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New Corrosion Monitoring Probe Combines ER, LPR, HDA, Floating B-constant, Electrochemical Noise and Conductivity Measurements

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ABSTRACT

Electrochemical measurement techniques have been extensively investigated in laboratories and universities. For corrosion monitoring in the field on a large scale, the linear polarization resistance (LPR) technique normally is used. Several initiatives have been taken to extend LPR measurements with other electrochemical techniques in order to get more reliable information and enable to measure besides uniform corrosion, localized corrosion such as pitting, crevice corrosion and stress corrosion cracking.

Especially in the oil and gas industry, the Electrical Resistance (ER) technique is being used widely. ER probes measure metal loss very accurately at sensitivity up to 0.01 µm. Both ER and electrochemical technologies are combined with installation and evaluation of corrosion coupons. This paper describes the combination of the two technologies ER and electrochemical in one probe. The advantage of this technology is that use of corrosion coupons may be redundant if required. Evaluation of corrosion coupons is time consuming and retrieval may be dangerous. The combined probe will allow measuring the following signals online:

- Uniform corrosion by LPR with floating B-constant and harmonic distortion analysis (HDA).
- Uniform corrosion (metal loss) by ER.
- Localized corrosion (pitting, crevice corrosion and stress corrosion cracking) by electrochemical noise (EN).
- Conductance.

Key words: corrosion monitoring, LPR, HDA, electrochemical noise, ER, electrical resistance, 2" corrosion probes, 2" access fittings, coupons.

INTRODUCTION

In the industry, especially in the oil and gas one, corrosion monitoring is used on a large scale. The annual market is estimated at >USD 100.000.000,-. The most used are probes and coupons in accordance with the 2-inch access fitting system. Such probes and coupons can be retrieved during operation (under pressure). Probes and coupons are often placed nearby each other in two access fittings. Retrieval and analysis of coupons is very time consuming and can be dangerous. Modern probes which combine several measuring techniques and operate online to the DCS (process computer) sometimes can make use of coupons redundant as noise measurements can detect localized corrosion. Besides, combined probes and coupons have been developed. Such combinations make a second 2-inch access fitting redundant.

This paper describes the ER and electrochemical measuring principles and shows the advantages on the use of combined probes.

ELECTRICAL RESISTANCE CORROSION MONITORING

The basic principle of electrical resistance measuring is very straightforward:
The electrical resistance of the corroding element is compared to a similar element that is not corroding but at similar temperature. In this way, resistance changes caused by temperature changes are being eliminated. Figure 1 shows the basic circuit for ER measurements:
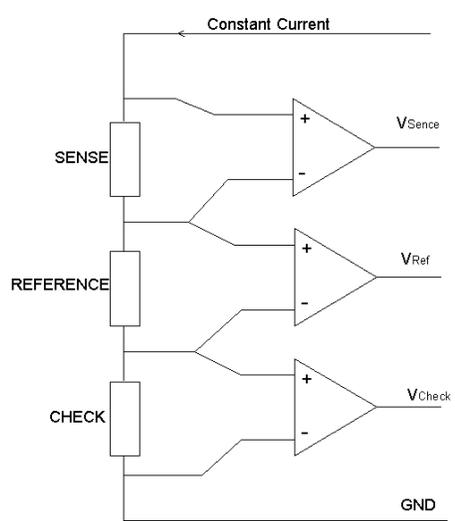

Figure 1: Basic circuit for ER measurements. In practice the three elements are one piece of metal, e.g. a steel strip for a flush probe (figure 2). The sense element is exposed to the corrosive medium and the reference and check element are covered by the resin in the probe body.

ER probes are made in large variety of configurations however the 2-inch probes for the retrievable 2-inch access fittings are most widely used. The sensing element can be of flush, tubular or wire type. ER technology also is used for non intrusive corrosion monitoring. In that case, the pipe or vessel wall itself functions as 'corroding element' . However, the thicker the element, the lower will be the measuring

resolution. For probes the element thickness is 0.25-5 mm. Normally, non-intrusive ER technology has a relatively lower resolution (1/1000 of the wall thickess) because the wall is thicker than an intrusive probe element. Figure 2 shows a combined probe with one ER element and 3 bar-shaped elements for electrochemical measuring (LPR elements).

Also buried ER probes are used for monitoring the outside of coated buried pipelines under cathodic protection. The function of the probes is to monitor the cathodic protection system and influences of AC currents and other stray currents from railroads, electricity transport and other cathodic protection systems.

