make a bridge with professional cases that young graduated may be faced with. In the present work, we considered one of the numerous professional situations in which theoretic and practical corrosion teaching may be involved: to anticipate possible long term corrosion for arch wires made of austenitic stainless steel, to be much more aware of the importance of the contents in key elements as chromium, and also to define pertinent protocols for performing tests representative enough. By elaborating a series of model steels with various Cr contents using the specific means of a laboratory of research in materials sciences, and by preparing – in the practical works room – experimental devices and electrolytes to be closest possible to the real conditions of use of arch wires (buccal environment characteristics: human body temperature, complex saliva composition), a supplementary experiment conducted in parallel to the usual series of practical works may also contribute to justify/legitimate further the training in the anticipation of corrosion problems and the possible ways for their limitations.



Keywords: corrosion practical works, orthodontic arch wire, model steel, artificial saliva, Cr content influence

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1 Introduction

1.1 General

Corrosion is a particularly important problem for the sustainability of metallic structures and components, with huge economic consequences for industries (Davis, 2010). The typical phenomena and rates of corrosion are thus to be known by metallurgists, materials scientists, mechanical engineers, chemists ... More or less extended corrosion courses are integrated in teaching provided by universities and schools of engineers for general scientific and technical formation or for specializations in solids chemistry and materials science (Mainier et al., 2013) (Brajendra, 2019). Corrosion teaching for Master level generally starts with the introduction or reminders of some fundamental data of chemistry (mainly inorganic; oxidation-reduction, acids and bases...), thermodynamics (chemical potentials, Pourbaix's/E-pH diagrams...), diffusion... After having demonstrated and used many formulas in the frame of directed exercises, the acquired knowledge is consolidated by a series of practical work sessions, e.g. up to ten sessions spread over one or two months in the timetable.

1.2 Usual practical works in corrosion

Different practical workstations can be considered. One can think first to simple experiences consisting in the immersion for 2 to 3 hours, of part of pure metals (Fe, Cu, Zn...) or alloys (carbon steels, austenitic stainless steels) in solutions of various types (from simple water or molar sulfuric acid, to Cr^{VI} or Mn^{VII} very oxidant solutions with various concentrations and with presence of chlorides or not). After slight rinsing and drying, nude eye observations followed by examinations using a binocular magnifier or a low magnification optical microscope, allow concluding that the metallic material is in its immunity, corrosion, or passivation domain, that it is subject or not to pitting or crevice corrosion... or that the substance added to the solution acts or not as a corrosion inhibitor. Measurements of potential and pH can be also carried out to compare results to the corresponding Pourbaix's diagrams (Pourbaix, 1991). At the other side of the technique complexity, other experiences/measurements can be also involved to specify notably corrosion rates: exploiting follow-up of free potential, applying the Stern-Geary (polarization resistances) (Rajagopalan, & Dey, 1976) and Tafel (Bockris, & Reddy, 1977) (accurate values of I_{corr}

and of the Tafel anodic and cathodic coefficients β_a and β_c), performing potentiodynamic or cyclic polarizations, and why not electrochemical impedance spectroscopy (EIS) if the required device is available.

1.3 An idea for a deepening practical work

In addition with all these simple but essential manipulations to experimentally explore corrosion phenomena and kinetics, another type of experiment which can be easily done in parallel is the follow-up of the corrosion process:

- on long time (e.g. a month, rather than a couple of hours),
- of dental ternary alloys chemically close to real alloys (e.g. Fe(base)-Ni-Cr, simplified versions of austenitic steels),
- especially elaborated as a series of alloys with varying content in one element (e.g. Cr, the content of which is of high importance for the corrosion behavior),
- in contact with a solution re-producing the usual working milieu in term of composition (e.g. an artificial saliva),
- and in term of temperature (e.g. 37°C, human body temperature, thus of a buccal milieu).

Such experiments, started at the beginning of the series of practical work sessions, and interrupted near the end of this series, do not take necessary a long time of preparation, and may additionally bring useful knowledge to students in a typical corrosion situation which can be met in the “true life” (beyond pure metals, simple solutions, room temperature only...).

The topic of this article is to describe such new additional longtime practical work recently tested by us, from the detail of realization to some observed results and their interpretations and meanings.

2 Practical work materials needed

2.1 Defining and elaborating the alloys to expose to corrosion

Considered that this long-term practical work is imagined in the dentistry domain, several families of alloys can be concerned, depending on the objective of the dentist. As examples one can cite:

- the “High Noble” alloys (containing more than 60 wt.% of Au + Pt + Pd), “Noble” alloys (containing also precious metals but less than 60 wt.% of Au + Pt + Pd) or “Predominantly Base” alloys (based on Ni or Co, containing 20–30 wt.%Cr and about 10 wt.% of Mo or W, and minor elements as Si, Al...), as parent alloys for the fixed partial dentures (FPDs) (De March et al., 2009);
- Titanium alloys (including the famous TA6V) for dental implants (Nicholson, 2020);
- Beta Titanium and Ni–Ti (shape memory alloys) or stainless steels for archwires for orthodontics (Harini & Kannan, 2020); some of them are presented in Figure 1.

Figure 1. Some examples of commercial orthodontic arch wires available on the market.



Due to the high resistance to low temperature wet corrosion of their base elements, the “HN”, “N” and even “PB” alloys for FPDs and the Ti or NiTi alloys for implants and archwires are not suitable for observing evident corrosion even after one or two months of immersion. In contrast some modified versions of stainless steels can suit to the present pedagogic goal.

Indeed, the commercial arch wires made of stainless steels are also very resistant against corrosion but versions impoverished in chromium may allow interesting behavior for students. For this practical work we were inspired by the base chemical

composition of the arch wire presented in Figure 1 (left side, “Brand A”): Fe(bal.) – 9.01 Ni – 19.07 Cr – 1.05 Si – 0.32 Ti (all contents in wt.%) (Amini et al., 2012). Instead Fe(bal.) – 9Ni – 19Cr we finally choose Fe(bal.) – 10Ni – 18Cr. This is still very close to the “Brand A” arch wire and, in addition, more familiar for our students who know the AISI 304 and AISI 316L that they work on during the regular practical work sessions.

