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D A F T A R I S I

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Teknik Sipil dan Perencanaan:

Teknologi Industri

THE EFFECT OF SENSITIZATION TEMPERATURE ON STRESS CORROSION CRACKING OF STAINLESS STEEL AISI 304 IN SULPHATE ACID ENVIRONMENT

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ABSTRACT

Austenitic stainless steel 304 (AISI 304) has a good corrosion resistant, but a heat treatment process can cause a sensitization phenomenon and the corrosion resistant will decrease. One of the most serious corrosion problems that can occur is stress corrosion cracking (SCC). This study is to determine the effect of sensitization temperatures (i.e. 500 ^oC, 670 ^oC, and 840 ^oC) on the SCC phenomenon of AISI 304 in sulphate acid environment with total immersion method. It was observed for the specimen with sensitization temperatures of 500 ^oC and 670 ^oC, the dominant corrosion form is pitting corrosion, where the highest corrosion rate was occur on the specimen with sensitization temperature of 500 ^oC after 336 hours of exposure. Average of Corrosion Rate of specimen with sensitization temperatures of 500 ^oC, 670 ^oC, and 840 ^oC after 480 hours of exposure are 2.76 mm/y, 1.58 mm/y, and 0.9 mm/y. Stress corrosion cracking occurs on the specimen with sensitization temperature of 840 ^oC after 480 hours of exposure.

Keywords: *stress corrosion cracking, sensitization temperature, and total immersion.*

ABSTRAK

Baja tahan karat austenitik AISI 304 memiliki ketahanan korosi yang baik, namun bila mengalami perlakuan panas dapat terjadi fenomena sensitisasi sehingga akan menurunkan ketahanan korosinya. Fenomena korosi yang dapat terjadi terutama adalah korosi retak tegang (SCC). Penelitian ini dilakukan untuk mengetahui efek temperatur sensitisasi (500 ^oC, 670 ^oC, dan 840 ^oC) terhadap fenomena SCC pada AISI 304 di lingkungan asam sulfat dengan metoda perendaman total. Dalam penelitian ini ditemukan bahwa temperatur sensitisasi 500 ^oC dan 670 ^oC akan menyebabkan korosi *Pitting*, angka laju korosi tertinggi ditemukan pada specimen dengan temperatur sensitisasi 500 ^oC setelah 336 jam perendam. Laju korosi rata-rata setelah 480 jam perendaman untuk temperatur sensitisasi 500 ^oC, 670 ^oC, dan 840 ^oC masing-masing adalah 2.76 mm/y, 1.58 mm/y, dan 0.9 mm/y. Fenomena korosi retak tegang ditemukan pada spesimen dengan temperatur sensitisasi pada 840 ^oC setelah 480 jam perendaman.

Kata kunci: korosi retak tegang, temperatur sensitisasi, dan perendaman total.

INTRODUCTION

Austenitic Stainless Steels are the most widely applicated steel in industry, about 70-80% of stainlless steel production. AISI 304 is also the most popular Chrom stainless steel that has a good corrosion resistance and mechanical properties. AISI 304 is the iron-based alloy that contains more than 12% Chromium that can forms a protective oxide film on the surface to hence the corrosion resistance. Oxide film can regenerate naturally when the surface of steel was damage. However, the precipitation process of Chrom-Carbide due to the heat treatment processes or welding processes can cause the sensitization phenomenon and later will promote a spesific corrosion phenomenon, namely environmentally induced cracking (EIC).

Environmentally Induced Cracking (EIC) is general term for brittle mechanical failure that results from a synergism of tensile stress, susceptible material, and corrosive environment. Corrosion rate is usually quite low and design stresses to cause EIC are often below the yield stress. Stress Corrosion Cracking is the popular phenomenon at EIC (Jones, 1991).

Sensitization is the phenomenon based on the Chromium-depleted zone at the grain boundary regions. For example, in welding processes of various stainless steels, Heat Affected Zone (HAZ) will be sensitized in which cause Chrom (Cr) move to the grain boundaries while at the other regions will have less Chrom even can be considerably below 10 – 12 % that required to create an oxide film. When Cr moves to the grain boundaries, it will precipitates to forms $Cr_{23}C_6$ and the regions with less Cr content will have less corrosion resistant and known as Chromium-depleted zone (Decker & Langer, 1987).

