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Swedish Radiation Safety Authority

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Research

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Corrosion of copper in distilled water without molecular oxygen and the detection of produced hydrogen

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SSM perspective

Background

The KBS-3 repository concept suggested by SKB for final disposal of spent nuclear fuel is based on containment and retardation. These basic safety functions are built upon a multi barrier principle involving three barriers: canister, buffer and host rock. Of these three barriers, the canisters outer copper shell is initially of particular importance since it completely isolates the spent fuel from the surrounding mobile groundwater. However, during the extremely long assessment period the copper canister will eventually degrade by mechanical load and/or copper corrosion. Subsequent failure of the copper shell will result in radionuclide releases. Predictions of canister degradation rates are for this reason very important in the safety assessment for the repository.

In this report, one degradation mechanism for the outer copper shell, corrosion of copper, is considered and especially corrosion of copper in water without dissolved oxygen often referred to as anoxic corrosion. Corrosion of copper in groundwater environments can take place both with and without dissolved oxygen. Since the repository is located about 450 m below the ground surface, corrosion of the outer copper shell will take place in anoxic ground water for the absolute vast majority of the assessment period.

In ground water without dissolved oxygen copper can corrode either due to presence of oxidants naturally present in groundwater like sulfide or by water itself. During the last years, corrosion of copper by pure anoxic water has been debated more and more intensely. Experimentally, formation of hydrogen gas which indicates that copper corrodes in water has been presented and theoretically it has been shown that copper can corrode in pure water if the concentration of copper ions and hydrogen gas pressures both are low. However, quantification of the extent of copper corrosion in anoxic pure water diverges between a traditional thermodynamics approach and hydrogen gas pressures measured experimentally, and there is a lack of understating of why this difference exists.

Objectives

The aim of this project is to study the influence of temperature on the measured steady state partial pressure of hydrogen during exposure of copper in water without dissolved oxygen and to characterise the corrosion product formed.

Results

In this report, results are presented for copper which has been exposed to pure anoxic water in the temperature interval of 21°C to 55°C up to a total of 19 000 hours. Characterisations of copper surfaces after exposure have been performed ex-situ, meaning after termination of the experiment and exposing the specimen to normal atmospheric environment. Ideally characterisation of surfaces should have been performed with the specimens in the reaction chamber without oxygen supply but this was not possible in the experimental set-up used. Thus it cannot be excluded that formed species on surfaces could have been altered during handling of specimens between exposure and surface analysis.

The results from the surface analysis of exposed copper specimens indicate that the reaction products are predominately comprised of oxide and hydroxide. Furthermore, based on the visual appearance, the reaction products formed are solid and of a three dimensional character. Moreover, depth analysis by ion sputtering shows that hydrogen is present at greater depth from the surface and inwards compared to oxygen. This indicates that corrosion of copper in anoxic water involves a mechanism in which hydrogen atoms present in water molecules form hydrogen gas which partly dissociate and diffuse into the copper metal as hydrogen atoms.

During exposure of copper to anoxic water, the measured partial pressure of hydrogen gas increases until a steady state pressure eventually occurs. After the steady state pressure has been obtained the hydrogen pressure becomes practically constant in the experimental set-up used. The steady state partial pressure of hydrogen has been found to show similar temperature dependence as the temperature dependence for the water auto-ionisation constant, at least between 22°C to 56°C. This finding provides an interesting clue in the understanding of corrosion of copper in anoxic water.

As mentioned in the background, it must be emphasised that safety assessment of the KBS-3 repository concept is not only dependent on how fast copper will degrade by either corrosion in pure anoxic water or normal sulphide corrosion. The combined effect of all the three barriers needs to be considered for evaluation of the containment safety function. Moreover, the retardation safety function also needs to be considered during assessment of SKB's safety assessment for a spent fuel repository.

Need for further research

The results in this report show that the temperature dependence of steady state hydrogen pressures is similar as for the auto-ionisation constant of water and that a solid reaction product appears to be formed on the copper surface during exposure. These findings clearly demonstrate that more work is needed to understand the mechanism of copper corrosion in the repository environment. It is proposed that emphasis in future research should address the diverging results from traditional thermodynamics and experimentally measured hydrogen pressures when copper is exposed to pure anoxic water.

Project information

Contact person SSM: Jan Linder
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Contents

Summary

This paper reports on hydrogen pressures measured during the long-term immersion (~19 000 hours) of copper in oxygen-free distilled water. Hydrogen gas evolution is from copper corrosion and similar pressures (in the mbar range) are measured for copper contained in either a 316 stainless steel or titanium system. Copper corrosion products have been examined ex-situ by SEM and characterized by X-ray photoelectron spectroscopy (XPS) and secondary ion mass spectrometry (SIMS). XPS strongly indicates a corrosion product containing both hydroxide and oxide. SIMS shows that oxygen is mainly present in the outer 0.3 μm surface region and that hydrogen penetrates to depths in the substrate well below the corrosion product.

Introduction

Release of hydrogen upon immersion of a metal in liquid water is evidence for reaction between the metal and the water itself [1]. A summary of ultrahigh vacuum experiments and considerations of stabilities of various products in metal-water reactions has been presented in [2]. However, a printing error in [2] concerning the stability of a hydrogen-free reaction product means that a hydrogen- containing reaction product (CuOH) is favoured over a hydrogen-free reaction product in the Cu-water reaction. This fact is not generally known. The first report of hydrogen gas evolution from Cu-corrosion at room temperature in liquid distilled water in the absence of any applied potential and in the absence of O_2 was made 27 years ago [3]. This has been followed by relatively recent work [4-6]. The measured hydrogen gas pressure will ultimately be in the mbar (10^2 Nm^{-2}) range. This pressure is of the order of a thousand times higher than the hydrogen gas pressure in ambient air [7]. The present paper reports on hydrogen pressures measured in long-term exposure experiments, up to ~ 19 000 hours.