Six alloys were elaborated especially for this practical work. Their names and their targeted chemical compositions are displayed in Table 1, with a reminder line containing the reference commercial alloy. From the Fe-10Ni-18Cr alloy representing a simplified version of this real arch wire stainless steel acting as reference, three new compositions were defined by dividing per 2 the chromium content, from on ally to the following one (18 → 9 → 4.5 → 2.25). A Cr – free alloy was also added to complete the list and to allow eventually revealing a possible influence of the 10 wt.% Ni by comparison with pure Fe.

Table 1. Chemical compositions of the commercial alloy and of the six model alloys prepared for the longterm practical work.

<i>Weight contents (Fe: bal.)</i>	<i>Ni</i>	<i>Cr</i>	<i>Other</i>
Brand A stainless steel	9.01	19.07	1.05 Si, 0.32 Ti
Fe-10Ni-18Cr	10	18	/
Fe-10Ni-9Cr	10	9	/
Fe-10Ni-4.5Cr	10	4.5	/
Fe-10Ni-2.25Cr	10	2.25	/
Fe-10Ni-0Cr	10	0	/
Fe-0Ni-0Cr	0	0	/

To elaborate these alloys we used a high frequency induction furnace existing in the Institut Jean Lamour laboratory. 20g of each composition prepared from pure Fe, Ni and Cr (Alfa Aesar, more than 99.9% of purity) were accurately weighed (precision of the balance: $\pm 0.1\text{mg}$). Metals mix were placed in the water-cooled copper crucible of the furnace (Figure 2 left), the fusion chamber of which was thereafter closed by placing a silica tube between the crucible and the surrounding induction

coil. By increasing the 110kHz current voltage up to 5kV, the induced Foucault's currents produced heat in the metals and fusion was completed after about 2 minutes (Figure 2 right). The levitating molten alloy was maintained during 10 minutes to be sure that it was chemically homogenized. Progressive decrease in voltage input allowed liquid state cooling, solidification and solid state cooling. The 20g obtained ingots (Figure 3) were extracted from the furnace, half an hour later.

Figure 2. After having placed the Fe, Ni and Cr metallic parts in the copper crucible (left), induction heating in a pure Ar atmosphere induced the melting of the mix (right).



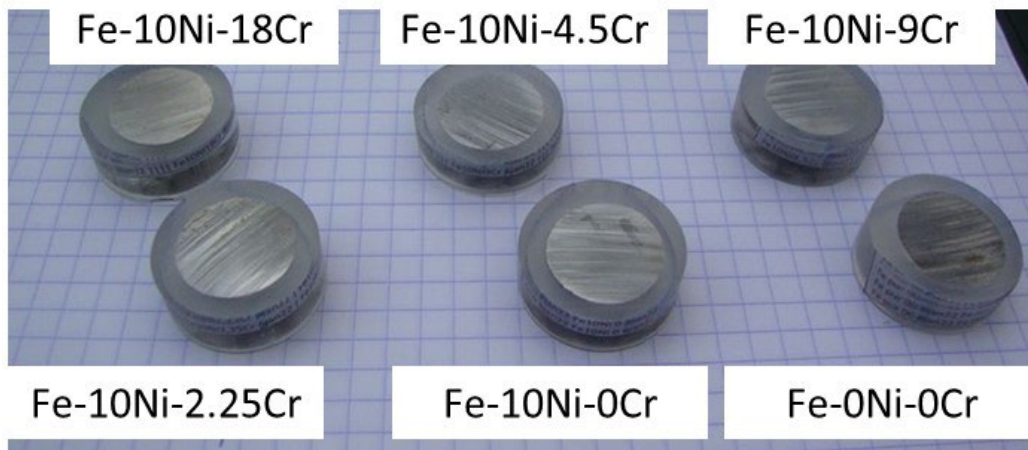
Figure 3. The six ternary alloys produced as 20g–weighing ingots.



Due to their particular shape not easy to maintain in the vice of the metallographic saw, all ingots were separately embedded in a cold resin mixture to obtain

cylinders containing them. Cutting was then possible and each of them was cut in two parts. One part was used for the chemical composition control (Energy Dispersive Spectrometer equipping a scanning electron microscope, present at the Institut Jean Lamour laboratory), and thereafter for the corrosion test (Figure 4).

Figure 4. The part per cut embedded ingot which was devoted to the corrosion tests.



The second part of each ingot was slightly sawn to allow inserting the denuded extremity of a plastic-gained electrical wire. Re-introduced in the rigid plastic mold a small quantity of additional liquid mix of resin hardener was poured to immerse the denuded **copper (Figure 5)**, to electrically isolate it from artificial saliva when this sample will be used as electrode for some electrochemical experiments (here: free potential follow-up to anticipate the corrosion results after one month of immersion).

Figure 5. The second parts of the ingots prepared as working electrodes.



2.2 Defining and preparing the artificial saliva; start of the test

The aqueous solutions which are usually used to simulate saliva are numerous. Among the simplest ones there is the {pH=7.4, 9g.L⁻¹ NaCl} one, and among the most complex ones there is the Fusayama–Meyer one (Table 2), which we propose use as an example, for students, of electrolyte much more complex than simple aqueous solutions. Preparing the Fusayama’s artificial saliva may be a good exercise for them. If some products are not available, this can be also a good exercise for students to calculate the masses of CaCl₂ (instead CaCl₂·2H₂O) or KH₂PO₄ (instead NaH₂PO₄·2H₂O) to add for replacing the missing products.

Table 2. Chemical composition of the artificial saliva to prepare for the corrosion test (+ rating of the pH to 5 by addition of droplets of HCl 0.1M or NaOH 0.1M).