It is known that the effect of sensitization phenomenon of austenitic stainless steel is a great importance, especially for the application at Oil Company, chemical and nuclear industry and also shipping company. Previous study has been investigated at boiling water condition and with the H_2SO_4 environment that the intergranular cracking may arise after 72 hours period of exposure (Jones, 1991).

In Austenitic stainless steel, an intermediate temperature range of 425 to 840 ^{0}C (840 – 1550 ^{0}F) Chrom carbides, $(Fe, Cr)_{23}C_6$ are insoluble and precipitate at grain boundaries. Above 840 ^{0}C the Chromium carbides are soluble; below 425^{0}C the diffusion rate of carbon is too low to permit formation of the carbides. Figure 1 show that the Chromium carbide precipitates are very high in Chromium, but the matrix alloy is depleted of Chromium in the grain boundaries.

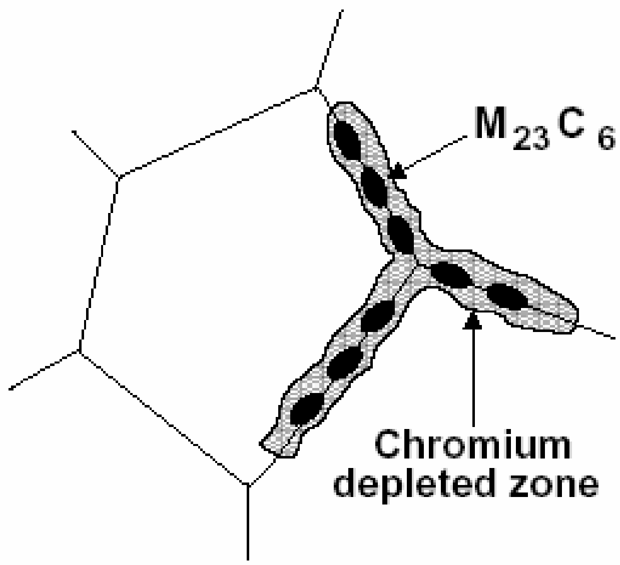
Previous researcher reported the microstructure of sensitized austenitic stainless steel. The specimen was prepared with a solution treatment at 1050 ^{0}C for 1 hour followed by water quenching. For the sensitization process, samples were immersed in a batch of solution-treated at 670 ^{0}C, in a muffle furnace for 4 hours. For the testing of sensitization, the specimen was subjected to the oxalic acid etch test. A ditch microstructure was taken to be confirmation of the sensitization (Singh, 1987).

Carbide precipitate (i.e. $M_{23}C_6$) also can induce by the welding process of austenitic stainless stell. It causes Chromium depleted zone near the grain boundaries and then decrease the stress corrosion cracking resistant of the welded stell as reported by Gooch, (1984).

Other researcher founded that SCC can occur when the steels are expossed to the “high pH” as well as “near-neutral pH” or “low pH” solution (Fang, *et al*., 2003). Stress corrosion cracking of AISI 321 stainless steel on acidic chloride environment also has been reported by other researcher (Huang, 2001).

Although massive progresses have been made, the corrosion forms and processes that associated with SCC of austenitic stainless steel on the sulphate acid environment are still not clear. The objective of this study was to determine the effect of sensitization temperatures on the SCC process of austenitic stainless steel AISI 304 on the sulphate acid (H_2SO_4) environment.

Three different sensitization temperatures (i.e., 500 ^{0}C, 670 ^{0}C, and 840 ^{0}C) have been chosen in this study. The variations of sensitizations temperature are based on the diagram of Time-temperture-precipitation for $M_{23}C_6$ of AISI 304 as shown in Figure 2 (Sourmail, *et al*., 2003).