Reference element: embedded

Check element: embedded

Sensing element: Corroding medium
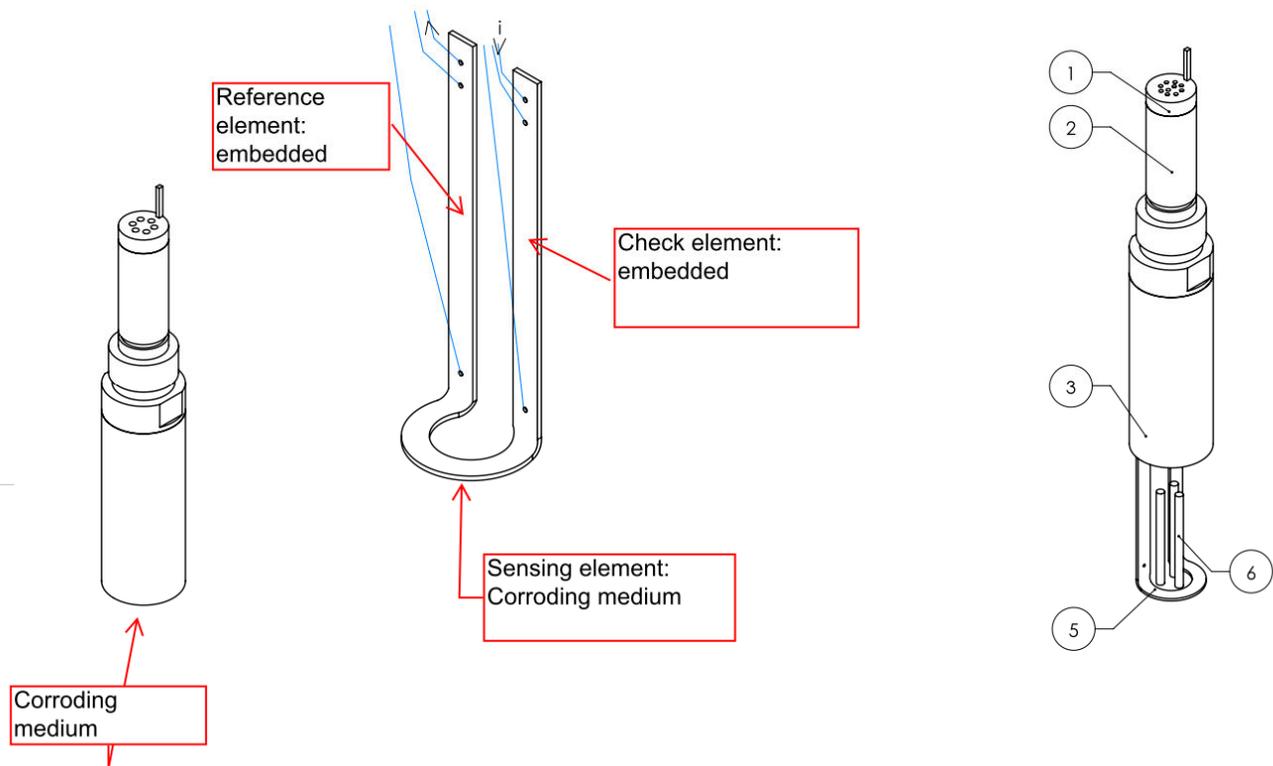
Corroding medium

Figure 2: LEFT: Combined 2-inch high pressure probe for ER (6 wires) and electrochemical measurements (3 wires). RIGHT: exploded view. Item 1: connector (9 pin). Item 2 and 3: probe body in stainless steel AISI 316. Item 6: electrochemical electrodes. Item 5: ER element. MIDDLE: further explanation of ER element. [4]

Figure 3 shows an example of data output for a 2-inch high pressure probe installed in a pipe for injection water on an oil platform. On November 09 2011 the probe element had been corroded 336 μm and no significant metal loss occurred until November 20. Between November 20 and December 04 the metal loss was 10 μm, which results in a corrosion rate of 0.26 mm/year. By injection of a corrosion inhibitor the corrosion was stopped.