<i>Fusayama’s saliva</i>	
KCl	0.4 g × L ⁻¹
NaCl	0.4 g × L ⁻¹
CaCl ₂ ·2H ₂ O	0.906 g × L ⁻¹
NaH ₂ PO ₄ ·2H ₂ O	0.690 g × L ⁻¹
Na ₂ S·9H ₂ O	0.005 g × L ⁻¹

After a last control of the initial pH of each cell containing an immersed sample (Figure 6), the cells were hermetically closed (to avoid water evaporation during the long duration of the immersion test) and placed in an oven (Figure 7) with constant temperature settled at 37°C.

Figure 6. pH control of the solution of each cell in which one of the samples will be immersed.

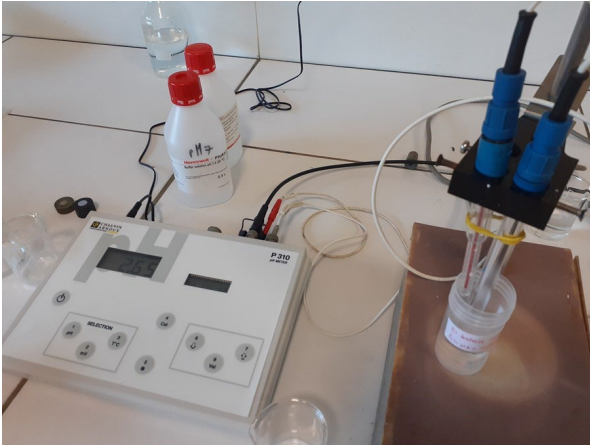


Figure 7. The six alloys immersed in six separated cells (left) and their introduction in the oven for the one month stay at 37°C (right).



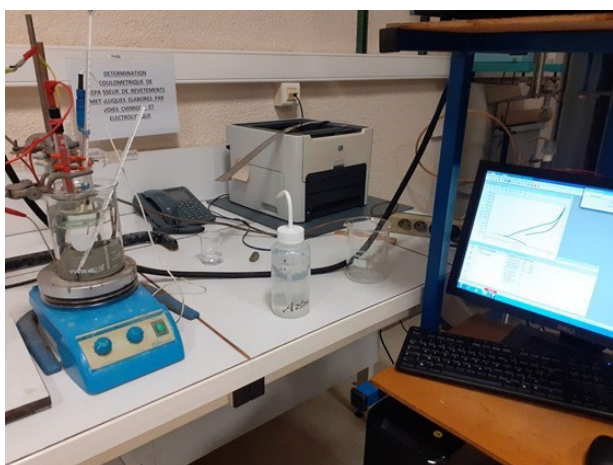
2.3 Materials for possible other tests which can be done in parallel

Prior to the start of the immersion corrosion tests, or in parallel with these tests as a specific practical work station, electrochemical experiments can be done by students to anticipate the results of the immersion tests. A first measurement which can be done, simply with a voltmeter or multimeter (with a recording function if possible) and a Calomel electrode for the reference of potential, is the follow-up of the potential of the electrode prepared at the same time as the sample for immersion (Figure 6). Comparison of the free potential of the electrode with E–pH diagrams (Pourbaix's diagrams) may allow anticipating the state of the alloy by the one of each of its constitutive elements (immunity, active state or passive state of Fe, Ni, Cr) and, why not, the oxidant species involved in the corrosion phenomenon (H^+ , dissolved O_2 ...). Further, if a potentiostat is available in the practical works room (Figure 8), recording the successive values of free potential may be easier, and its evolution versus time can be visualized in real time (e.g. progressing passivation when seeing an

increasing free potential, loss of the passive state when observing a fall of free potential...). In addition, periodical determination of the polarization resistance (R_p measured according the Stern–Geary method) and the determination of the Tafel coefficients β_a and β_c in order to obtain useful indications for the identification of the main species involved in the anodic reaction and in the cathodic one. Such Tafel run can be, for instance carried out after 1 hour of immersion, followed by the stopping of the test because of the modifications induced by Tafel experiment to both electrode and electrolyte.

Of course, it is compulsory to heat the artificial saliva at 37°C and maintaining it at this temperature, using not a professional apparatus (too easy), but by using cleverly the materials present in the practical work room. Thus, an additional work for student is conceiving something able to guarantee 37°C for electrolyte and electrodes for the short time necessary to perform the electrochemical measurements. A possible solution is visible on the left in Figure 8: a water bath made of two Bechers fit together, with, between the two, tap water heated by an electrical hot plate, the inner becher containing electrolyte and electrodes. The potentiostat (here a Versastat model from Ametek) driven by the VersaStudio software) is visible on the right of Figure 8.

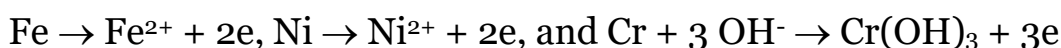
Figure 8. The {three electrodes} cell (left) conceived as a water bath (electrolyte and electrodes immersed), and a potentiostat driven by a software hosted in a computer (right).



3 Examples of results

3.1 Potential follow-up of the free potential measured with a multimeter

An example of free potential evolution is given in Figure 9 for the four ternary alloys considered in this work (from Fe-10Ni-18Cr to Fe-10Ni-2.25Cr). From the relative positions of the four curves, students may think that the Fe-10Ni-18Cr and Fe-10Ni-9Cr alloys are possibly in a passive state and while that the Fe-10Ni-4.5Cr and Fe-10Ni-2.25Cr ones are in an active state. Such hypotheses must be confirmed by consulting the Pourbaix's diagrams of Fe, of Ni and of Cr. If students place, in these Pourbaix's diagrams, the {pH, E} points corresponding to these four alloys they would obtain Figure 10 (Fe), Figure 11 (Ni) and Figure 12 (Cr). They are all in the corrosion domain of iron (stability of Fe^{II}: Fe²⁺), in the corrosion domain of nickel (stability of Ni^{II}: Ni²⁺) and in the passivation domain of chromium (stability of Cr^{III}: Cr(OH)₃ or Cr₂O₃). So, it can be expected that students propose, as the anodic reactions which can be anticipated for all alloys, the following ones:



Concerning the cathodic reactions, since the free potentials are all close to the H₂O/H₂ equilibrium, and much lower than the O₂/H₂O one, students ought to identify dissolved oxygen as the principal oxidant ($\frac{1}{2} \text{O}_2 + 2\text{H}^+ + 2e \rightarrow \text{H}_2\text{O}$). If a potentiostat is available, this can be confirmed by a Tafel experiment and the values obtained for the Tafel anodic coefficient (which depends on the number of exchanged electrons).