(a)

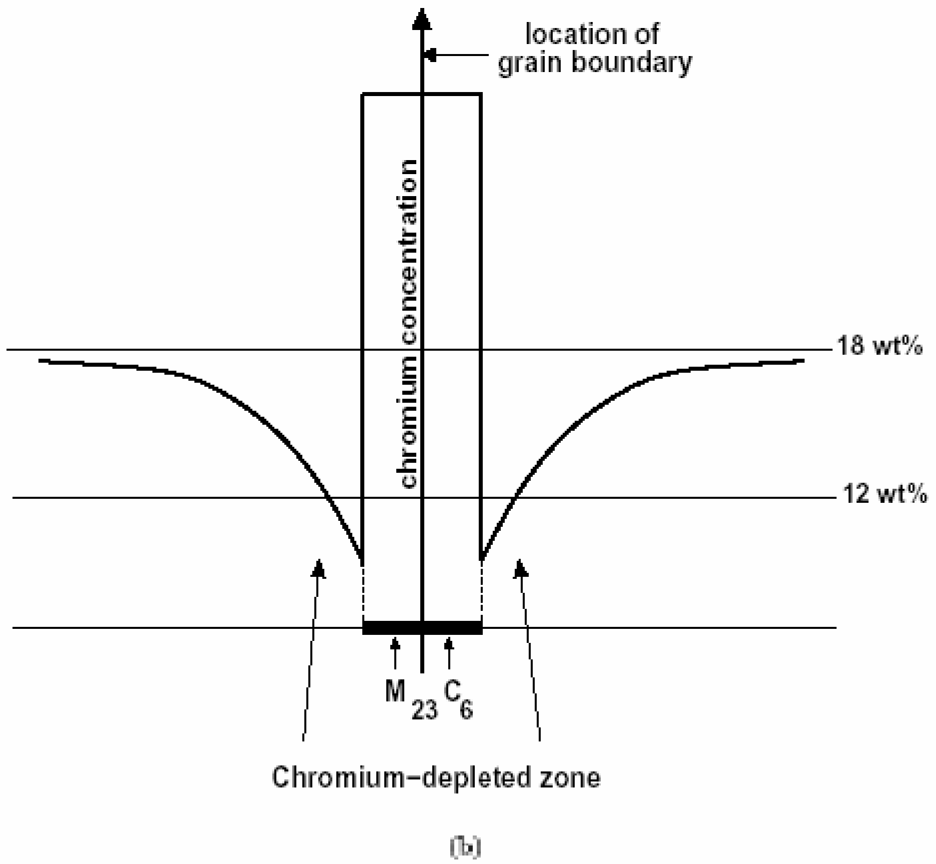


Figure 1 Chromium Depleted Zone at the Grain Boundaries (Too, C.H., 2002)
(a) Chromium depleted zone and $M_{23}C_6$ precipitates (b) Chrom concentration

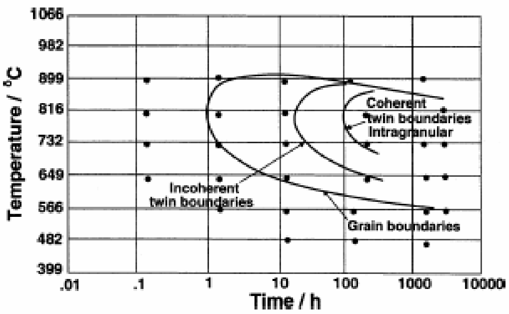


Figure 2 Time-Temperature-Precipitation for $C_{23}C_6$ in 304 austenitic stainless steel

METHODOLOGY

Material and Experimental Procedures

Material that used in this study was AISI 304 and specimen was prepared based on ASTM standard (Decker & Langer, 1987) and for preparation, cleaning, and evaluation was based on ASTM G 1 – 72 standards (Annual Book of ASTM, 1979).

Generally, a testing specimen was prepared by cutting the AISI 304 industrial standard plate into strips with a specific dimension (Figure 3).

For the sensitization process, the strips were then placed on the salt bath furnace that set in three different temperatures (i.e. 500 ^{0}C, 670 ^{0}C, and 840 ^{0}C) for 4 hours of sensitization time and then quenched in water.