It has been argued, however, that this detection of H_2 must be the result of something other than reaction of Cu with water. These arguments are mainly based on suggestions of other reactions and that the second law of thermodynamics is violated [8,9]. Due to the importance of the application of final storage of nuclear waste in Cu-canisters, a serious attempt was recently made to repeat the experiments in [4,5]. Results confirming the detection of hydrogen gas are reported in [10]. These previous experiments were performed with copper contained in glass in a 316 stainless steel system. Here we also report hydrogen gas measurements where the stainless steel vessel is replaced by titanium.

We also present *ex-situ* characterization of the previously formed reaction product with released hydrogen gas found in papers (4,5). These products are mainly formed during approximately two years exposure of Cu in distilled water in the 21-55°C temperature range. The *ex-situ* characterization is performed by field emission gun scanning electron microscopy (FEG-SEM), X-ray photoelectron spectroscopy (XPS) and secondary ion mass spectrometry (SIMS). We also point out some main features in the measurements of low hydrogen gas pressures as well as the detection of hydrogen in the copper substrate by SIMS.

Experimental

Figure 1a shows a photo and Figure 1b a schematic of the equipment for measurement of low pressures of hydrogen gas.

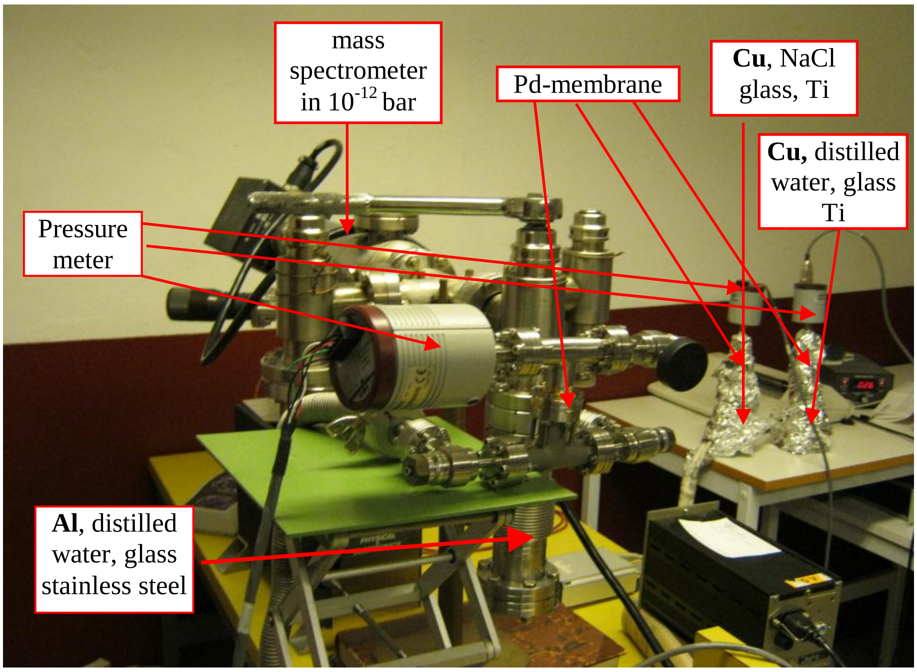
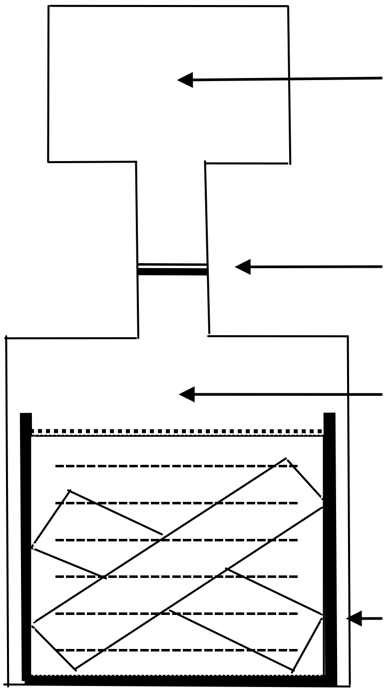


Figure 1a. *Equipment for pressure measurements of hydrogen gas from metals immersed in water. Mass spectrometer is used for verification that measured pressure is from hydrogen gas.*

Hydrogen gas pressures are measured with an absolute pressure meter and a mass spectrometer is employed for verification of hydrogen gas where an example of a mass spectrum is shown in [5]. All experiments are with a palladium membrane of a thickness 0.1-0.2 mm through which only hydrogen will be transported. An initial evacuation of the compartment placed on one side of the membrane will enable the detection of hydrogen gas with the absolute pressure meter. This evacuation must be thorough and made with an ultrahigh vacuum pump for at least 24 hours at room temperature. The other side of the membrane is in contact with the volume where hydrogen gas is generated. In effect, the measured hydrogen pressure is in a 180 cm^3 volume.

Depending on the detection limit of the pressure meter H_2 can be measured down to ~ $2x10^{-7}$ bar. 99.95 wt% copper sheet specimens, 0.1 mm thick, in the rolled condition supplied by Goodfellow were used. The grain size was ~ 20μm.



Initially evacuated 60 cm^3 volume with absolute pressure meter and, via a leak valve, connected to a mass spectrometer in Ultra high vacuum for verification of hydrogen gas.

Palladium foil, ∅ 16 mm, nominal thickness,100 μm

Gas volume with an absolute pressure meter and valve for removal of air. Volume of 120 cm^3 in the 18 800 h exposure of Cu-sheets.

