

The Effect of Copper (II) Nitrate Addition and Tensile Stress on SCC of C44500 Material Tube

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Abstract

The heat exchanger is an intermediary for two or more fluids that flow both inside and outside of the heat exchanger to distribute heat. This process was aimed to increase efficiency levels in the process industry. One of the suitable materials for the tube is UNS C44500, also known as admiralty brass consists of 70Cu-30Zn, which has high thermal conductivity and good corrosion resistance. However, admiralty brass was susceptible to stress corrosion cracking (SCC) when faced with nitrate since the tube was also loaded by internal stress. Moreover, SCC was also exacerbated by dezincification. The C-ring test method examined the effect of nitrate compounds and dezincification on SCC in C44500 Tube components. Tensile stresses varied between 15, 45, and 85% of yield strength, which were immersed into nitrate solutions consisting of NaNO_3 and $\text{Cu}(\text{NO}_3)_2$. Some specimens were washed using 20% HCl to bring up dezincification. The results obtained that SCC occurred at the highest $\text{Cu}(\text{NO}_3)_2$ concentration, identified by longitudinal crack and passivity breakdown. All of the dezincified specimens were found cracked due to SCC after immersion into 1 M $\text{Cu}(\text{NO}_3)_2$, while un-dezincified were failed when given tensile stress between 45% to 85% of maximum yield strength

Keywords: Copper (II) Nitrate, Tensile Stress, SCC, UNS C44500, Dezincification

1. INTRODUCTION

The heat exchanger is known widely as one of the most critical components in the industrial process, accommodating heat transfer processes of two fluids with different temperatures. The heat flows from a high-temperature region to the lower one. This equipment works based on the principle of conservation of energy, which states that heat energy flows in two types of fluids which neither could be created nor destroyed [1]. Instead, heat energy could be converted from one to another form vice versa. Conversion of energy becomes a critical aspect as industrial sectors are concerned more to increase efficiency level [2].

Most industries use shell & tube heat exchanger type, consisting of a single-tube with large diameter called as shell inserted by groups of smaller tubes which considered as tubes component. The smaller tube has an essential role during heat transfer processes, which acts as a separator and heat conductor between two fluids [3]. Material selection is strictly required to accommodate heat transfer processes. One suitable material is UNS C44500, also known as Admiralty Brass UNS C44500, consisting of Cu-Zn alloy metal with 70Cu – 30Zn. This material has several advantages when applied as heat exchanger tube material, such as good corrosion resistance, high thermal conductivity, and excellent machinability, which easily formed into mechanical components [4].

Although admiralty brass generally has good resistance to atmospheric corrosion, several recent studies had shown that admiralty brass was found susceptible to SCC, significantly when contents of Zn were depleted, mainly caused by dezincification which occurred when the material was exposed to particular fluid, such as water contained CO_2 or chloride compounds [5]. Those fluids would form another structure of brass rich in Copper (Cu), while compounds from fluids dissolved the Zn content. Severe dezincification will affect the brass to become porous and brittle. As a result, this material would become more susceptible to stress corrosion cracking.

Another factor that affects SCC is tensile stress, which occurs during the operation of the heat exchanger. Also, SCC on brass material in this study can be caused by two corrosive media types, such as nitrate compounds and solutions containing ammonia [6]. One of the main factors which significantly influenced SCC in admiralty brass is the presence of Cu^{2+} ions, which come from the $\text{Cu}(\text{NO}_3)_2$ (Cuprum Nitrate) compound. Allegedly, increasing the concentration of $\text{Cu}(\text{NO}_3)_2$ will accelerate SCC propagation in brass worse than ammonia [7].

Therefore, this study will be focused on Cu^{2+} ion reactions effect, tensile stress, and addition of chloride compounds to initiate dezincification which could increase the rate of SCC [7]. This research was conducted to determine the effect of the tensile stress level and dezincification on the stress corrosion cracking testing with C44500 brass tube specimens within a nitrate environment consisting of $\text{Cu}(\text{NO}_3)_2$ NaNO_3 compounds in particular concentrations. Methods of sample preparations were carried out according to ASTM G38, also known as the C-ring method [8].