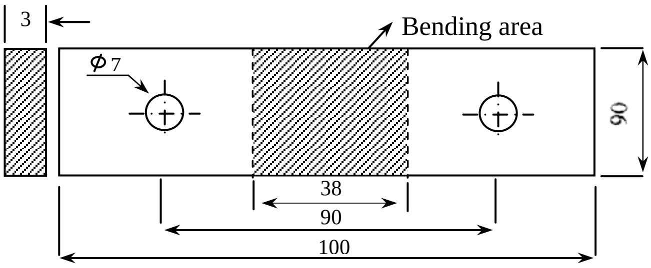


Figure 3 Dimension of the specimen

To clean the oxide film at the surface that caused by sensitisation process, specimen was immersed on the aquadestilata solution containing

10% (volume/volume) of HNO_3 for 1 hour at temperature of 60 ^{0}C (Annual Book of ASTM, 1979).

U-Bend Corrosion Test Specimen

After the cleaning process, the strips were then bent approximately 180^0 around a predetermined radius and maintained in this plastically (or elastically) deformed condition during the SCC test. The bending method was based on ASTM G30 and the final specimen is shown in Figure 4.

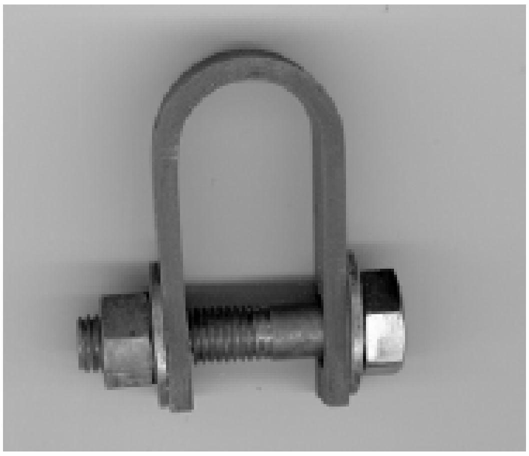


Figure 4 Final U Bend Specimen

Exposure Method

There are two types of SCC test expossure as suggested by the ASTM Standard, i.e. cyclic immersion and total immersion (Annual Book of ASTM, 1979). This study was conducted with total immersion method, where the specimen was immersed on the suphate acid until the evaluation time as shown in Figure 5 (not the exact scale).

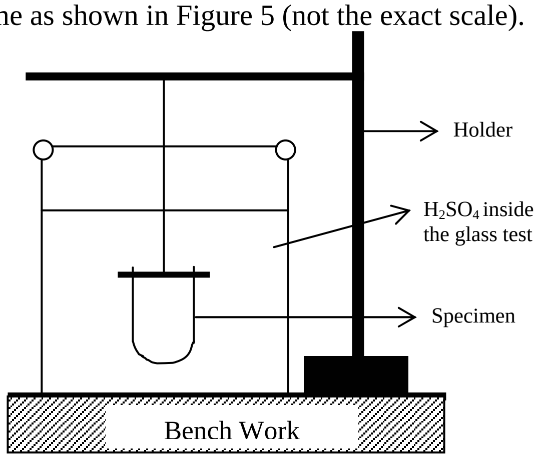


Figure 5 Total Immersion Method

Weight Loss Evaluation

The calculation of the average corrosion rate can obtain with the Weight Loss method based on the ASTM standard as follows (Jones, 1991):

Corrosion Rate = $(K \times W)/(A \times T \times D)$..1

Where:

K = a constant.
T = time of exposure in hours to the nearest 0.01 h
A = area in cm^2 to the nearest of 0.01 cm^2
W = mass loss in g, to nearest 1 mg
D = density in g/cm^3.

Corrosion Forms and Micro Structure

To better understanding of the initiation and propagation process of EIC, the evaluation will be based on the corrosion forms and micro-struture. Generally, several corrosion forms will associated with EIC of austenitic stainless steel at acidic environments, there are:

(a) Uniform corrosion,
(b) Pitting corrosion, and
(c) Intergranular, and
(d) SCC.

The evaluation of uniform and pitting corrosion will be based on the macro structure evaluation while inter granular and stress corrosion cracking were by the micro-structure analysis.

Pitting corrosion will be determined by the number of cases per surface area and the average deep of pitting's penetrations.

RESULT AND DISCUSSION

Comparison between the structure of non-sensitized and sensitized steels (i.e. 500 ^{0}C, 670 ^{0}C, and 840 ^{0}C) was presented in Figure 6. The micro structure of non-sensitized steel indicates the regular forms at the grain boundaries region, while on the sensitized ones indicate the dich structure. Higher sensitization temperature tends to produce dicher grain boundaries.