Soda glass container with 140 cm^2 exposed surface of Cu in 90 ml of distilled water

Figure 1b. *Schematic experimental set up for the present immersion tests of the Cu-sheets and detection of the associated hydrogen gas formation.*

Analysis of the previously exposed Cu-sheets was performed with SEM, XPS and SIMS. In the FEG-SEM micrographs an accelerating voltage of 20 kV was used. XPS-analysis was performed in a PHI 5500 spectrometer with a monochromated AlK_a source, SIMS in an PHI Adept 1010 with a primary 4 keV Cs ion beam, and secondary ions from approximately 0.1x0.1 mm were detected in a Cameca spectrometer with a primary 10 keV Cs source with detected secondary ions from 30 μm diameter and raster area of 200 μm x 200 μm.

Results and discussion

Detection and measurement of hydrogen

Figure 2a shows hydrogen gas pressures during the last 5 000 hours of 140 cm^2 exposed copper surface area in 90 cm^3 distilled water in a glass container within a stainless steel vessel, where the hydrogen gas detected at shorter exposure times is found in [4,5].

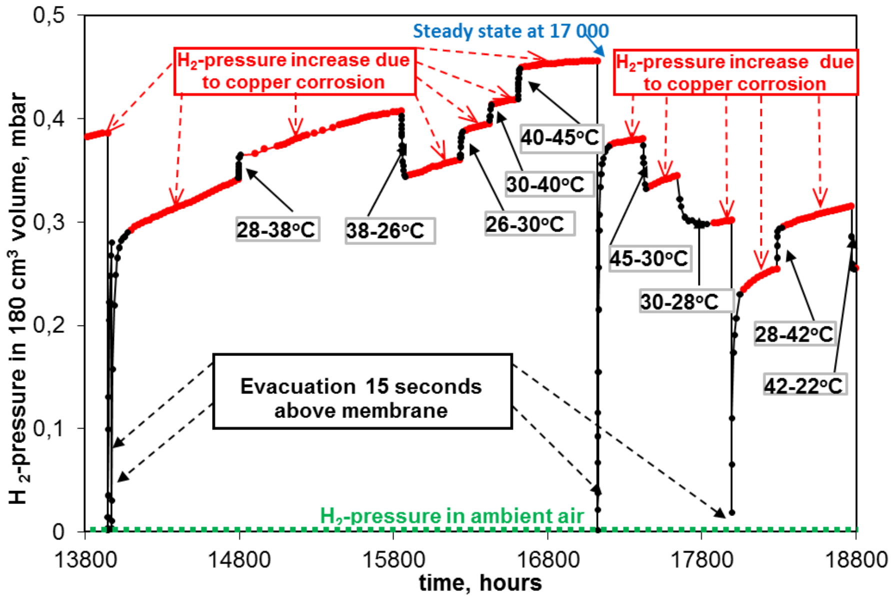


Figure 2a. *Measured H_2-pressure in exposure of 140 cm^2, 0.1 mm Cu to 90 ml distilled water at 21-26^{o}C at pH near 7. Evacuation of hydrogen gas above Pd-membrane and temperature influence of the Pd-membrane in the 22-45^{o}C range (shown within squared boxes) on measured H_2-pressure. Almost no change of H_2-pressure at 17 000 hours and therefore no overall corrosion of copper.*

The hydrogen pressure increase due to copper corrosion is clearly observed. It is also seen that temperature increases of palladium produce rapid increases in measured hydrogen content due to decreases in the amount of hydrogen in the palladium and vice-versa. At 17 000 hours there is almost no change of pressure and therefore no copper corrosion. Only a steady state (equilibrium) between formation (oxidation) and removal (reduction) of the corrosion product is present at 17 000 hours on an atomic level. This steady state does not depend on the hydrogen content in the palladium membrane. Figure 2a also indicates the hydrogen gas level in ambient air at

$\sim 5 \times 10^{-7}$ bar near the x-axis which is clearly much lower than the measured hydrogen gas pressures from copper corrosion.

It is possible to estimate the corrosion rate in number of moles per hour from the measured absolute pressure increase due to copper corrosion in the relevant volume and temperature. The relevant volume is the volume above the membrane plus the volume below the membrane since, in effect, there will be a negligible pressure difference over the palladium membrane. This is due to the short time of response relative to the real time of pressure change.