A new high sensitivity online 4-20 mA ER meter was developed. The resolution of this meter for a 0,5 mm thick element is better than 0,05 micrometer. See figure 4.
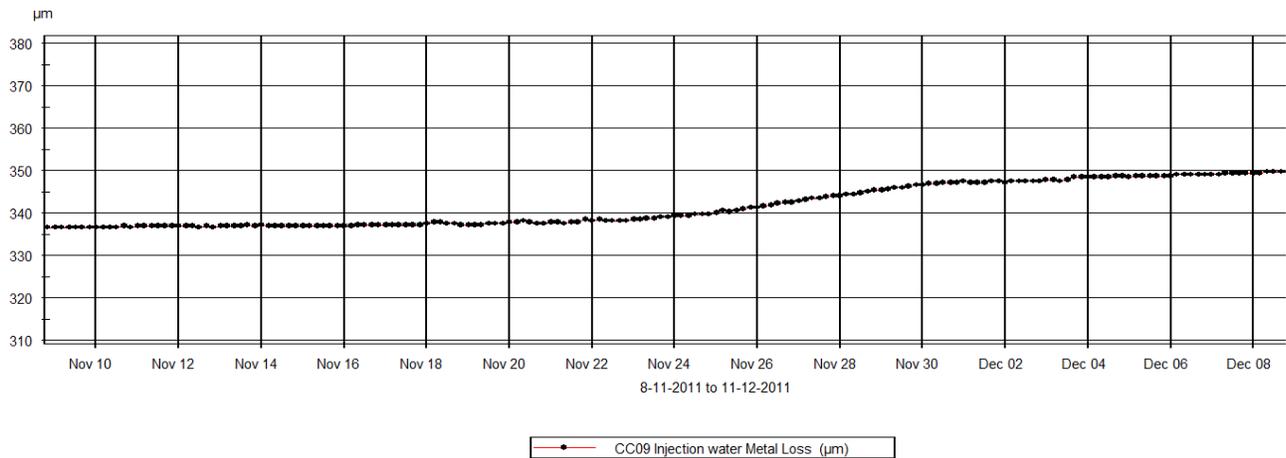
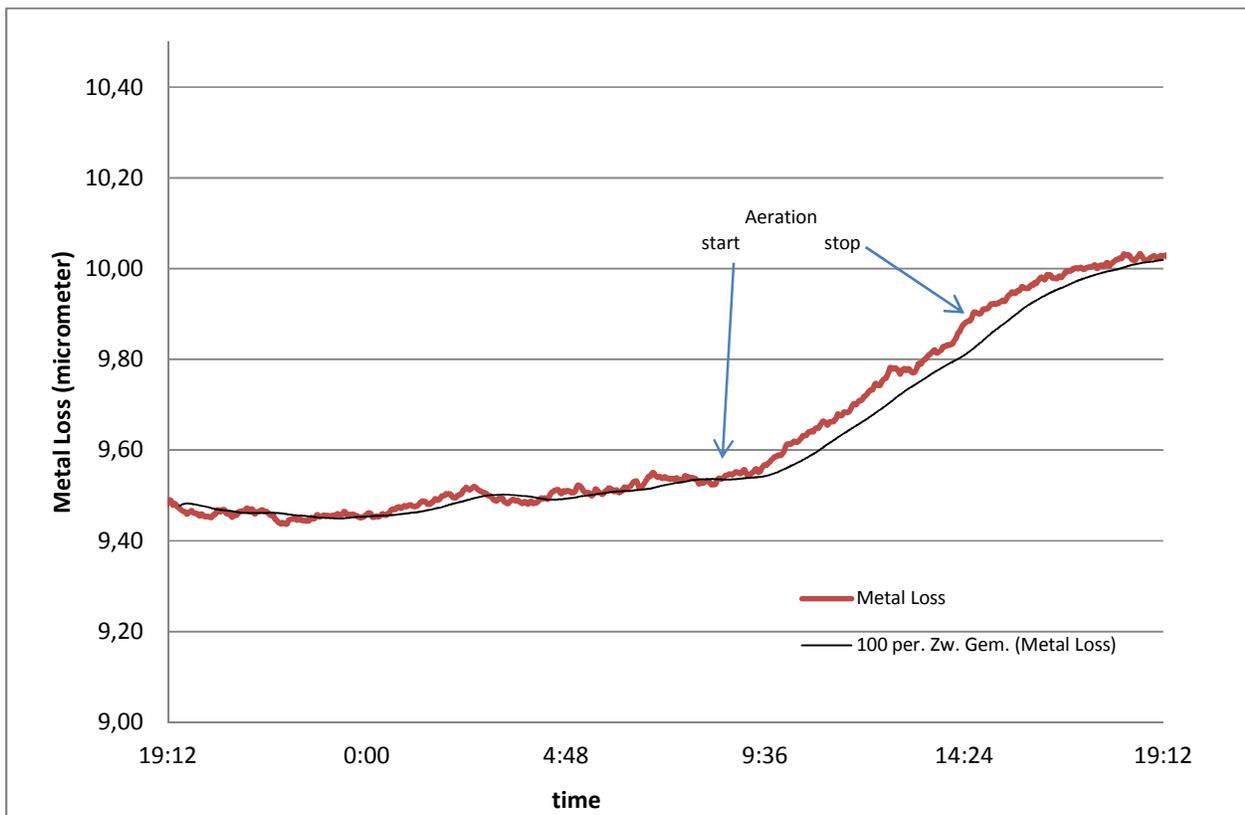*Figure 3: Metal loss of an ER probe (2-inch, flush element, 1 mm thick).*

Figure 4: New high sensitivity ER meter with 4-20 mA interface. Test during one day of carbon steel in artificial seawater at room temperature. From 8:30h to 16:00h the water was aerated, resulting into a steeper metal loss slope. Note the excellent measurement resolution of better than 0,05 micrometer. [1]

ELECTROCHEMISTRY UNIFORM CORROSION

The Corrtran MV[1], further defined as 'corrosion transmitter' is a corrosion monitoring device which uses electrochemical techniques to measure the corrosion rate of the process plant material. The aim of this section is to outline the basic principles of electrochemical corrosion theory as applied to electrochemical corrosion rate measurement and to discuss details of the implementation of these measurement techniques in the Electrochemical Corrosion transmitter.

Corrosion of metals usually occurs as electrochemical dissolution. The corroding metal surface supports at least two distinct electrochemical processes – the anodic and the cathodic reactions. At the anode, metal atoms oxidize to metal ions. The metal ions then either react further with the surrounding electrolyte to form soluble or insoluble corrosion products or simply diffuse away from the metal surface. Electrons are produced by the anodic process. At the cathode, a corresponding reduction reaction takes place, consuming the generated electrons. In acid aqueous environments, the cathodic reaction is usually that of hydrogen evolution. In neutral and alkaline aqueous environments, the cathodic reaction normally is the oxygen reduction.