2. METHODS

The research methodology can be summarized in the following figure



Figure 1. Methodology of Research

2.1 Initial Characterization

The preparation started with sample cutting for testing purposes. This tube has a diameter of $18.54 \text{ mm} \pm 0,01$ and $1,18 \text{ mm} \pm 0,01 \text{ mm}$. From the first observation, some crusts were found due to previous work in the petrochemical industry, as shown in Fig.2. Some specimens underwent tensile tests based on the ASTM E8 standard [9], as shown in Fig. 3. Whereas another piece was tested using OES (Optical Emission Spectroscopy) to identify and characterize the chemical composition of the tube, then directly compared to ASTM B111 [4].



Figure 2. Initial Condition of Tube Sample



Figure 3. Tensile Test Specimen in Accordance with ASTM E8.

2.2 Sample Preparation

The C44500 tube, also known as Admiralty Brass, was cut into 18 small specimens following ASTM G38, as shown in Fig.4 [10]. These 18 specimens were distinguished based on the initial condition. Nine of them were washed by ceramic cleanser containing 20% of HCl for 30 minutes to reveal the dezincification effect along to cross-sections, while nine others were immersed directly into nitrate solution.



Figure 4. C-Ring Specimen following ASTM G38.

This study conducted tensile stress parameters consisting of three variations, namely 15, 45, and 85% of the yield strength (σ_y). A tensile test was conducted for UNS C44500 material to obtain a yield strength value of 206 MPa. The yield strength of the specimen is still following ASTM B111-98 standard, with a minimum yield strength of 103 MPa [11].

The following description shows the reason for selecting tensile stress for the experiments:

1. 15% σ_y . Tensile stress applied below 20% of σ_y as the minimum applied stress required for admiralty brass [4].
2. 45% σ_y . Tensile stress applied was below the minimum strength level required for tube components based on ASTM B111-98, approximately 103 MPa [12].
3. 85% σ_y . Tensile stress is close to a maximum failure tensile stress.

2.3. C-Ring Test

2.3.1 Solution Preparation

In this study, nitrate solutions used are distinguished based on concentration and ratio between Copper (II) Nitrate ($\text{Cu}(\text{NO}_3)_2$) and Sodium Nitrate (NaNO_3), as shown in Table 1 and 2 for both dezincified and non-dezincified specimens, respectively. The addition of Sodium Nitrate would increase the pH level, in contrast with $\text{Cu}(\text{NO}_3)_2$, which initiate reduction of Cu ions instead, thus making specimens were more vulnerable to the SCC phenomenon, followed by increasing propagation rate.

Table 1. Notation for Dezincified Specimens

Parameter	pH	σ_y 15%	σ_y 45%	σ_y 85%
1 M Cu(NO ₃) ₂	3,30	1	2	3
0,1 M Cu(NO ₃) ₂ + 1,8 M NaNO ₃	4,75	4	5	6
0,01 M Cu(NO ₃) ₂ + 2 M NaNO ₃	5,90	7	8	9

Table 2. Notation for Not Dezincified Specimens

Parameter	pH	σ_y 15%	σ_y 45%	σ_y 85%
1 M Cu(NO ₃) ₂	3,30	10	11	12
0,1 M Cu(NO ₃) ₂ + 1,65 M NaNO ₃	4,68	13	14	15
0,037 M Cu(NO ₃) ₂ + 2 M NaNO ₃	5,01	16	17	18

2.3.2 pH Measurement

All nitrate solutions were tested using a pH meter to ensure the effects of Copper (II) Nitrate addition. The difference of nitrate-solution color represents each pH value, which depends on the amounts of Cu(NO₃)₂ that were added. Figure 5 shows the display of pH measurement.



Figure 5. Measurement of pH

2.3.3. Immersion Test

After preparation had been done, all specimens were carried out and directly immersed into 2L Erlenmeyer contains nitrate solution distinguished based on concentrations (Fig. 6). An immersion test was done for 116-120 hours, assuming that crack propagation would occur less than 72-96 hours [13][14]. Instead of vaporizing the solution, this experimental study was conducted by immersing all specimens due to the high-temperature level required for vaporating nitrate solutions, at least from 170 °C to 308°C [15].



Figure 6. Immersion Test

2.4 Data Calculation and Final Characterization

After 120 hours of immersion, those specimens were carried out for weighing to calculate corrosion rate from mass loss by using the precision scale with accuracy ± 0.1 mg. Before weighing, all deposits from each specimen were removed from surface. After obtaining mass loss data, the corrosion rate could be calculated by using the equation below:

$$CR = \frac{K \times W}{D \times A \times T} \quad (1)$$

Where:

- CR : Corrosion Rate (mmpy)
- W : Mass Loss (grams)
- K : Constant Factor
- D : Specimen Density (g/cm^3)
- A : Surface Area (cm^2)
- T : Exposure Time (hours)

In general, the calculation of corrosion rates uses mpy (mils per year) unit as a reference, while one mil is equivalent to 10^{-3} inches. Thus, the unit expresses the reduction of thickness in specific material due to corrosion phenomena every 10^{-3} inches in a year. When converted to international units (metrics), 1 mpy is equivalent to 0.0254 mmpy, while 1 mmpy (millimeter per year) is equivalent to 39.37 mpy while 1 mmpy (millimeter per year) is equivalent to 39.37 mpy [5].