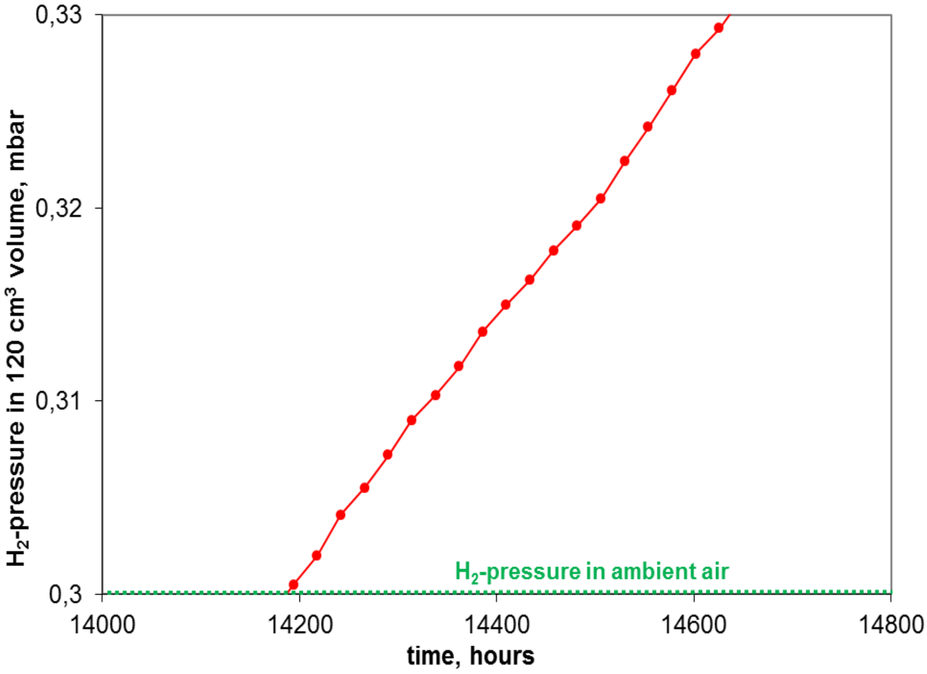


Figure 2b. *Detailed data from Figure 2a.*

Since 1 dm^3 contains approximately 0.04 mole H_2 at room-temperature and pressure of 1 bar, we have from Figure 2b that the measured increase of H_2-pressure of 0.03 mbar in approximately 400 hours corresponds to a production rate of hydrogen gas of $\sim 4 \times 10^{-10}$ moles per hour. This production rate of hydrogen gas is present both at 14 500 and 18 500 hours as seen in Figure 2a.

An estimate of metal corrosion rate based on $Me+H_2O \rightarrow Me_xO_y + H_2$ (measured) with an assumed density of 4 gcm^{-3} gives 2/126 x 4 = 0.063 g of Cu-corrosion for formation of a corrosion layer of 1cm. Hydrogen contained in the corrosion product, in the copper metal and in the palladium membrane must be estimated and this amount of hydrogen depends on the dimensions of the corroding sample and the Pd-membrane. The true corrosion rate may be at least a factor of three higher than that calculated from hydrogen gas release rates. With this factor taken into account the corrosion rate for the copper sheets is estimated to 0.01-0.1µm per year (8640 hours) at room temperature.

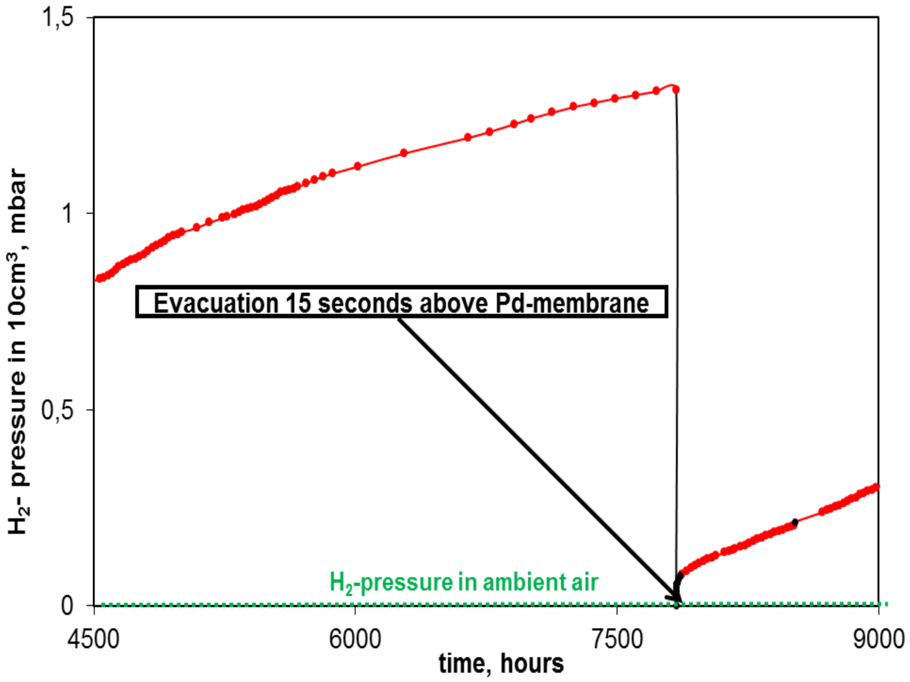


Figure 3. *Measurement of* H_2 *during exposure of 20 cm*^{2}*, 99.95% Cu to distilled water at approximately 50*^{o}*C. Glass tube placed in a drilled titanium bar with exposed area of 75 cm*^{2}*.*

Figure 3 shows hydrogen gas measurements of 20 cm^2 copper sheets in 15 ml of distilled water in a glass container within titanium. Several tests, including lower temperature of the palladium membrane compared to the temperature of Cu, took place up to 4 500 hours. In this way the transport of hydrogen gas in a water film on the palladium surface could limit the kinetics of measured hydrogen but these results are outside of the scope of this report. A more or less time independent release rate of hydrogen gas pressure in the mbar range (10^2 Nm^{-2}) is also found in Figure 3 and this takes place at ~ 8 000 hours.

A steady-state of hydrogen gas generation in Cu-corrosion is clearly not dependent on either stainless steel or titanium as the background material. This is illustrated in Figure 4 where the same steady state is obtained with these two background materials. The steady-state can be interpreted as a balance between the formation of a reaction product and removal (reduction) of the same reaction product. We believe that this reaction product is CuOH and is the first formed reaction product in distilled water. However, this might transform to Cu_2O with some intrinsic hydrogen both in the oxide and in the Cu-metal.