The rates of the anodic and cathodic process, the anodic and cathodic currents (not current densities) must match, as there can be no net build-up of electrons in the metal other than a minor quantity of electrons used for polarization of the metal. The anodic and cathodic reactions do not occur at the same point on the electrode surface. On a uniformly corroding electrode they will normally be at adjacent points on the metal surface and the anodic and cathodic areas will move in time on the electrode surface. In the case of pitting corrosion, a pit will act as the anode whereas the surrounding surface will be predominantly cathodic.

The relative sizes of the anodic and cathodic areas are important, as the rate of penetration is determined by the anodic current density (current per unit area). In the case of pitting corrosion a large cathodic area of a relatively small cathodic current density may support a high anodic current density (and hence high penetration rate) in the small anodic area of the active pit.

There are two aspects to electrochemistry: thermodynamics and kinetics. Thermodynamical considerations determine whether a reaction can occur at particular pH and overpotential levels, kinetic considerations then determine the rate of that reaction. For example, at a particular pH and overpotential, the oxygen reduction reaction may be thermodynamically possible, but due to a very low concentration of oxygen in the solution, its rate could be very low.

Each of the anodic and cathodic reactions will have a reversible potential – at some potential the rate of the forward process (e.g. metal oxidation and dissolution) will equal that of the reverse process (e.g. metal ion reduction and re-deposition onto the metal surface). Hence, even in the simple situation where the anodic process is metal dissolution and the cathodic process is hydrogen evolution there will in fact be four processes possible. In practice, the anodic and cathodic reactions will be polarised away from their reversible potentials to some common potential – the mixed or 'free corrosion' potential, Ecorr. This causes the metal dissolution direction of the anodic reaction to be preferred and similarly the hydrogen evolution direction of the cathodic reaction will predominate.

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[1] Corrtan MV is a trade name

The anodic or cathodic currents (and hence the corrosion current - Icorr) cannot be measured directly but may be determined indirectly by measuring the potential-current response of the corroding electrode to an externally applied perturbation.

Assuming that both the anodic and cathodic corrosion processes can be described by the Butler-Volmer equation and that the free corrosion potential is sufficiently far from the reversible potentials of the two partial reactions, then the potential-current response of a corroding electrode is given by:

$$I = I_{corr}\left[exp\left(\frac{\Delta E}{b_a}\right) - exp\left(-\frac{\Delta E}{b_{ca}}\right)\right]$$

where ΔE is the applied overpotential from Ecorr and ba and bc are the anodic and cathodic Tafel constants (related to the Tafel slopes βa and βc by b = β/ln10).

The Tafel slopes are related to temperature etc. by:

$$\beta_a = \frac{RT}{\alpha_a Z_a F}$$

$$\beta_c = \frac{RT}{1 - \alpha_c Z_c F'}$$

where R is the universal gas constant, T absolute temperature, F the Faraday constant, z_a and z_c the number of electrons transferred in the anodic and cathodic reactions, and α_a, α_c the reaction symmetry factors. In practical terms, β is about 120 mV at room temperature for a single electron transfer, 60 mV for a two electron process etc.

Expansion of the exponentials as series, neglecting higher power terms and rearranging gives:

$$I_{corr} = \frac{\beta_a \beta_c}{2.3(\beta_a + \beta_c)} x \frac{1}{R_c}$$

Where the polarization resistance

$$R_\rho = \frac{\Delta E}{\Delta L}$$

This is the Stern-Geary equation, which forms the basis for the linear polarization resistance method of corrosion rate measurement. It simply states that the corrosion current may be calculated from the slope of the potential-current curve at the free corrosion potential (polarization resistance, Rp); the proportionality constant (Stern- Geary constant, B) being related to the Tafel slopes.

Once the corrosion current (Icorr) is known, it simple to calculate the thickness of metal dissolved from a given electrode area in a given time period - the penetration rate.

$$penetration\ rate = Icorr\ x\frac{atomic\ weight\ x\ time}{Faraday\ constant\ x\ valency\ x\ density\ x\ area}$$

Penetration (or corrosion rate) is usually presented in the units of mm/year or mils/year (mpy).

In real conditions the pure activation control assumed in these calculations may not apply – the rates of the anodic, cathodic or both processes may be limited by factors such as the rate of movement of reacting species to and from the electrode surface. The above equations may then be modified to take diffusion (also termed concentration polarization) control of one of the reactions into account - the Tafel constant of the diffusion limited process will tend to infinity.

For example, if the cathodic process is diffusion limited then:

$$Icorr = \frac{B_a}{2.3} x \frac{1}{R\rho}$$

It is important to note that all of these forms of the Stern-Geary equation assume that the areas of the anodic and cathodic processes are equal – this will rarely be the case in practice.

The Stern-Geary constant usually lies somewhere in a relatively narrow range of between about 10 and 50 mV, with 20 mV being a typical value.