3. RESULT AND DISCUSSION

3.1 Material Analysis

Some tube samples were tested using OES, whereas the others underwent a tensile test. The result of the chemical analysis by OES is shown in Table 4. Based on the ASTM B111 standard, this material tube is known as C44500, or "admiralty brass," where contents of Cu and Zn are 70.187% and 28.939%, respectively [12].

Table 3. Chemical Composition of Tube Material

Element	Percentage (%wt)	
	OES Result	ASTM B111-98 [12]
Cu	70.1869	70-73
Zn	28.9385	25.57 – 29.08
Sn	0.8650	0.9 – 1.2
Fe	0.01093	0.06 (max)
P	0.00018	0.02-0,10
Pb	0.008933	0.07 (max)
S	0.00171	N/A

Source: ASTM B111, Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock," ASTM International, 1998

The tube sample was also subjected to a tensile test for determining its tensile properties according to ASTM E8, with the result was shown in Table 2. The most important parameters during this study were the determination of Δ , which represent the tensile stress applied for each specimen, as explained by equations (2) and (3) below:

Table 4. Mechanical Properties Result from Tensile Test

Mechanical Properties	Dark Section	Bright Section	Average
σ_y (MPa)	210	202	206
σ_u (MPa)	346	353	350
E.L. (%)	51	54	52

$$OD_f = OD - \Delta \quad (2)$$

$$\Delta = \frac{(f\pi D^2)}{4EtZ} \quad (3)$$

Where,

- OD : Outer diameter before applied with stress [mm]
- OD_f : Outer diameter after applied with stress [mm]
- Δ : Change of diameter [mm]
- f : Desired stress [MPa]
- D : Mean of diameter [mm]
- t : Wall tube thickness [mm]
- E : Modulus of elasticity [MPa]
- Z : Correction factor from D/t, available in ASTM G38.

Based on equations (2) and (3), also supported by mechanical properties data, including the geometry of specimens according to ASTM G38 [10], measurement, and some literature studies, the value of Δ for each level could be obtained as shown in Table 5:

Table 5. Value of Δ From Each Tensile Stress Level

Tensile Stress (%σ _y)	Δ (mm)
15%	0,06
45%	0,20
85%	0,38

Where, E = 110 GPa, σ_y = 206 MPa, OD = 18.54 mm, t = 1.18 mm.

3.2 SCC Results

3.2.1 Crack Length

In order to determine the effect of dezincification on SCC propagation rate, crack depth measurements were made for specimens 1,2,3,6, and 9 as dezincified specimens and 10,11,12 specimens for not dezincified specimens. Measurement of crack length was immediately done after immersion test due to catastrophic SCC propagation, leading to failure. The observation was done using a stereomicroscope, and the results are shown by Fig. 7, then plotted into a bar diagram shown in Graph 1 and 2.