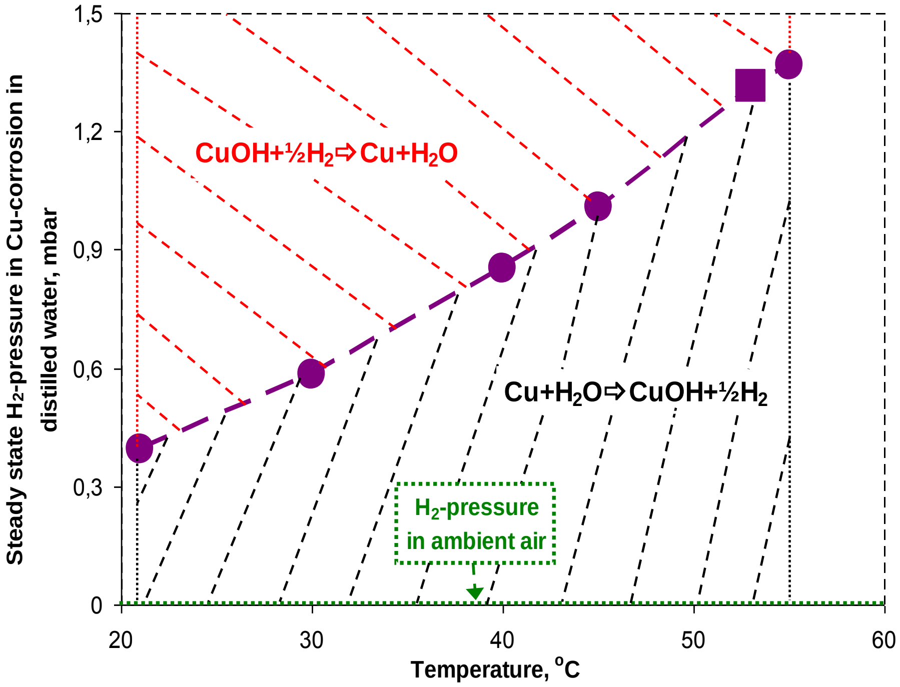


Figure 4. *Steady state H_2-pressure vs. temperature in Cu-corrosion in distilled water.*

Increased temperature causes increased presence of OH^- due to auto-ionisation of liquid water and therefore increased steady state H_2-pressure. Filled circles from [5] where background was with Stainless Steel 316L and filled square from present work where background was with Ti. Size of marks correspond approximately to error bar in the measurements.

In air-contact this transformation can also lead to formation of CuO. Under certain conditions no transformation of CuOH may take place. The steady state H_2 pressure in distilled water increases with temperature: H_2 = 0.395 mbar at 21°C, 0.584 mbar at 30°C, 0.853 mbar at 40°C, 1.009 mbar at 45°C and 1,368 mbar at 55°C [5]. Therefore temperature has a predictable influence, via the presence of OH^- and H^+, on the steady state H_2-pressure. In Cu corrosion in distilled water we consider:

At a steady state, formation of a reaction product equals removal (reduction) of the same reaction product.

In Figure 4 at pressures lower than a steady state pressure the reaction product forms:

$Cu + H_2O \rightarrow CuOH + 1/2H_2$ (1)

where a part of the released hydrogen in (1) can be monitored as H_2 in the gas volume above the immersed Cu and another part enters the Cu-metal.

At pressure higher than the steady state pressure the reaction product is reduced back to metallic copper and water:

$CuOH + 1/2H_2 \rightarrow Cu + H_2O$ (2)

Upon a temperature decrease at the steady state, e.g. from 55 to 43°C, a resulting reduction of the reaction product takes place since the concentration of OH^- is lower at the lower temperature. This reduction is also seen in Figure 4a in reference [5].

Several parameters can influence the rate of H_2 release to the gas-phase where the measurement takes place. Some of these are time of exposure, temperature and H_2-pressure. In addition to these parameters the concentration of hydrogen in the Cu-metal will also influence the measured H_2-pressure. Especially the influence of hydrogen in the Cu-metal on corrosion rate is hard to predict. In Table 1 the rates of measured H_2-release at certain times, temperature and H_2-pressure in the 18 800 hours immersion of Cu are collected and taken from [4,5] and Figure 2 in this report. The H_2-release to the gas-phase, reasonably far from a steady state, is expressed as measured increased H_2-pressure per hour in the 120 cm^3 volume.

Table1. *H_2-release rate to the gas-phase at various time and temperature. Measurement of rate at a H_2-pressure which is reasonably far from pressure at a steady state.*

H_2-release rate [mbar h^{-1}]	Time of exposure [h]	Temperature [°C]	H_2-pressure [mbar]
$3x10^{-4}$	2 900	45	0,5
$4x10^{-4}$	5 500	52	0,7
$0.6x10^{-4}$	14 300	23	0,3
$0.5x10^{-4}$	18 300	23	0,35

From Table 1 we judge the H_2-release rate to the gas-phase increases by approximately a factor 8 when the temperature is increased from 23 to 52°C. However, as already mentioned, this rate can change for many reasons and is therefore hard to predict. On the other hand it is relatively straight forward to calculate the amount of released H_2 to the gas phase from [4,5]. We find that the amount of hydrogen released to the gas phase based on measured hydrogen gas pressure, volume and temperature is ~3×10^{-5} mole in the 18 800 hours exposure. Assuming formation of entirely CuOH with a density 4g cm^{-3}, this corresponds approximately to a 0.1μm thick reaction product.

Characterization of corrosion product

Secondary electron microscopy, SEM

SEM micrographs are shown in Figure 5. The upper left (a) micrograph is a copper sample which has been stored in ambient air for more than 3 years, whereas b, c, and d are