Figure 9. The evolution of the free potentials of the four alloys over more than an hour in the Fusayama's artificial saliva at 37°C.

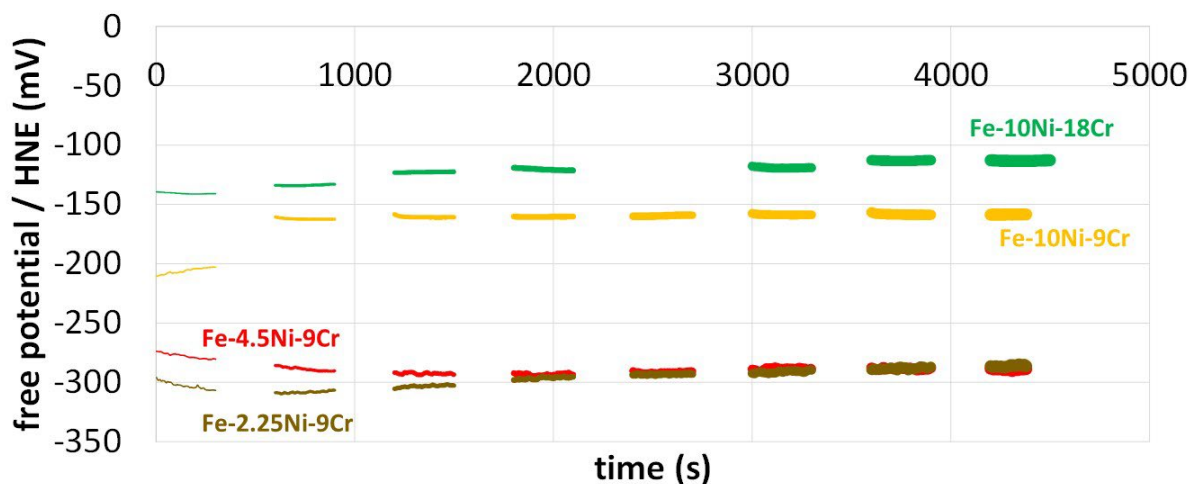


Figure 10. Positions of the last recorded values of the free potential in the Pourbaix's diagram of Fe.

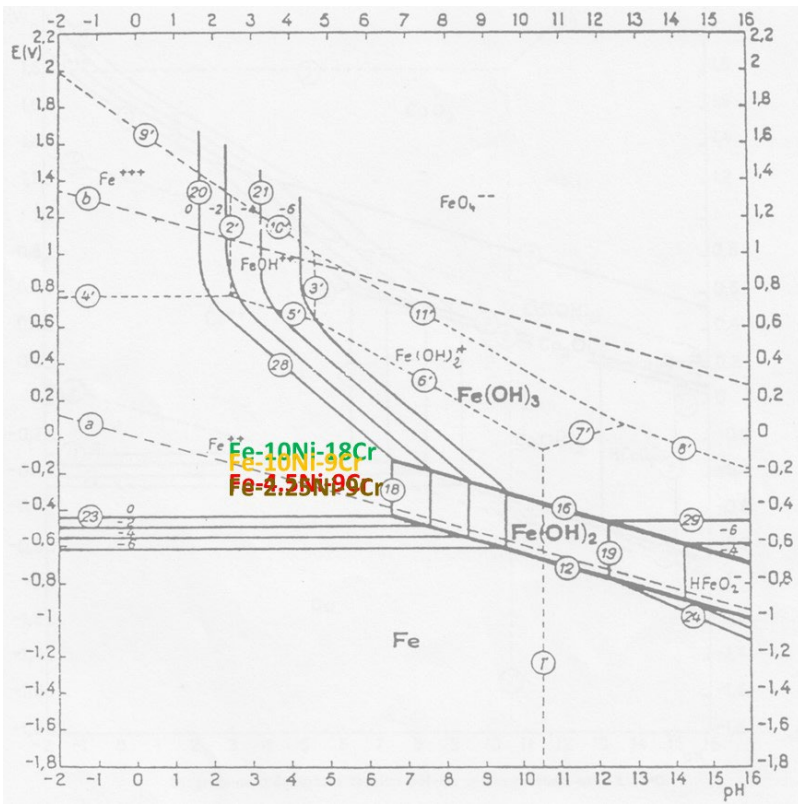


Figure 11. Positions of the last recorded values of the free potential in the Pourbaix's diagram of Ni.