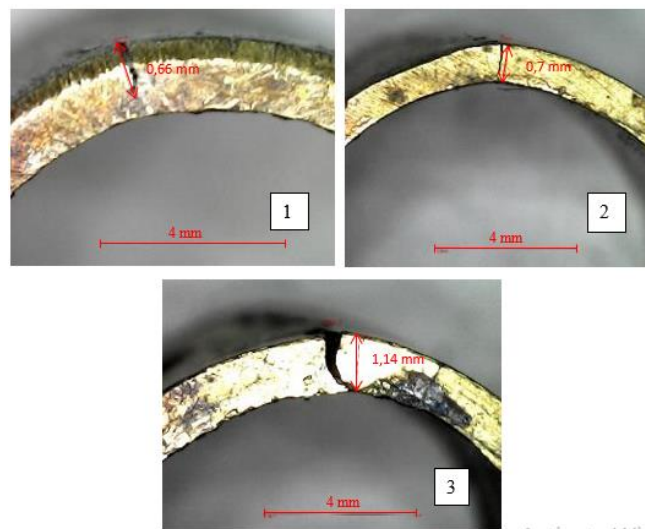


Figure 7. Crack Length Measurement

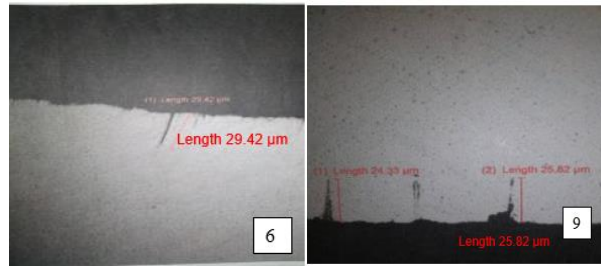
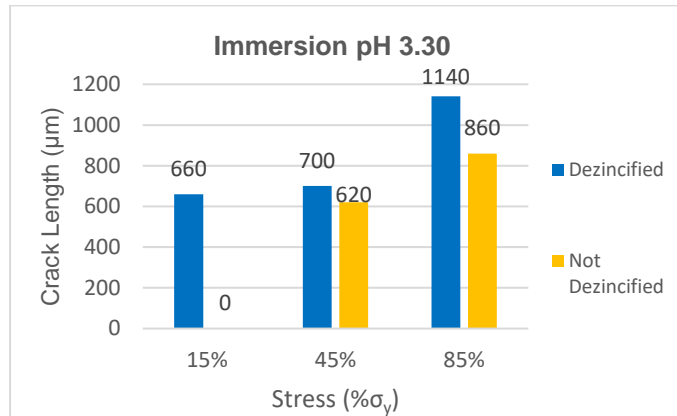
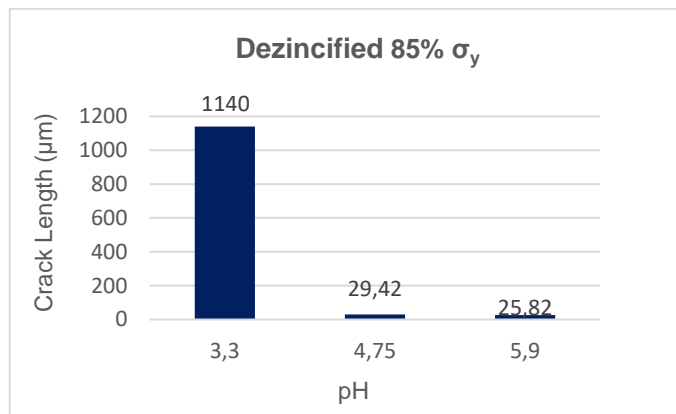


Figure 7. Crack Length Measurement (continued)



Graph 1. Crack Length Based on Stress Level

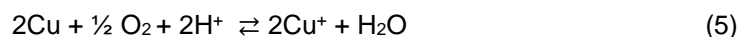


Graph 2. Crack Length Occurred on Dezincified Specimens With 85% of Yield Strength

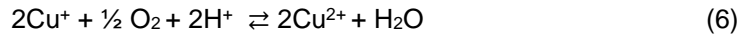
Based on results from Graph 1, it can be concluded that higher the tensile stress received by the specimen, the longer the crack depth. Otherwise, with a higher concentration of $\text{Cu}(\text{NO}_3)_2$, the SCC rate would increase significantly. The reduction of Cu supports this passivity breakdown, which explained the chemical reaction mechanism as shown below [7]:



The reduction process will dissolve Cu in the brass specimen whenever immersed into a corrosive medium, accompanied by the presence of dissolved oxygen occurs through the reaction below;



Reactions above were taken place in two steps:

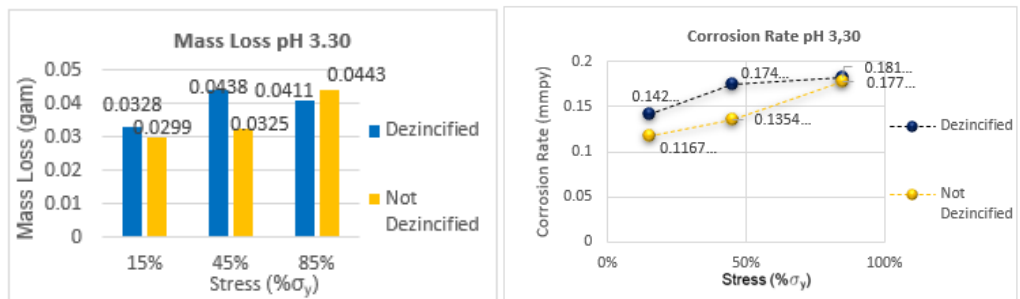


Dezincification, also known as selective corrosion, frequently occurred in Cu-Zn (Brass) alloys. This phenomenon would remove the Zn element, which remained a large amount of Cu content with a porous and brittle structure that indicates a significant decrease in mechanical properties; thus, the possibility of SCC occurring in this area would be more substantial. Generally, the dezincification phenomenon occurred in brass components directly contacted with contaminated water and particular working fluids [5].