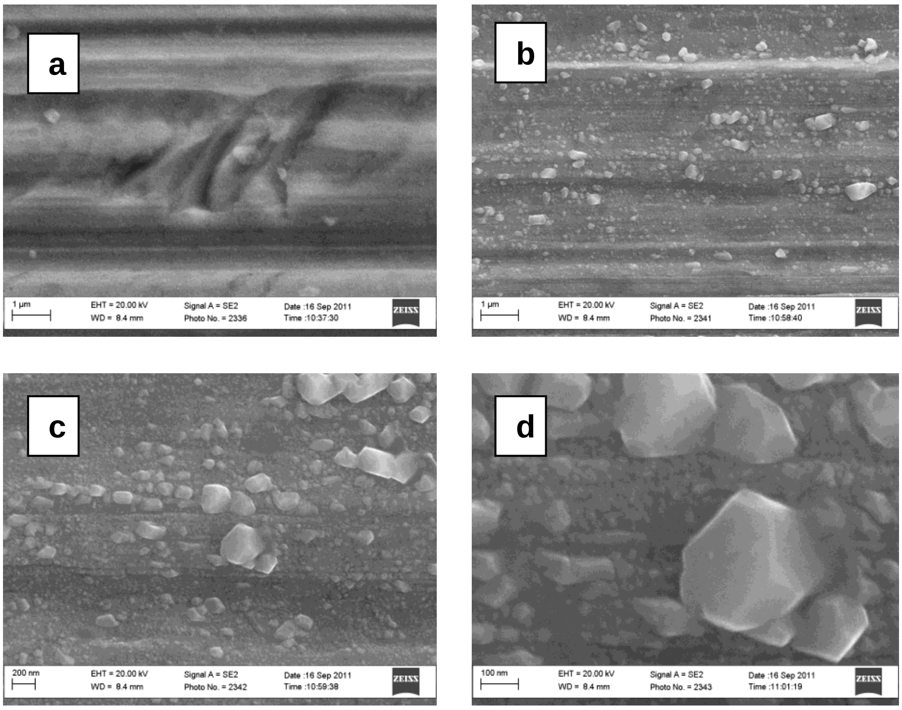


Figure 5. *SEM on Cu-samples. a is air exposed for more than 3 years at room temperature and b, c and d, after 2.2 years in distilled water without* O_2 *at 21-55°C plus 0.5 year in air at room-temperature.*

different magnifications from a Cu-sample exposed to O_2-free distilled water for 2.2 years followed by a 6 month exposure to ambient air. Corrosion product is evident, but may have changed in composition and structure due to the air exposure. The SEM micrograph in Figure 6 is of a sample exposed to distilled water for approximately one year and exposed to air for only 5 minutes before insertion into the SEM. Also here a significant corrosion product is observed. However, we can not completely rule out that the time in the SEM-apparatus, approximately 5 minutes under high vacuum, can alter the reaction product.

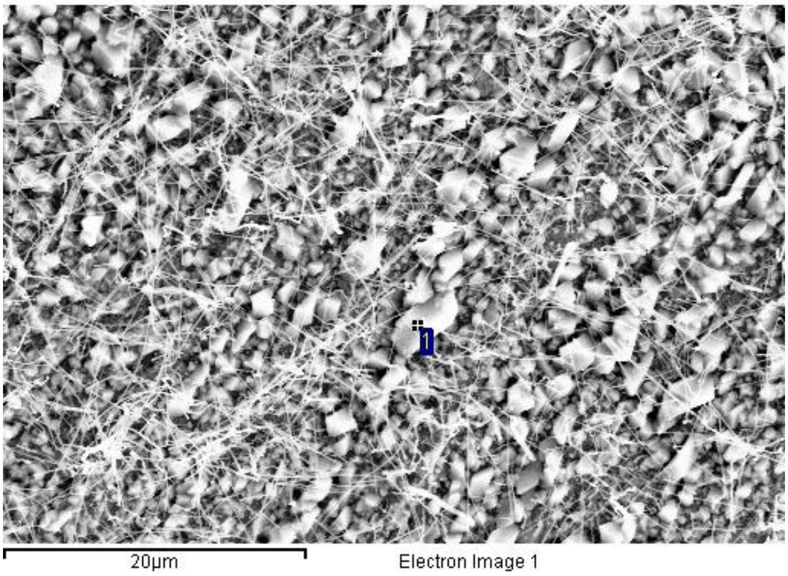


Figure 6. *Reaction product on Cu-foil after exposure to distilled water in absence of O_2 approximately 50^oC for one year and 5 minutes in air at room-temperature.*

X-ray photoelectron spectroscopy, XPS

In this technique the signal originates from < 5 nm of the outer surface of a sample. It is from the surface out-coming electrons which determine this information depth and not the incoming X-rays which penetrate the surface in the order of 1000 nm.

The copper used in the 18 800 hours exposure has been analysed with XPS. Figure 7 shows the oxygen (O1s) spectrum compensated for charging based on the carbon 1s peak.

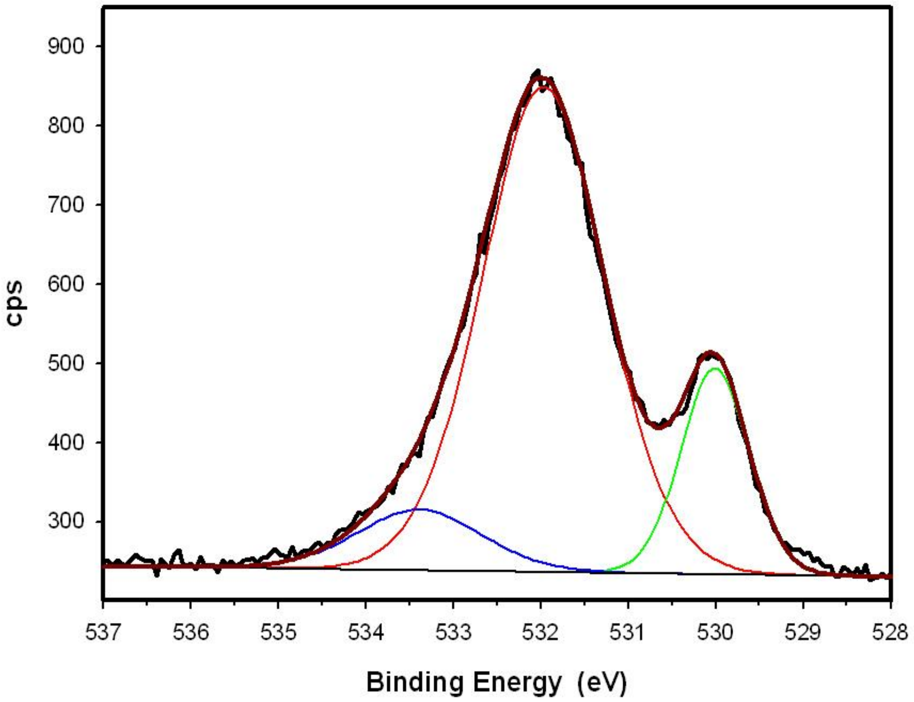


Figure 7*. O1s XPS-spectrum from Cu exposed to distilled water for 18 800 hours.*