It can be hypothesized that the black dots on the grain boundaries and ditch structures were associated with the precipitation of $Cr_{23}C_6$ as also suggested by other researchers (Huang, 2001; Sourmil, 2003; and Too, 2002).

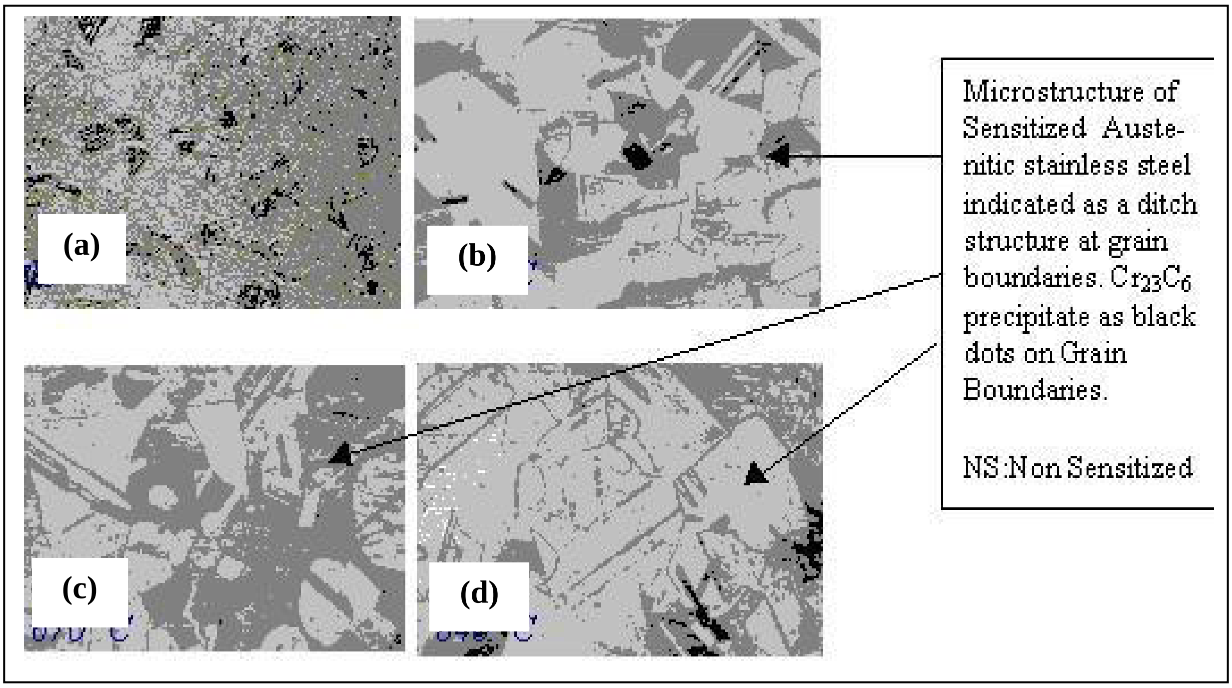


Figure 6 Micro Stucture of Stainless Steel AISI 304 (magnification 360x)
(a) Non-Sensitized (b) Sensitized at 500 ^{0}C (c) 670 ^{0}C, and (d) 840 ^{0}C

Table 1 Result of the Corrosion Test

Sensitization Temperature (^{0}C)	Time of Expossure (Hours)	Weight Loss (grams)	Corrosion Rate (mm/y)	Average of CR (mm/y)
	48	0.19	2.1	
500	192	0.37	1.02	2.76
	336	2.93	4.49	
	480	3.23	3.44	
	48	0.19	2.1	
670	192	0.33	0.9	1.58
	336	0.88	1.35	
	480	1.83	1.95	
	48	0.14	1.51	
840	192	0.15	0.41	0.90
	336	0.19	0.29	
	480	1.29	1.4	

Data of weight loss, Corrosion Rate (CR) as well as average of CR over the exposure time were presented on Table 1. The CR was calculated based on equation (1).

Weight loss trend during the exposure time for 3 different sensitization temperatures can be presented as relation between weight loss and time of exposure as shown in Figure 7.