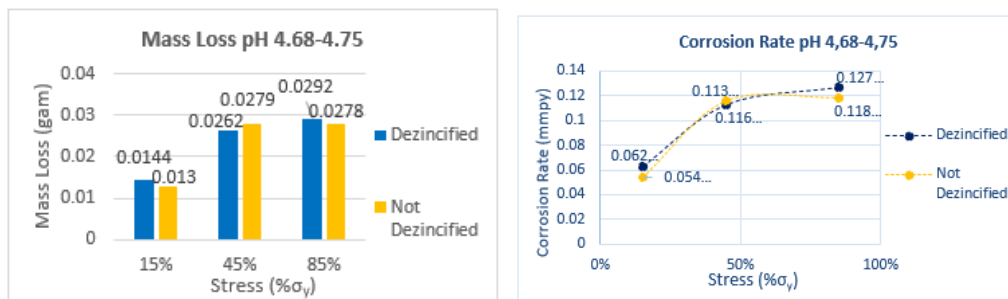
From this study, it could be concluded that dezincification had a significant role which could affect severe crack of the specimen as the primary cause of increasing propagation rate, evidenced by all of the dezincified specimens were found cracked, especially in specimens 2 and 3, which severely failed due to catastrophic SCC. Those failed specimens were measured to obtain crack depth data immediately after the immersion test. The influence of dezincification as one of SCC's main factors in brass could also be proven by severe cracks that occurred from dezincified specimens that were longer than those not dezincified by observation at tensile stress between 45%-85% and immersion test into 1 M Cu(NO₃)₂. On the other hand, neither crack nor deformation was found at specimen 10 after being subjected to a tensile stress of 15% yield strength as the lowest parameter used.

3.2.2 Mass Loss and Corrosion Rate

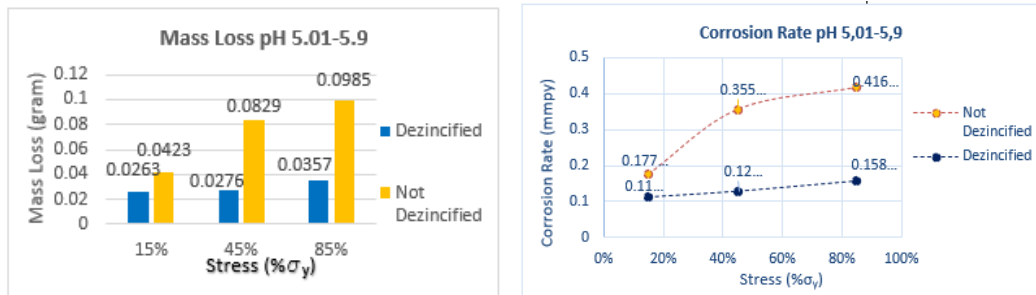
After crack lengths were observed and measured, all of them were weighed using the precision scale with 0,1 mg accuracy to obtain mass loss data due to different mass between initial condition and final condition after immersion.



Graph 3. Mass Loss and Corrosion Rate at pH Level 3.30



Graph 4. Mass Loss and Corrosion Rate at pH Level 4.68-4.75



Graph 5. Mass Loss and Corrosion Rate at pH Level 5.01-5.9

Graph 3 shows the test results with pH 3.30, where the dezincified specimen had a higher corrosion rate than the not dezincified one, affecting all tensile stress levels. This phenomenon occurred because the solution only contained high concentrations of $\text{Cu}(\text{NO}_3)_2$ without NaNO_3 . Thus, the solution increases the corrosion rate and dissolved Cu ions from the specimen surfaces during immersion.

Data obtained from Graphics 4 and 5, respectively, showed that the dezincification phenomenon affected increasing corrosion rate for all levels of tensile stress at a specific concentration of the solution, including a solution of pH 3.30. At the same time, no significant effect was found when immersion at solution pH of 4.68 – 4.75. However, an anomaly found at the highest pH solution, 5.01 – 5.9, showed that corrosion rate occurred at non-dezincified specimens were significantly higher than those of dezincified. Probably, this anomaly was influenced by the addition of NaNO_3 to increase the pH level. As a result, deposits which formed were corroded the specimen's surface uniformly.