The spectrum in Figure 7 can be fitted to two peaks, 532.7 and 530.6 eV which correspond, respectively, to hydroxide and oxide [11]. A better fit is obtained for three peaks (532.0, 530.1 and 533.5). The last small peak at the highest energy could be due to adsorbed water as identified by Grosvenor et.al. for iron exposed to water vapour [12]. In any event, the XPS data (including analysis of the Cu 2p spectrum) indicate predominantly copper hydroxide and copper oxide.

Secondary ion mass spectroscopy, SIMS

In dynamic SIMS the incoming ions sputter the surface and in this way a depth-profile of detected ions is obtained**.** A quantitative depth scale can be obtained with a relevant calibration and an advantage is to normalise the detected ions with the emitted metal isotopes. (CuO/Cu, O/Cu and H/Cu in this case). SIMS-ions of O (Figure 9) and H (Figure 10) as well as CuO (Figure 8) and CuH are a measure of oxygen and hydrogen by this technique. The further detailed interpretation of the difference of these measurements is still elusive.

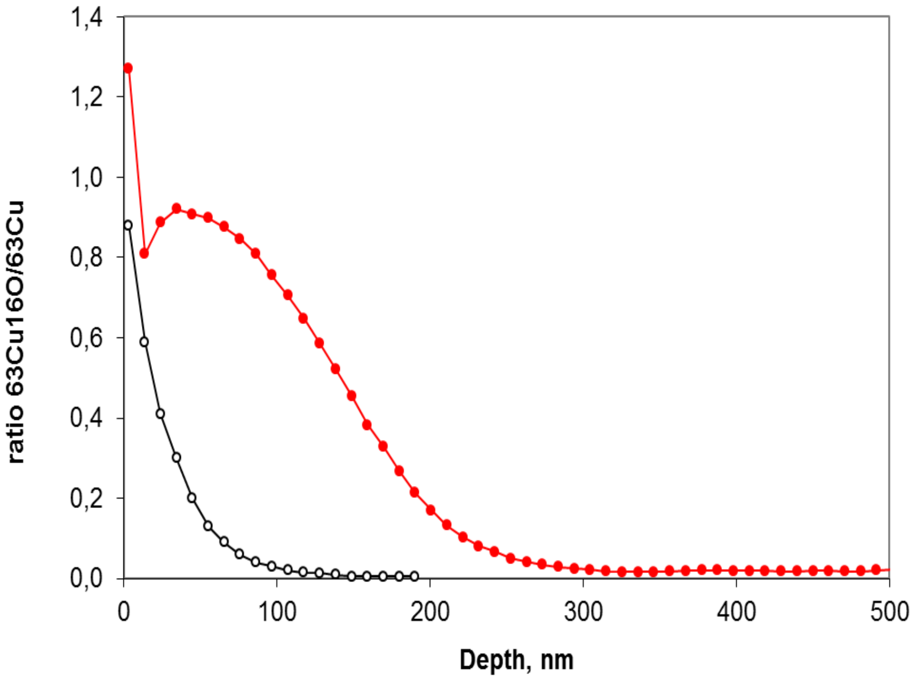


Figure 8. *Dynamic SIMS analysis of oxygen in outer part of Cu-samples. Black 3 years in air and red 1 year in air plus 2 years in liquid water in the absence of* O_2*.*

SIMS analysis with ion beam sputtering was performed to examine the corrosion products in-depth and to detect hydrogen. Figure 8 illustrates dynamic SIMS analysis of oxygen in the outer part of a Cu-sample exposed to air for three years and another to air for one year and liquid water in the absence of O_2 for two years. Clearly this SIMS analysis at NRC, Ottawa, Canada shows that the oxide on the water-exposed sample is much thicker than that produced in air. This was confirmed by SIMS analysis performed at NUS in Singapore of a similarly exposed sample (Fig. 9).

As seen in Fig. 10, for the water-exposed sample, hydrogen is found below the oxygen which is mainly on the surface. This seems reasonable since diffusion of hydrogen is higher than for oxygen in most, if not all, materials. An advantage for detection of hydrogen in SIMS is the possibility of the use of D_2O instead of H_2O due to a lower background of D in the SIMS-spectrometer. However, Cu may not react to the same extent in D_2O [13].