There are a number of methods available for the determination of the Stern-Geary's constant exact value. A commonly used approach is to estimate its value by comparison to weight loss measurements. This method however cannot track any changes in the chemistry of the corrosion process.

An alternative approach is to measure the electrode response over a relatively wide range of applied potentials and then to use mathematical curve fitting to estimate the Tafel slope values.

Harmonic distortion analysis (HDA) relies on a particular property of the mathematical description of the electrode potential-current response (the difference of two exponential curves), namely that the curvature of the response is such that if a sinusoidal potential perturbation is applied to the electrode then the current response will contain harmonics of the applied sine in proportions related to Icorr and to the Tafel slopes.

To a first approximation:

$$I_{corr} = \frac{I_1^2}{\sqrt{(48(2I_1 I_3 - I_2^2)}}$$

$$\beta_a, \beta_c = \frac{I_2 \pm \sqrt{(48(2I_1 I_3 - I_2^2)}}{I_1}$$

where I_1 is the magnitude of the resulting current at the applied frequency whereas I_2 and I_3 are the magnitudes of the second and third harmonics respectively.

A dynamic measurement, such as when using a sine potential perturbation, requires a consideration of the frequency domain response of the corroding interface. In reality the metal-electrolyte interface is not

purely resistive, but has a frequency dependent reactive component caused by the interfacial double layer capacitance and by mass transport (diffusion) effects.

To a first approximation the metal-electrolyte interface has a response of an equivalent circuit of this form :
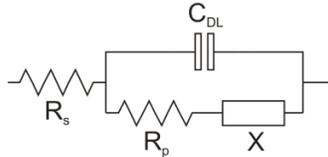
Rs is the ohmic resistance of the electrolyte, Rp is the polarization (charge transfer) resistance, CDL is the interfacial double layer capacitance and X is an element representing the response of other processes, such as diffusion etc.

At high frequencies the impedance of CDL will be low, effectively shorting the parallel components Rp and X, and the measurement will detect mainly Rs. Similarly, at low frequencies the series combination of Rs, Rp and X will predominate [2, 5, 6, 7, 8].

ELECTROCHEMISTRY LOCALIZED CORROSION

In practical situations the corrosion attack is often not uniform. Localized corrosion (such as pitting attack, crevice corrosion, stress corrosion cracking etc.) can occur and can lead to considerably higher local penetration rates than those of general (uniform) corrosion.

Perturbative electrochemical measurements average over the area of the test electrode

– for example a small pit of a high corrosion current density (high corrosion rate) may exhibit a Rp similar to that of a large area of low corrosion current density (low corrosion rate), making it impossible to distinguish the two by an external measurement.

Electrochemical noise techniques measure the time fluctuations of either the corrosion potential (Ecorr) or of the current between two 'identical' electrodes to give a qualitative estimate of the degree of localisation of the corrosion attack process. Electrochemical potential noise measurement relies on the detection and analysis of time fluctuations of the corrosion potential (Ecorr).

The corrosion potential is measured as the potential difference between the test electrode and a second, stable, reference electrode (half-cell). As outlined above, the corrosion potential is an equilibrium value at which the anodic and cathodic currents are equal (though opposite in polarity). Any short term fluctuations of the rates of these two processes will cause a corresponding change in the corrosion potential.

Consider for example a metal surface covered with a passive oxide film where a localized dissolution event (such a break in the passive film due to mechanical stress) occurs. The defect area will momentarily become anodic as the exposed base material reacts with the electrolyte and the passive film reforms. This increase in the anodic current will cause a corresponding momentary increase in the

cathodic current drawn from the remaining passive surface and the overall potential of the electrode will change to a new value at which the anodic and cathodic current are again equal.

The corrosion potential fluctuations are usually of a low magnitude (μV to mV) and of a low frequency (< 1Hz). Different morphologies and types of localized corrosion attack give rise to different time variations of the corrosion potential. For example passive film breakdown is characterized by sharp corrosion potential changes, each followed by a slower exponential recovery, whereas crevice corrosion tends to cause almost periodic fluctuations.

Electrochemical current noise measurement uses the detection and analysis of fluctuations of a current between two nominally 'identical' electrodes connected through a low resistance current measuring device such a 'zero resistance ammeter' (ZRA). If the two electrodes are undergoing uniform corrosion then the measured current will be very low or zero, since the anodic and cathodic areas will be intermixed and evenly distributed over each of the electrodes. Electrons from a particular microscopic anode site will travel only a short distance to the nearest cathodic site, most likely located on the same electrode.

The situation will be different if localized corrosion attack occurs. For example a break in a passive oxide film on one of the electrodes will cause current flow not just between the defect site on that electrode and the surrounding passive cathodic area, but also between the defect site and the still passive cathodic area of the other electrode. This current can then be measured and analyzed as electrochemical current noise.

It is generally difficult to quantify electrochemical potential or current noise measurements as to relate them precisely to the morphology or rate of the localized attack. Empirical, qualitative, approaches are usually used.