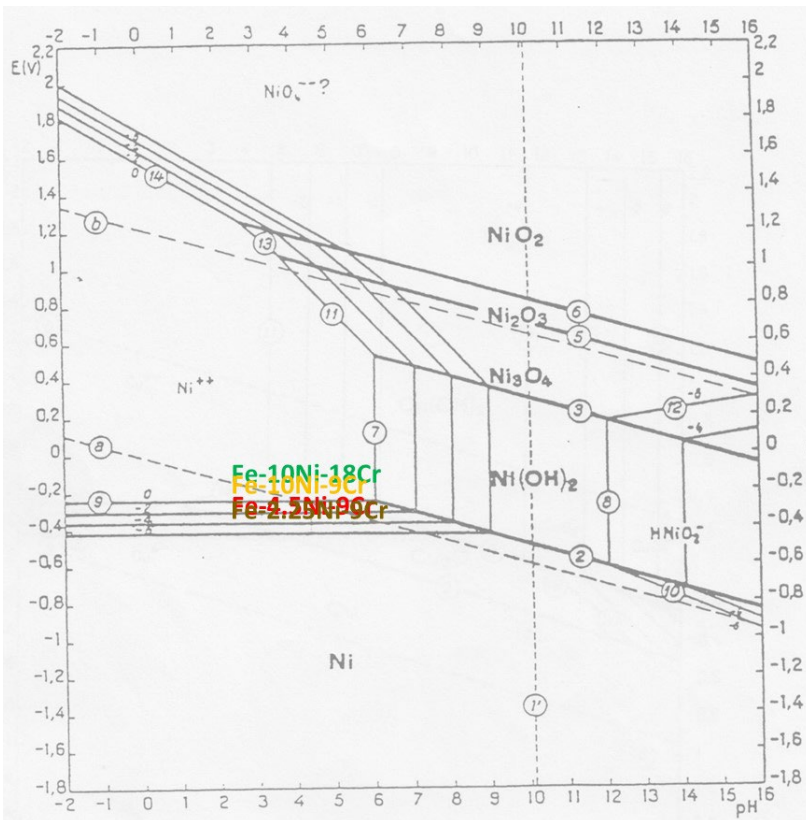
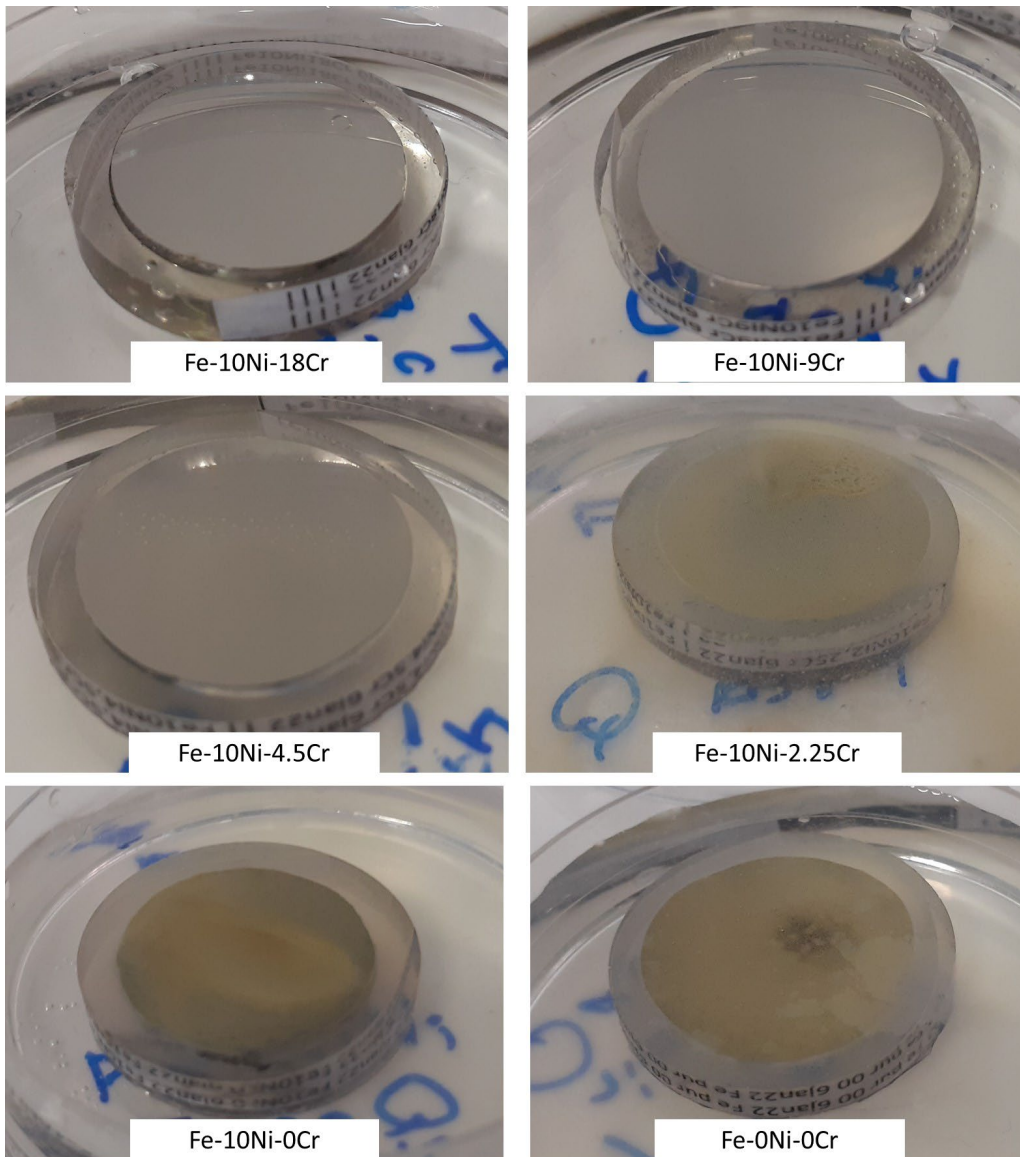