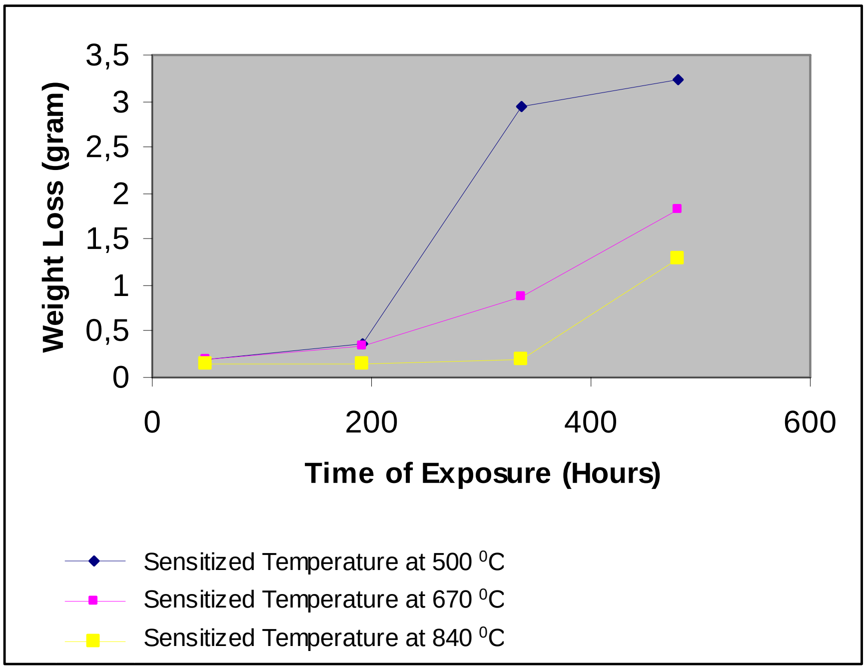


Figure 7 Trend of Weight Loss during Exposure

Table 2 Corrosion Forms and the Regions

Sensitized Temperature	Time of Expossure (Hours)	Corrosion Forms	Corrosion Regions
	48	Pitting	Peak dan 15^o
500 ^{o}C	192	Pitting	15^o, 30^o
	336	Pitting	Peak, 15^o, 30^o
	480	Pitting	Peak, 15^o, 30^o
	48	Pitting	15^o
670 ^{o}C	192	Pitting	15^o, 30^o
	336	Pitting	Peak, 15^o, 30^o
	480	Pitting	15^o, 30^o
	48	Uniform	All surface
840 ^{o}C	192	Pitting	15^o, 30^o
	336	Pitting	Peak, 15^o, 30^o
	480	Pitting, Inter-granular & SCC	Peak, 15^o, 30^o

Based on Table 1 above, the highest CR is 4.49 mm/y where it was occur on the specimen with sensitization temperature of 500 ^{0}C after 336 hours of exposure.

The average CR number of sensitization temperature of 500 ^{0}C (CR = 2.76) was higher than 600 ^{0}C (CR = 1.58) and 800 ^{0}C (CR = 0.90) respectively. Based on Figure 7 above, until 192 hours of exposure, the trend of weight loss of every sensitized specimen were relatively equal. After 192 hours, the specimen that sensitized on 500 ^{0}C gives the highest weight loss number. Since the weight loss number was calculated based on the data of uniform corrosion, the other corrosion forms will be evaluated separately

Table 3 Penetration of Pitting Corrosion after 480 hours of exposure

Time of Exposure (Hours)	Sensitized Temperature (^{o}C)	Number of Pitting Corr.	Average Penetration
	500	68	0.07%
480	670	147	0.14%
	840	138	0.12%