For example the standard deviation of electrochemical potential noise can be used to describe the intensity of (or propensity to) localized corrosion attack. In the case of electrochemical current noise a similar descriptor may be calculated from the mean (d.c.) current between the two electrodes and from the standard deviation of its fluctuations.

$$localised\ corrosion\ index\ = \frac{standard\ deviation\ of\ current}{rms\ value\ of\ current}$$

ELECTROCHEMICAL CORROSION TRANSMITTER IMPLEMENTATION

A three electrode test cell configuration with a potentiostatic circuit cell control is used as this offers the best possible control of the potential of the test electrode and of the applied electrochemical perturbation. In a potentiostatic arrangement the test cell is placed in the feedback loop of an amplifier. The feedback arrangement controls the applied potential difference between the test electrode (WE) and the reference electrode (RE) by supplying a polarization current through the third, auxiliary, electrode (AE). The advantage of this arrangement over the sometimes used simple two electrode probe is that it eliminates any contribution of the reference and auxiliary electrode processes to the

measurement result. This is especially important when a dynamic measurement technique such as HDA is used.

The Corrosion Transmitter uses the combination of linear polarization and harmonic distortion analysis (HDA) for corrosion rate measurements. For LPR measurements a constant, the 'B-constant' has to be given by the corrosion engineer. Unique for the corrosion transmitter is that it uses HDA for measuring the B-value, making this 'constant' floating. During corrosion measurements the B-value is optimized. The HDA theory claims it can determine the corrosion current i-corr by measuring the harmonics induced by the "corroding electrodes" when you apply a sinusoidal excitation voltage. It also claims to do this without knowing the Tafel constants (or B-values). This is how the corrosion transmitter uses it: from the HDA i-corr can be calculated, just in dependency of the first three harmonics (i1, i2, i3). Actually the Stern-Geary equation and the excitation voltage is applied. The Stern-Geary equitation also gives Icorr, but dependency of B, i1 (if sinusoidal excitation is used). Now if icorr (from HDA) and icorr (from LPR) is set equal and solve that equation for B you actually get an equation for B in dependency of i1, i2, i3 and the excitation voltage. If this result is filtered over some time (minutes to about an hour) and after applying some averaging, a decent value for B can be set; the 'floating B-constant'.

The corrosion transmitter uses the HDA measurement to compute the Stern-Geary B parameter in each separate measurement cycle. The B factor values are then filtered using a low pass filter algorithm to reduce measurement scatter. The filtered value of B and the most recently obtained value of the polarization resistance (derived from the cell current at the applied frequency only) are then used to calculate Icorr and hence the corrosion rate. This approach combines the best features of the standard linear polarization resistance measurement with the additional advantages of the HDA technique and works extremely well in practice.

Electrochemical current noise (ECN) is applied for localized corrosion detection. The more current and potential noise, the more localized corrosion activity.

The corrosion transmitter uses a small amplitude sine wave (20 mV peak to peak) of a frequency of 0.1 Hz as the perturbation waveform. This is generated as a series of small voltage steps (96 per cycle). 20 cycles of this perturbation are applied to the probe test electrode, the resulting cell current is measured using an accurate analog to digital converter and analyzed by discrete Fourier transform algorithms to give the amplitude of the response both at the applied frequency and at the harmonic frequencies.

The corrosion transmitter uses a high frequency (200 Hz) measurement to determine the electrolyte resistance Rs. The amplitude of this waveform is adjusted during the measurement to between 2 and 50 mV peak to peak, in order to make the best use of the dynamic range available in the circuitry. The measured conductance is available as one of the output variables. As Rs includes the resistance of any film present on the electrode surface, it may be used to monitor scale and film formation.

The corrosion transmitter circuitry is capable of carrying out both potential and current electrochemical noise measurements. Electrochemical current noise is more suited to the detection of localized attack propagation and is used as the default technique to calculate a dimensionless and normalized 'localized corrosion index' variable, ranging from between zero and one.

During the noise measurement the test (WE) and the auxiliary (AE) electrodes are effectively shorted together through a small current sensing resistance. The coupling current between the two electrodes is sampled at regular intervals and its mean and standard deviation values are calculated using a running moment algorithm which avoids the need for intermediate storage of the measurement data. As default, during the noise measurement, corrosion transmitter uses a one second sampling rate and a time record length of 1000 samples.

At the end of the noise measurement period the localized corrosion index is computed as the ratio of the standard deviation of the current fluctuations to the root-mean-square (rms) value of the same data. Tests carried out during the electrochemical corrosion transmitter development have shown that this ratio usually exceeds a value of about 0.3 for electrodes undergoing localized corrosion.