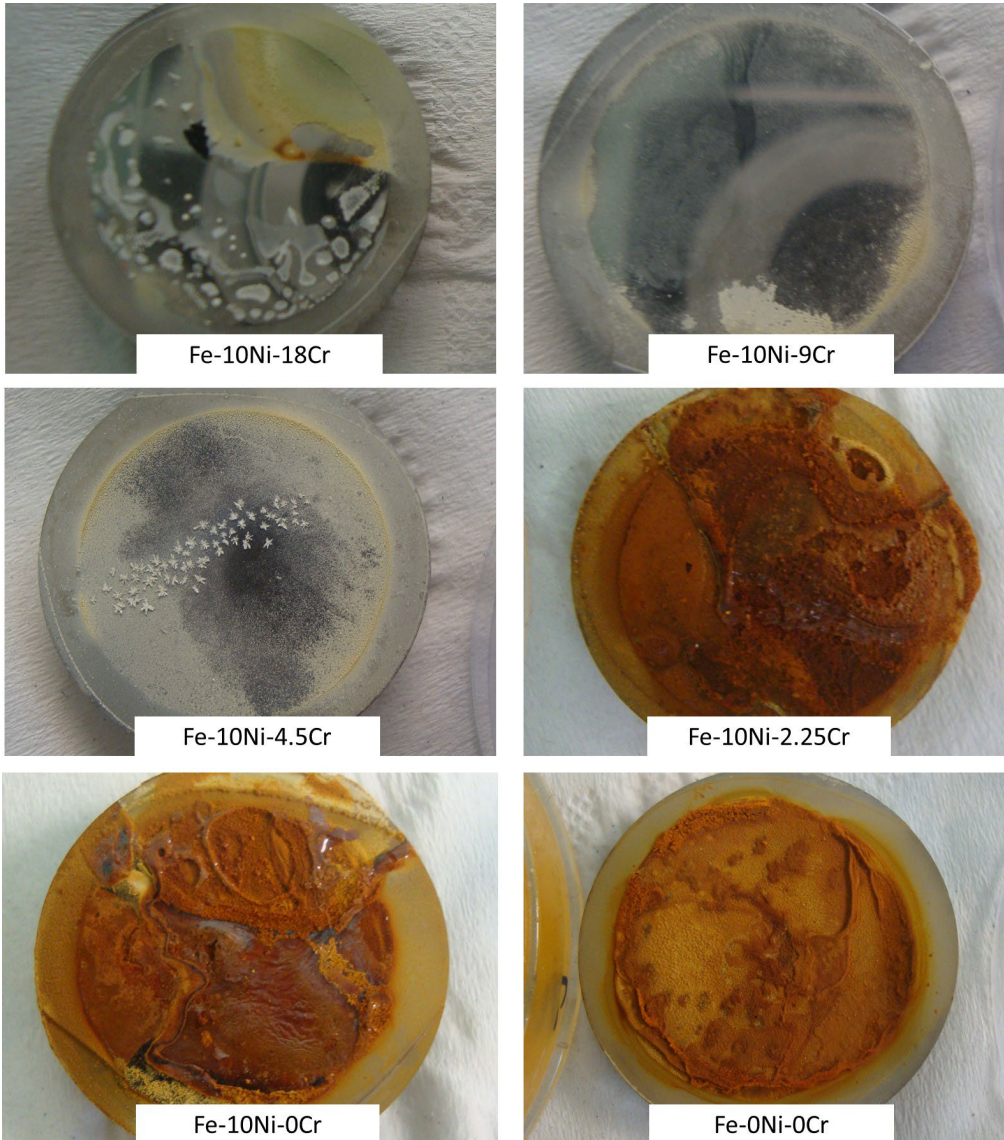
Figure 13. Surface states of the samples after four days of immersion in Fusayama's saliva at 37°C.



After these four days the students may notice that the surfaces of the Fe–10Ni–2.25Cr, Fe–10Ni–0Cr and Fe–0Ni–0Cr alloys are obviously affected by corrosion (presence of brown corrosion products). The three chromium–richest samples are themselves seemingly covered by a thin pale white layer. For longer immersion times these phenomena intensify and after one month it is clear that the Fe–10Ni–2.25Cr, Fe–10Ni–0Cr and Fe–0Ni–0Cr are severely corroded (Figure 14): thick rust scale is now present on the surface of these samples and student may observe that rinsing with tap water does not allow removing these corrosion products, in contrast with the three chromium–richest alloys (Fe–10Ni–18Cr, Fe–10Ni–9Cr and even Fe–10Ni–4.5Cr). Considering these last samples, deposits formed by precipitation

due to solvent (water) evaporation, even limited, must be distinguished from real corrosion products.

Figure 14. Final surface states of the samples after one month of immersion in Fusayama's saliva (37°C).

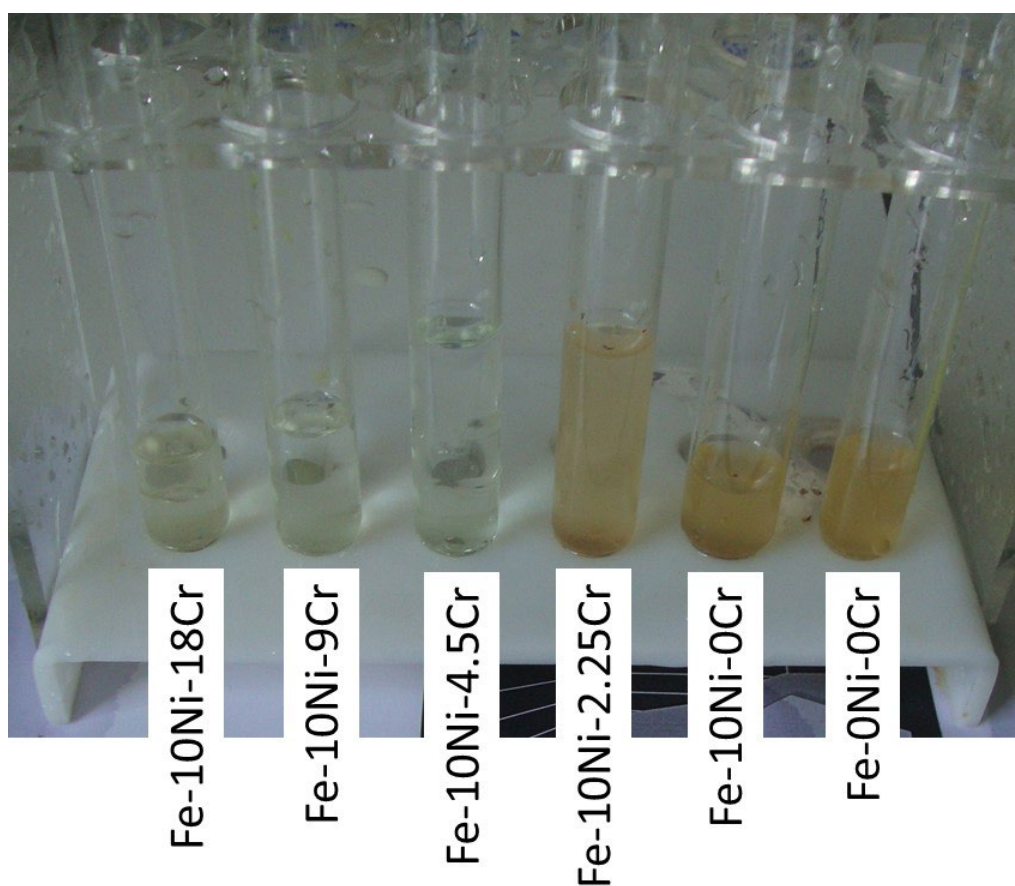


3.3 Characterization of the corrosion products

At the end of the {1 month}–immersion the artificial saliva remaining in the cell and the water of sample rinsing can be collected to carry out analysis for identifying the species present in the electrolyte. Since Fe ions are the best markers of corrosion, one can add some droplets of $K_3Fe(CN)_6$ or $K_4Fe(CN)_6$ to the collected electrolytes in each of the six test tubes. Students may imagine the result: blue coloration can be