3.3 Stereography

Observation methods were using two methods in this study: stereography and metallography. Stereography observations were made to observe the morphology of the cracked area caused by dezincification, which was observed at the severe crack area of specimen 3. Besides, observation was also for non-dezincified specimen, labeled as number 12 with highest tensile stress applied.



Figure 8. Specimens Condition After Immersion for Dezincified (left) and Non-Dezincified (right) Specimens

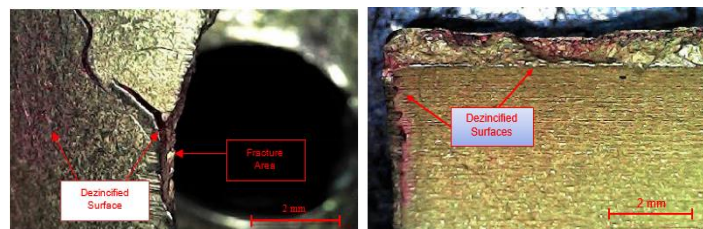


Figure 9. Stereography of Dezincified Area

Figures 8 and 9, show that the fracture surface of specimen 3 had changed from yellow into a reddish color because dezincification affects the phenomenon of stress corrosion

cracking in admiralty brass. In contrast, the others that were not dezincified were changed into the dark due to Cu reduction during the immersion test.

3.4 Metallography

Metallography observation was conducted to determine the microstructure and appearance of the grain boundary condition of the specimen, determine the type of crack that occurred, and measure the depth of crack as a result of penetration by nitrate compounds. Before metallography, the specimen was cut with an observation point along the cross-section, followed by Figure 10 below. Specimen observations were divided into three parts to determine the effect of dezincification on stress corrosion cracking phenomenon and the type of dezincification that occurred in the specimen after being washed by 20% HCl ceramic cleaning solution for 30 minutes.

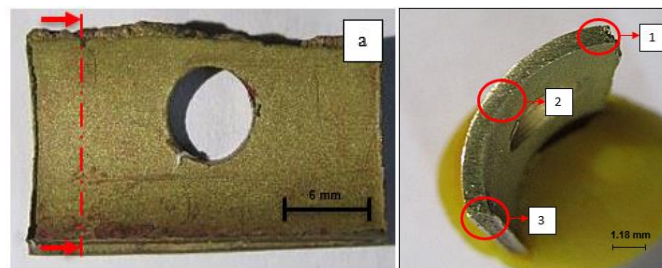


Figure 10. Cross-Section of Specimen 3

The microstructure of the specimen in the cross-section of the tube was observed using an optical microscope (Nikon Eclipse MA 200), shown in the following Figures.