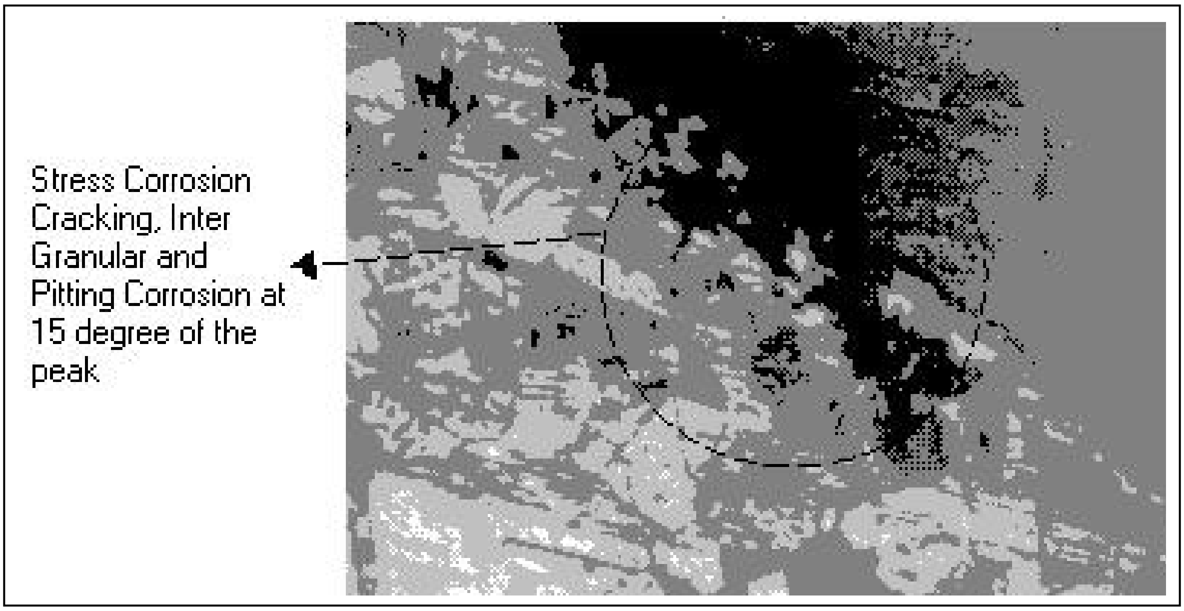


Figure 8 SCC and IG Corrosion of sensitized specimen at 840 ^{0}C (magnification 180x)

Data of the forms and corrosion region that evaluated with the macro and micro-structure analyses was presented on Table 2. The Evaluation of penetration of pitting corrosion after 480 hours of exposure is shown on Table 3.

Based on Table 2 above, stress corrosion cracking and IG corrosion were not occur on the specimen that sensitized at 500 ^{0}C or 670 ^{0}C but only pitting corrosion, even after 480 hours of exposure. For specimen that sensitized at 840 ^{0}C, SCC was initiated by uniform after 48 hours of exposure, followed by pitting and finally inter-granular and SCC corrosion after 480 hours. The inter-granular as well as SCC forms that founded in this study are presented in Figure 8. It can be hypothesed that the sensitization temperature of 840 ^{0}C is the most potential one on inducing the EIC of stainless steel AISI 304 when exposed on sulphate acid environment. Based on Table 3 above, the sensitization temperature of 670 ^{0}C was give the highest number of pitting corrosion as well as average pitting penetration, followed by 840 ^{0}C and 500 ^{0}C respectively

SCC process and the corrosion forms during the exposure time of sensitized steel at themperature of 840 ^{0}C, can be described as follow:

Uniform Corrosion → Pitting nucleation → pitting growth → Inter-Granular → SCC.

Our finding of the continous process of stress corrosion cracking that initiated by several corrosion forms was in good agreements with results that had already reported by other researchers (Meng,. *et al.*, 2003 and Krawiec,. *et al.*, 2006).

CONCLUSIONS

This study reported the validation and process of SCC of austenictic stainless steel AISI 304 with various sensitized temperatures and expossed at sulphate acid environment and it can be concluded as follows:

1. Sensitized temperature of 500 ^{0}C give the higest average of corrosion rate (2.76 mm/y) and followed by 670 ^{0}C (1.58 mm/y) and 840 ^{0}C (0.9 mm/y).
2. Dominant corrosion form of specimen sensitized at 500 ^{0}C and 670 ^{0}C is pitting corrosion without inter-granular as well as SCC, even after 480 hours of exposure.

Inter-Granular and SCC forms were occur on the specimen that sensitized at temperature of 840 ^{0}C.

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