expected in case of presence of Fe^{2+} ions in the solutions, especially for the electrolytes present in the cells which contained the Fe–10Ni–2.25Cr, Fe–10Ni–0Cr and Fe–0Ni–0Cr samples (according to the visual observations done concerning the surface states of these samples. The clear electrolytes corresponding to the Fe–10Ni–18Cr, Fe–10Ni–9Cr and Fe–10Ni–4.5Cr did not turn blue (left side of Figure 15) which can be understood (no corrosion, thus no formation of ferrous anions). But the same observation is done concerning the rust–orange electrolytes corresponding to the Fe–10Ni–2.25Cr, Fe–10Ni–0Cr and Fe–0Ni–0Cr samples (right side of Figure 15), which is much more surprising.

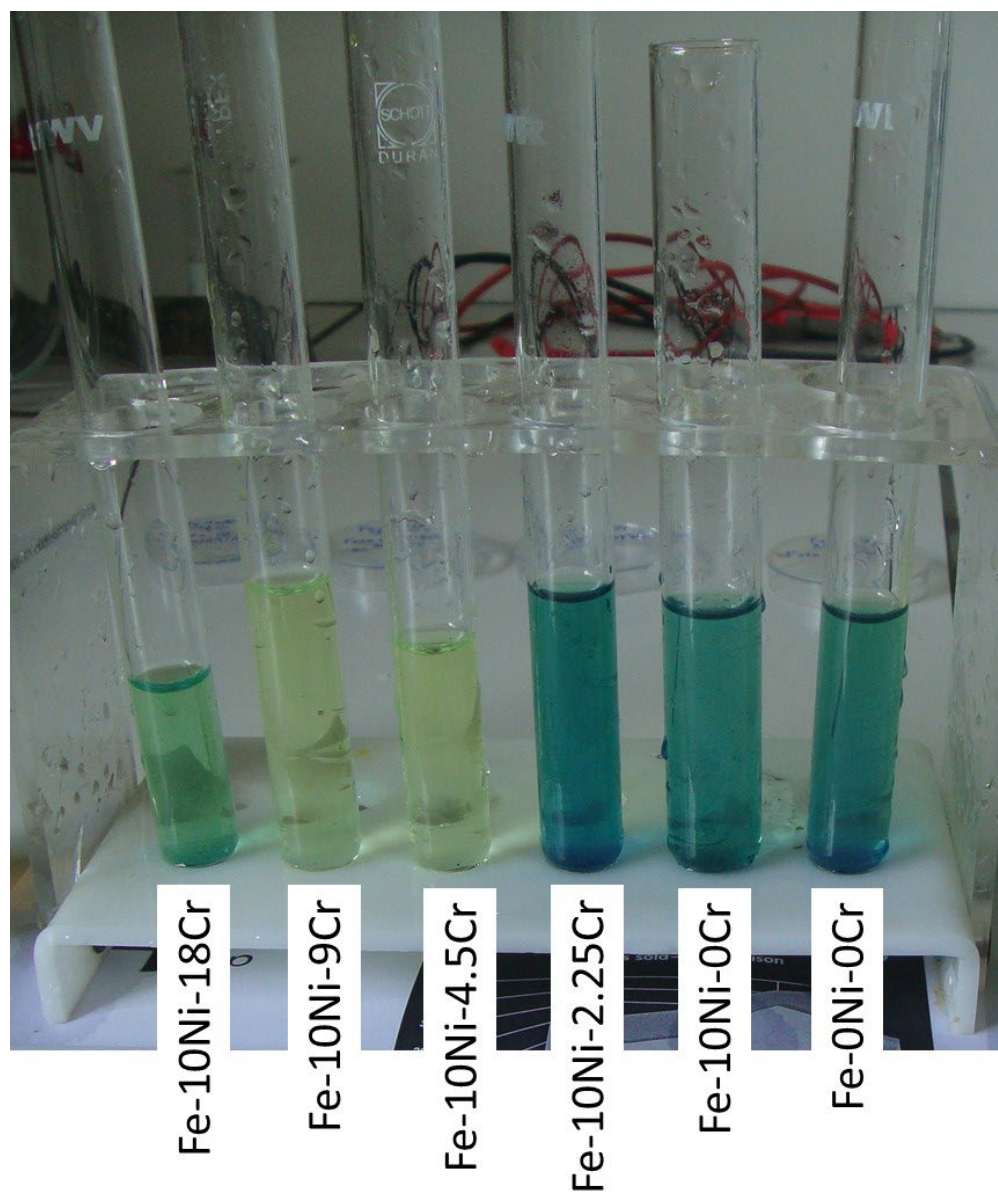
Figure 15. The series of test tubes containing the electrolyte collected in the six cells, after addition of $\text{K}_3\text{Fe}(\text{CN})_6$ or $\text{K}_4\text{Fe}(\text{CN})_6$.



By measuring the pH of the solutions and after having decided to acidify them, students may obtain the coloration of the solutions by formation of the complex ions (Turnbull's blue or Prussia's blue): this is thus successful. Blue colorations demonstrate the presence of ferrous ions in the electrolytes corresponding to the Fe–10Ni–2.25Cr, Fe–10Ni–0Cr and Fe–0Ni–0Cr samples (right side of Figure 16), while no blue coloration occurs for the electrolytes corresponding to the other samples,

except for the Fe–10Ni–18Cr sample, curiously, for which slight blue coloration can be noted (left side of Figure 16). For this later sample, it is true that a slight rust tint appeared locally on the {1 month}–immersed sample surface, close to the interface with resin (top left macrograph in Figure 14).