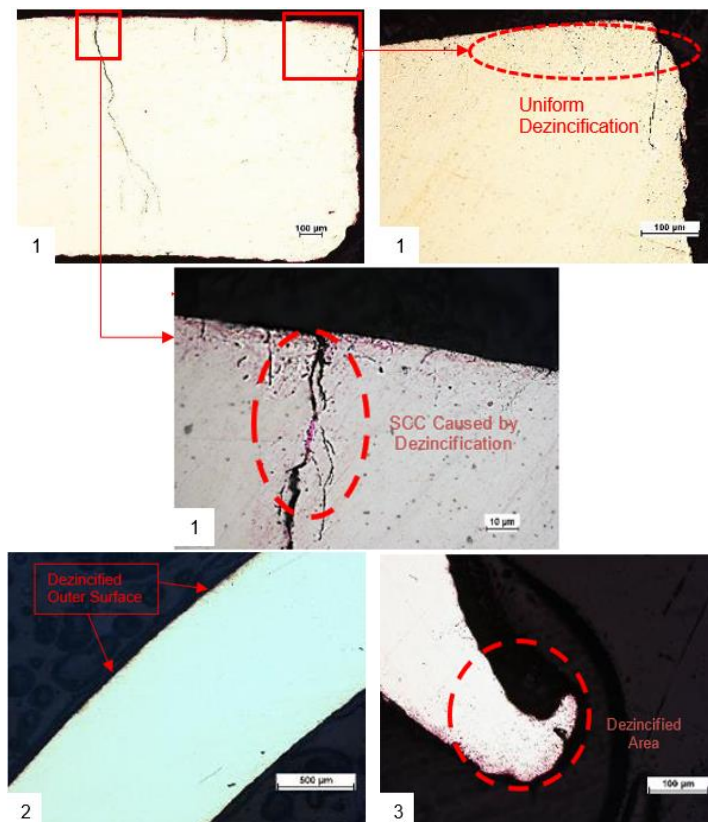


Figure 11. Metallography of Dezincified Specimen

Figure 11 shows the dezincification effect that led Admiralty Brass to become more vulnerable to SCC, as many cracks were found at the specimen tip caused by uniform dezincification. According to the metallurgical aspect, brass containing Zn > 15% often undergoes dezincification. The loss of Zn content in brass was also accelerated by conditions such as high temperature, increasing chloride content, low flow rates, and differential aeration [5]. The dezincified and non-dezincified specimens microstructures were observed, as shown in Figure 12.

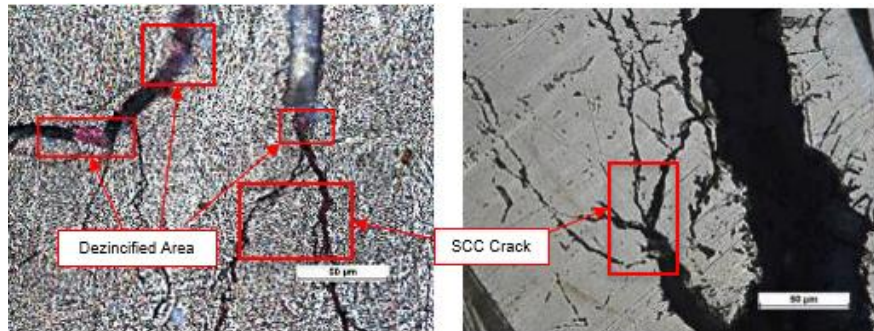


Figure 12. Crack Observation, Dezincified (left) and Not Dezincified (right)

Figure 12 shows the surface crack morphology in both dezincified and non-dezincified specimens. It can be seen that the dezincified specimen has a reddish surface around the crack area which indicates depletion in Zn (%wt) content due to dissolution by the HCl compound contained in the ceramic cleanser. As a result, the dezincified specimen would remain porous and brittle structures which accelerate SCC propagation [5].

Furthermore, metallography observation was also conducted to observe the cracks that occurred on admiralty brass. Generally, two possible types of crack occurred as the result of the SCC phenomenon; intergranular and transgranular. As shown by Figure 13, it was concluded that SCC in brass specimen would occur by transgranular crack:

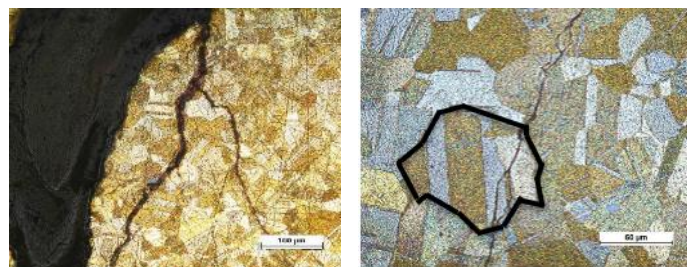


Figure 13. Microstructure of Transgranular Crack

Figure 13 above could explain that transgranular crack occurred at all of the brass specimens after immersion. This theory was referred to K. Lian *et al.*, who stated transgranular commonly occurred in brass with Zn content >18% wt), dezincified or not [16]. In addition, the two specimens also meet three prerequisite conditions that trigger the phenomenon of transgranular cracking, according to the theory proposed by Scully *et al.* [17]:

1. Alloy transition occurred both actively and passively, so the reaction would produce a surface with a higher potential. For example, the formation of oxides and/or metal elements.
2. An alloy that exhibits a coplanar dislocation arrangement caused the alloy matrix to have a higher hardness rate.
3. Specific electrochemical reactions occurred on the surface. For example, Cl⁻ penetrates the passive layer of alloy.

3.5 Deposit Characterization and Corrosion Product

At the first observation, the specimen has golden yellow color shown in Figure 14. After immersion test into nitrate solution, the morphology of those specimens surface was changed into several colors, such as dark gray and greenish-blue forms, which depends on pH-level from each nitrate compound.