Figure 5 shows the corrosion behavior of stainless steel AISI 316L in water with 0,01M sodium chloride, during a temperature scan up to 60 °C and back to 20 °C. Clearly the pitting temperature is shown by the corrosion transmitter.
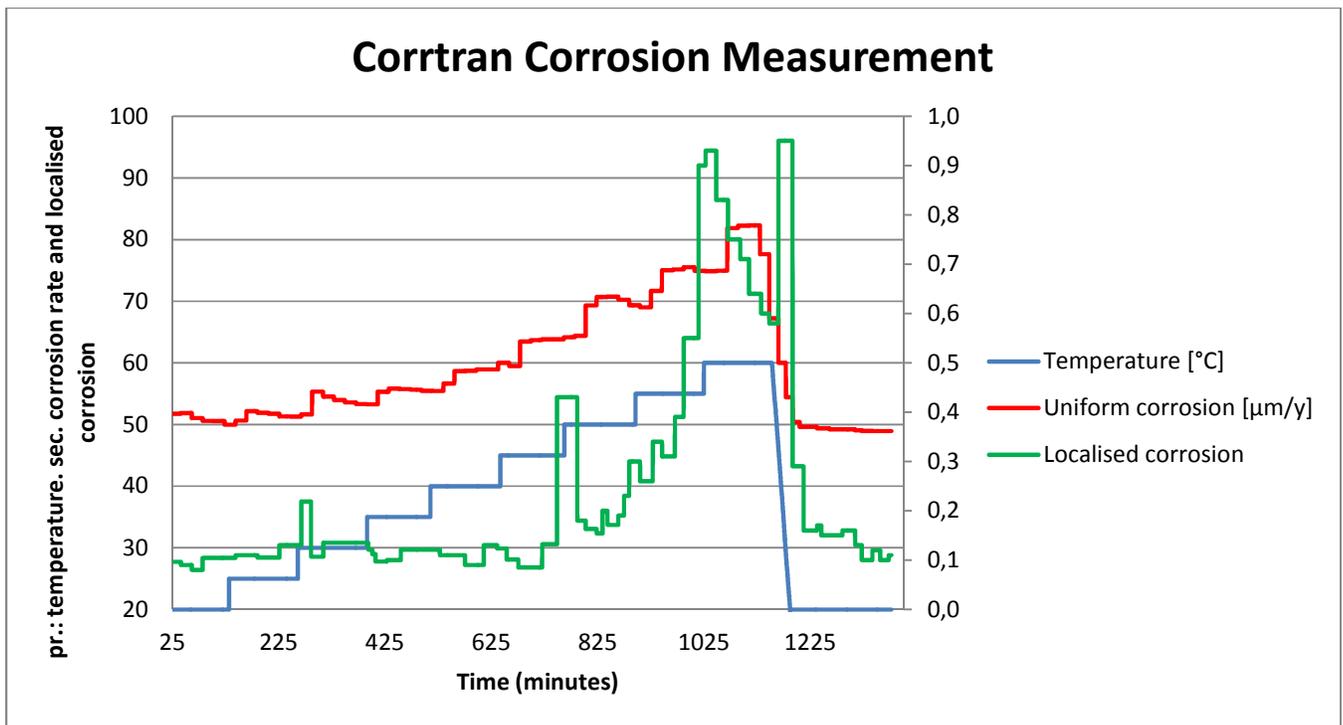

Figure 5: Output of corrosion transmitter during laboratory measurement of stainless steel AISI 316L in water with 0,01M sodium chloride. Red line: uniform corrosion. Green: localized corrosion. At value >0,5 corrosion mainly will be localized distributed (mainly pitting corrosion in this case). [3]

DATA ANALYSIS OF COMBINED PROBE ER/EC

Twenty or thirty years ago engineers had to manually download data, often on remote locations such as offshore platforms or in the desert. Nowadays most sensors are online to the DCS computer, and very easy to get access to, via intranet or internet. The combined probe ER (electrical resistance) / EC (electrochemical) uses modern but very robust industrial interfaces: 4-20 mA as reliable analog interface + HART for digitalizing the signals. Similar to other sensors, such as temperature or pressure sensors, data can be represented in any graph, and alarms can be set if an output exceeds a set-limit. The combined probe gives 4 linear outputs to the DCS via an intrinsic safe 4-20 mA-HART signal:

- Uniform corrosion by linear polarization resistance (LPR) with floating B-constant measured and calculated by harmonic distortion analysis (HDA).
- Uniform corrosion (metal loss) by electrical resistance (ER).
- Localized corrosion (pitting, crevice corrosion and stress corrosion cracking) by electrochemical noise (EN).
- Conductance.

Each of the 4 signals can be set to an alarm after which a corrosion specialist analyzes the data as follows:

- Compare the uniform corrosion data of the ER and electrochemical probe.
- Define the likelihood on and severity of localized corrosion by analyzing the 'localized corrosion output' and both 'uniform corrosion' outputs.
- Check on any conductance variations by analyzing the 'conductance' output.

Of course this online probe can be used for many other applications such as corrosion inhibitor control and water treatment efficacy.

Besides, the probe can be used for process control functions other than corrosion measurements. These functions can include dissolved oxygen monitoring, biofilm activity and chlorination processes. Figure 6 depicts a flush 2-inch combined probe with schematic data interface to safe zone and DCS computer.