Figure 16. The series of test tubes containing the electrolyte collected in the six cells, after addition of $K_3Fe(CN)_6$ or $K_4Fe(CN)_6$ followed by acidification.



The previous manipulations can be made with not expensive materials and products. Of course, if a X-ray diffractometer or a Scanning Electron Microscope equipped with an Energy Dispersion Spectrometer is available not far from the

practical work room, using them can allow identifying the corrosion with certainty and thus the samples which were really affected by corrosion.

4 Potential Benefits for the formation of the students

By preparing and starting such a long term experiment in parallel with the series of practical work sessions (Figure 17), students have the opportunity to apply their corrosion teaching contents to a concrete situation more complex than the useful – but necessarily limited – elemental tests involving simple alloys and simple solutions in simple conditions.

Figure 17. Example of possible organization of the series of corrosion & protection practical work sessions with inclusion of the long term immersion test and its exploitation

Session I: All students: preparation of the samples, the artificial saliva, the cells and the oven; start of the immersion tests; design of the apparatus for the practical work station F (how maintaining 37°C for during the short tests scheduled in PW station F)

	Practical work station A e.g. 3 hours immersion of Fe samples in various solutions* followed by observations	Practical work station B e.g. galvanic coupling of different metals and alloys in various solutions*	Practical work station C e.g. Fe and other metals without or with anodic, cathodic, or mixed inhibitors	Practical work station D e.g. application and tests of various protective coatings (metallic or not)	Practical work station E e.g. Stern-Geary, Tafel, electro-chemical impedance spectroscopy (potentiostat)	Practical work station F Predictive short tests in 37°C artificial saliva: free potential measurement and comparison with E-pH diagrams, Tafel plots (potentiostat) ... & control, observation and photography of the current surface states
Session II	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6
Session III	Group 6	Group 1	Group 2	Group 3	Group 4	Group 5
Session IV	Group 5	Group 6	Group 1	Group 2	Group 3	Group 4
Session V	Group 4	Group 5	Group 6	Group 1	Group 2	Group 3
Session VI	Group 3	Group 4	Group 5	Group 6	Group 1	Group 2
Session VII	Group 2	Group 3	Group 4	Group 5	Group 6	Group 1

*: in terms of pH (very acid, acid, neutral, basic, very basic), of nature of oxidant species (acids, Cr^{VI}, Mn^{VII}, N^V, O₂ aerated or desaerated), and of concentrations in oxidant species, other types of important species such as Cl⁻...

Session VIII: All students: exiting the long term immersed samples from the oven, delicate rinsing of the corroded samples, photography (XRD, SEM, EDS if available), final pH measurement of the six collected volumes of aged artificial saliva... interpretation of the results, conclusions

In the specific case of the additional practical work given as example in the previous pages, the benefit for students are:

- discovering the complexity of the chemical compositions of alloys really employed by comparison with pure metals (here: austenitic stainless steels based on Fe, Ni and Cr, and containing additionally minor elements and trace elements, as well as of the complexity of some model electrolytes (by comparison to sulfuric acid, sodium chloride aqueous solutions, simple buffer solutions...))
- alloys involved in a domain that many of them well knew in a recent past or well known in their personal situation: arch wires for orthodontic treatment (very frequent, nowadays)
- the necessity to simplify problems to keep good chances to understand what occurs in the phenomena of interest (here: ternary simple Fe–10Ni–18Cr alloy for simulating the real steels employed for arch wires, and Fusayama's saliva to reproduce natural human saliva)
- staying modest and aware of the partial representativity of the test, by evocating the limits of representation of the model alloy (minor and trace elements as well as the necessarily plastically hardened state of arch wires) and of the model saliva (human saliva possibly contains additional species, and bacteria, oxygenation, acidity and temperature may evolve depending of the activity of the child or of the adult: food, beverage, sport...)
- working out experimental apparatus and methods using the available materials (here: a homemade water bath system for rating the electrolyte at human body temperature for the electrical measurements, an oven for long duration immersion tests)
- succeeding in an indirect (identification of Fe^{2+} in solution with limited material, the reagents present in the practical work room) or a direct (e.g. XRD or SEM if available) characterization of the corroded states
- discovering in practice that some simple and rapid measurements (free potential) and comparisons with thermodynamic data (Pourbaix's diagrams) may allow anticipating long term results
- of course confirming the importance of the presence of chromium and of its content on the resistance against corrosion on long term
- and observing that Ni, despite its slightly higher nobility than iron, fails in improving corrosion resistance of iron even if it is present with content as high as 10 wt.%.

5 Conclusion

The organization of practical works session of classical sessions but with inclusion of a project dimension opening toward a concrete more complex case may enhance the motivation of students. Indeed, on the one hand, they consolidate their base knowledge, and on the other hand, they have the possibility to apply this theoretical and technical knowledge to a case looking much more to a real corrosion problem (which, further, may have important consequences since concerning human health: risk of release of Fe^{2+} ions, and certainly of Ni^{2+} too, in the buccal milieu). This can be done with more or less geometries depending on the materials available in the practical work room or in its neighborhood.

To finish, one can also say that the practical work supervisor can use this discovery of a threshold in Cr content to respect for avoiding corrosion, to explain to students that similar problem of critical content in chromium (but also in aluminium, in silicon ...) exist also in other fields, for instance for superalloys. These high-performance metallic alloys, which are designed for applications at 1000°C and beyond, must contain at least 20 wt.% Cr for nickel-based superalloys, or at least 30 wt.%Cr for cobalt-based superalloys, to resist corrosion by molten mixtures of sulfates and to oxidation by hot gases (as this occurs in aeronautic or power generation turbines). In this case, students may usefully associate passive state to chromia-forming behavior (very slow hot corrosion) and active state to fast hot corrosion of the whole alloy. Such extrapolations are opportunities to open minds to problems that can be difficult to discover in classical practical works.

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