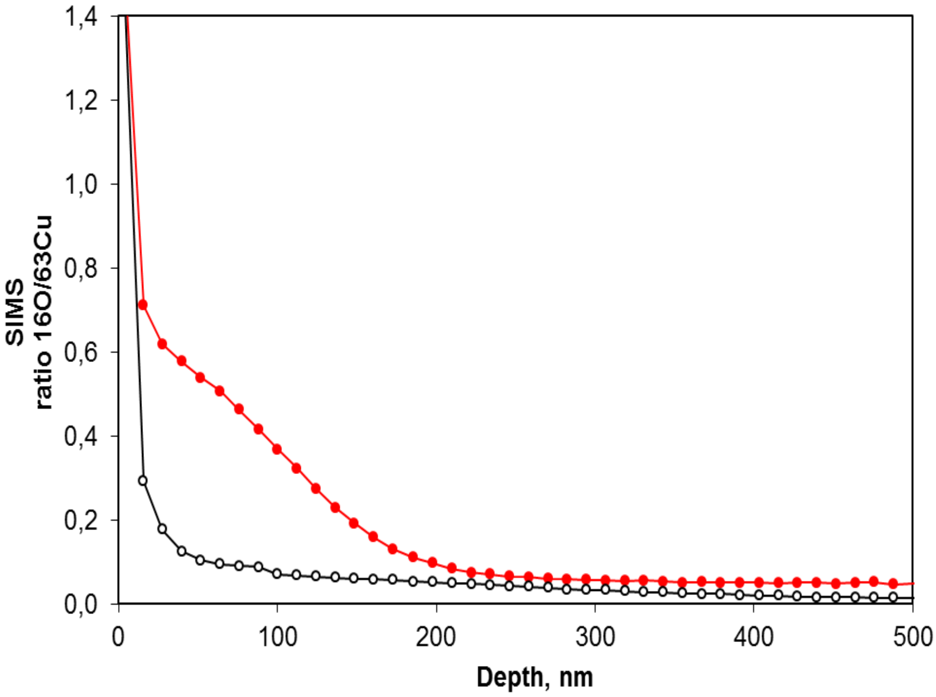


Figure 9. *Dynamic SIMS analysis of oxygen in outer part of Cu-samples. Black 3 years in air and red 1 year in air plus 2 years in liquid water in the absence of* O_2.

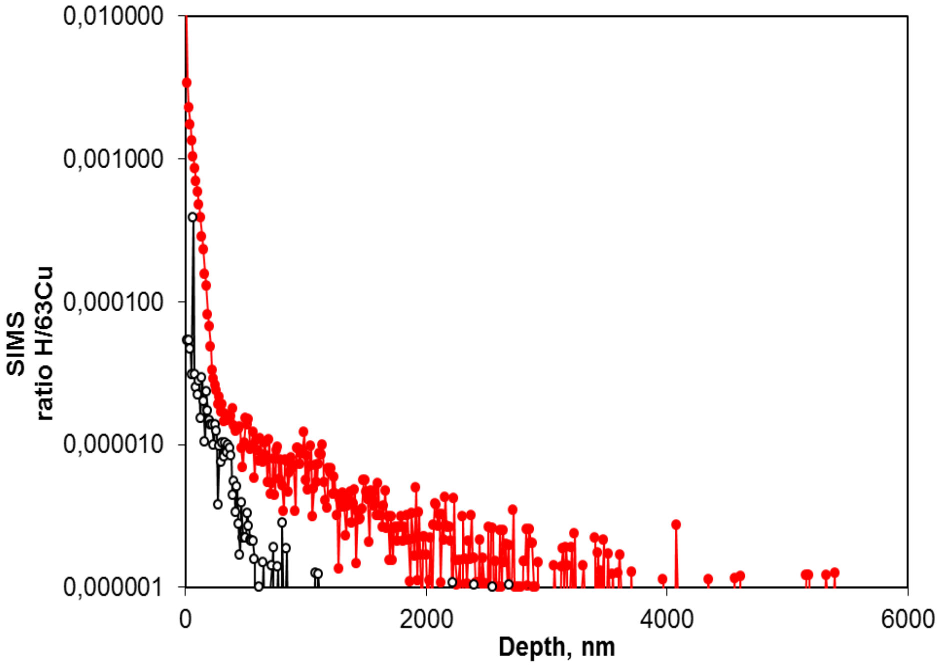


Figure 10. *Hydrogen detection in SIMS. Black 3 years in air and red 1 year in air plus 2 years in liquid water in the absence of* O_2.

Conclusions

Based on the results in this study the following can be concluded:

I Cu reacts with water producing hydrogen as a by-product.

II Hydrogen gas in the mbar pressure range can be detected from the reaction between copper and water without dissolved O_2.

III In the temperature range 22-56°C there is a maximum hydrogen gas pressure which is interpreted as a balance between reaction product growth and removal (reduction) of the same reaction product.

IV Hydrogen can be detected in the Cu-metal from the reaction between copper and water without dissolved O_2.

V The conclusion in **I** requires knowledge that the pressure of H_2 in ambient air is lower than that measured from Cu-corrosion. (Actually measured up to a thousand times lower)

VI Use of an UHV system in the immersion will ensure the absence of O_2 in liquid water, which is a prerequisite for the experimental observations in **I-IV.**

VII A solid reaction product is formed during reaction of Cu with water without dissolved O_2. This product is characterized by SEM, XPS and SIMS.

VIII Oxide grains, interpreted as copper corrosion products, up to approximately 0.5 μm are observed in SEM-micrographs.

IX An analysis of binding energies in XPS strongly indicates a reaction product containing both hydroxide and oxide.

X Depth profiles in SIMS show that oxygen is mainly present in the outer 0.3 μm.

XI Depth profiles in SIMS show that hydrogen is present at depths in the copper substrate well below that of oxygen.

Acknowledgements

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The Swedish Radiation Safety Authority has a comprehensive responsibility to ensure that society is safe from the effects of radiation. The Authority works to achieve radiation safety in a number of areas: nuclear power, medical care as well as commercial products and services. The Authority also works to achieve protection from natural radiation and to increase the level of radiation safety internationally.

The Swedish Radiation Safety Authority works proactively and preventively to protect people and the environment from the harmful effects of radiation, now and in the future. The Authority issues regulations and supervises compliance, while also supporting research, providing training and information, and issuing advice. Often, activities involving radiation require licences issued by the Authority. The Swedish Radiation Safety Authority maintains emergency preparedness around the clock with the aim of limiting the aftermath of radiation accidents and the unintentional spreading of radioactive substances. The Authority participates in international co-operation in order to promote radiation safety and finances projects aiming to raise the level of radiation safety in certain Eastern European countries.

The Authority reports to the Ministry of the Environment and has around 270 employees with competencies in the fields of engineering, natural and behavioural sciences, law, economics and communications. We have received quality, environmental and working environment certification.

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