Combined probes can be delivered from the second half of 2013. Later integration of the ER meter and Corrosion transmitter into one instrument box has been planned. Currently the two instruments are delivered in one stainless steel cabinet (ATEX), see figure 6.
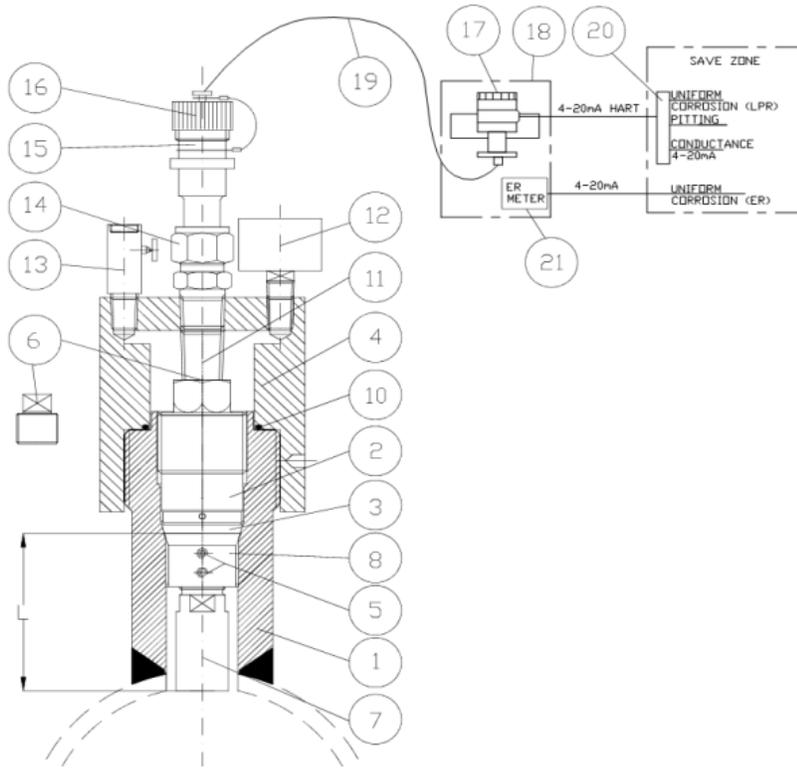
Figure 6: 2-inch flush probe (item 7) in high pressure 2-inch access fitting (item 1). Right: Schematic data interface and wiring scheme. The ER-meter (item 21) and corrosion transmitter (item 17) fit in a stainless steel cabinet (item 18). The entire setup is intrinsic safe for oil and gas zones.

COMBINATION OF PROBE AND COUPON

New is the installation of a combined probe and coupon in one fitting. The advantages of this combination above separate probes and coupons are evident:

1. Only one instead of two access fittings are required.
2. The probe (the electrochemical corrosion transmitter) will tell when to retrieve the coupon. If the corrosion transmitter shows increasing localized corrosion output, the coupon can be retrieved for further investigation on pitting corrosion, cracking, weld root corrosion or crevice corrosion.

Installation of such combination has been planned for a high temperature wellhead for a gas platform. A duplex stainless steel fracture coupon with electrochemical probe has been foreseen. Similar to pitting corrosion, stress corrosion cracking shows strong increase in electrochemical noise. The localized corrosion output signal of the corrosion transmitter will tell when to retrieve the coupon for further investigation on stress corrosion cracking or other localized corrosion. An example of this probe/coupon is depicted in figure 7. Combinations of ER probes, EC probes and coupons can be extended. Both the coupon as the ER-element can function as EC element simultaneously. This can result in a three-element probe with three functions: ER, EC and coupon retrieval depending on ER/EC signal. This

sophisticated corrosion probes only need 6 contact pins, which means similar connector as for standard ER or LPR probes can be applied[2].
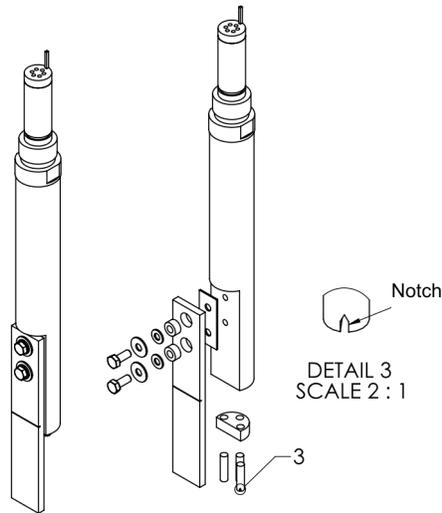

Figure 7: Combined 3-element probe and fracture coupon with a notch for measuring stress corrosion cracking in a high pressure / high temperature duplex stainless steel flowline as planned to be installed on an offshore gas platform in the Northsea. Also the work electrode contains a notch (3). [4]

CONCLUSIONS

Combination of the corrosion monitoring techniques ER, LPR, HDA and EN into one probe will provide significantly more information with regard to corrosion behaviour of pipes, vessels, pipelines etcetera. Both uniform and localized corrosion are measured. Additional to this combined probe, a coupon can be added. The two inch access system will provide significant space for combining ER, Corrtran and a coupon in one probe.

Combination of a coupon and probe in one fitting is very interesting because this will make use of second access fittings redundant.

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[2] On combined probes and coupons technology: patents pending.

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