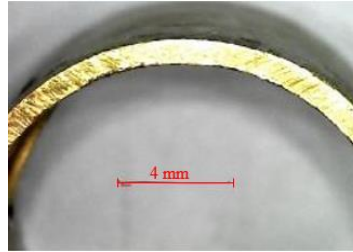


Figure 14. Initial Condition of Admiralty Brass Tube

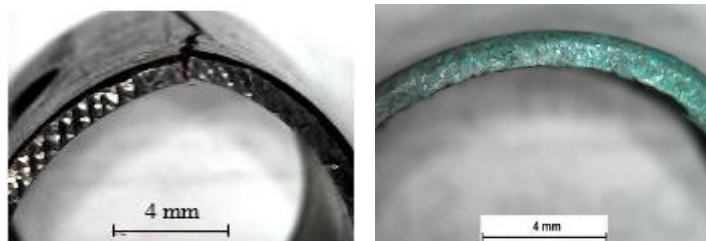


Figure 15. Change of Tube Colors After Immersion Test Into pH 2,7 and 5,01 solution

The surface deposits were characterized using SEM-EDS with spot analysis in certain areas. Table 6 and Figure 21 below shows EDS results from deposits obtained from non-dezincified specimens. The specimen was immersed into a solution with pH 5.01, which produced green deposits, and a lower pH produced dark surfaces.

Table 6. Chemical Composition of Deposit After Immersion Into pH 5,01 solution

Element	Weight (%wt)	Atom (%)
Cu	63.20	30,19
O	36,80	69,81

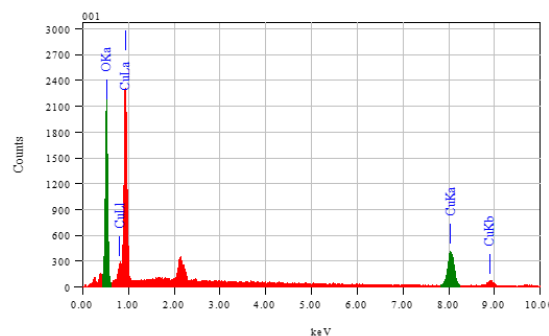
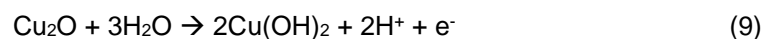


Figure 15. EDS Spectrum of Deposit Obtained from Immersion Into pH 5,01 Solution

Based on spot analysis observation, only two elements were detected in the deposit, Cu, and O, without Zn as an alloying element, and other elements that could be considered impurities. The Cu_2O deposit was formed according to equation (9) to form a $\text{Cu}(\text{OH})_2$ with the increase in the solution pH.



The chemical reaction above could be assumed that Zn has completely dissolved during immersion. Thus, the deposited compound formed is $\text{Cu}(\text{OH})_2$, which is indicated by the ratio between Cu and O atoms were 3:7, equivalent to 1:2.3 [18].

4. CONCLUSION

Based on the experiment, there is an increase in the crack depth with the increase in $\text{Cu}(\text{NO}_3)_2$ concentration. All specimens immersed in 1 M $\text{Cu}(\text{NO}_3)_2$ (pH 3.30) cracked due to SCC, exception for specimen 10 (15% σ_y), which did not experience any crack or deformation. Specimens immersed in the test solution with pH 4.75 and 5.9) resulted in tiny cracks, with crack depths of 29.42 and 25.82 mm, respectively. There is an increase in the crack depth with increased tensile stress. The crack depth can be observed in specimen 1 (15% σ_y), specimen 2 (45% σ_y), and specimen 3 (85% σ_y) of 660, 700, and 1140 mm, respectively for the dezincified condition. For non-dezincified specimen 11 (45% σ_y) and specimen 12 (85% σ_y) occurred crack depths of 620 mm and 860 mm, respectively, just prior to catastrophic failure.

Dezincification of the specimen formed pitting as the initiation stage of the SCC crack, followed by propagation of branching cracks with varying depths. From the results of metallographic observations, the fractures that appeared in both dezincified and not dezincified specimens were transgranular. In general, dezincified specimens had a higher corrosion rate than not dezincified specimens, except in the pH range of 5.01-5.9 where specimens with no dezincified conditions had significantly higher corrosion rates, 0.177, 0.356, and 0.416 mmpy (millimeters per year), respectively. It was suspected that those specimens which not dezincified s had occurred uniform corrosion due to high concentration of NaNO_3 were added so that large amounts of patina $\text{Cu}(\text{OH})_2$ were formed as deposit compounds with its color greenish-blue.

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