



STRESS CORROSION CRACKING OF NICKEL ALUMINIUM BRONZE
UNDER AMMONIA-CONTAINING ARTIFICIAL SEAWATER

By

MR. WITCHAPONG SOCHU

A DISSERTATION SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF DOCTOR OF PHILOSOPHY

PROGRAM IN ENGINEERING

THAMMASAT SCHOOL OF ENGINEERING

THAMMASAT UNIVERSITY

ACADEMIC YEAR 2020

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DISSERTATION

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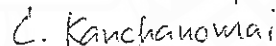
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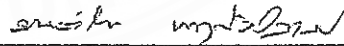
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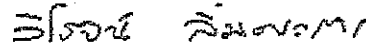
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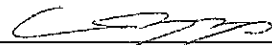
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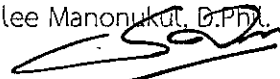
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Dissertation Title	STRESS CORROSION CRACKING OF NICKEL ALUMINIUM BRONZE UNDER AMMONIA-CONTAINING ARTIFICIAL SEAWATER
Author	MR. Witchapong Sochu
Degree	Doctor of Philosophy Program in Engineering
Faculty/University	Thammasat School of Engineering Thammasat University
Dissertation Advisor	Prof. Dr. Chaosuan Kanchanomai
Academic Years	2020

ABSTRACT

With the growth of urbanization and industries, the seawater near coastal areas has become polluted, and the nickel aluminium bronze (NAB) components around coastal areas are affected by ammonia-containing seawater. Unfortunately, there are some limitations for the evaluation of stress corrosion cracking (SCC) of NAB under ammonia-containing seawater. Firstly, the influence of the ammonia concentration in seawater on the stress corrosion cracking (SCC) of thin NAB components with large plastic zones at the defects (i.e., plane stress condition) has not been evaluated before. Secondly, for a thick NAB component with small plastic zone at the defects (i.e., plane strain condition), some parts of SCC experiment are needed to be improved, i.e., the accurate measurement of crack length and the suitable fracture mechanics parameter at crack front.

Under plane stress condition, SCC experiments on NABs under artificial seawater and ammonia-containing artificial seawater were conducted using a 4-point bending technique. The elastic-plastic fracture mechanics parameter (J -integral) was evaluated using finite element analysis (FEA). The J -integral successfully characterized the crack growth rate (da/dt) under the present corrosive environments. SCC was possible under both artificial seawater and ammonia-containing artificial seawater. The threshold J -integral for susceptibility to SCC (J_{SCC}) and fracture toughness (J_c) were the highest for SCC under artificial seawater and decreased as the amount of ammonium hydroxide added to the artificial seawater increased.

Under plane strain condition, the self-deformed specimen (i.e., T-type wedge opening loaded specimen or T-WOL specimen) is used for SCC experiment. Unfortunately, the direct measurement of crack length during SCC experiment is not accurate because of the influences of liquid environment and corrosion product. In the present work, an elastic-plastic finite element analysis was applied to estimate the crack length from the measured crack-opening force and the measured crack-mouth opening displacement. To measure the crack-opening force during SCC experiment, the instrumented reaction pin was numerically designed, constructed, and calibrated. Crack lengths from indirect crack length measurements were compared with those from direct measurements. Both results were in good agreement, and the present indirect crack length measurement can be used for SCC experiment with T-WOL specimen. Moreover, under the combined effects of stress and a corrosive environment, the protective layer at the crack tip of NAB component can be damaged, and the resistance to crack propagation can be reduced. Therefore, the measurement of crack-tip opening displacement (CTOD) should be applied to characterize the crack propagation rate (da/dt) during SCC experiments. In the present work, CTOD of a T-WOL specimen with a built-in loading bolt was initially determined using 3D FEA. The influence of stress concentration from side groove on CTOD was clearly evaluated, i.e., the fluctuation of CTOD occurred near the groove-tip plane. However, CTOD became stable on > 90% of thickness of T-WOL specimen. Therefore, CTOD at mid-thickness plane of T-WOL specimen could be applied for SCC experiment.

Keywords: nickel aluminium bronze; stress corrosion cracking; ammonia-containing seawater; plane stress/plane strain; J -integral; T-type wedge opening loaded specimen; crack-opening force; crack-tip opening displacement; finite element analysis

ACKNOWLEDGEMENT

Foremost, I would like to express my sincere gratitude to my advisor Prof. Dr. Chaosuan Kanchanomai for the continuous support of my Ph.D. study and research. His motivation, patience, enthusiasm, and consistent guidance helped me in all the time of research and writing of this thesis.

Besides my advisor, I would like to thank the rest of my thesis committee: Assoc. Prof. Dr. Wiroj Limtrakarn, Asst. Prof. Dr. Monchai Pruekwilailert, Assoc. Prof. Dr. Jirapong Kasivitamnuy and Dr. Anchalee Manonukul, for their encouragement, insightful comments, and hard questions.

I thank my colleagues in Fracture Mechanics and Fatigue laboratory, Department of Mechanical Engineering, Thammasat University, i.e., Dr. Nitikorn Noraphaiphaksa, Mrs. Suparat Bootchai and Ms. Kotchaporn thadsoongnoen, as well as all the students for the stimulating discussions, great supports, and for all the fun we have had throughout the time of my Ph.D. study.

This research was performed with the financial support by Thammasat School of Engineering (TSE) and Ph.D. scholarship from Thammasat University. The nickel-aluminum bronze was supplied by the Royal Thai Navy.

Last but not the least, I would like to thank my parents, for support and encourage all the time of my life. Also, I am extremely grateful to Ms. Yarawee Saelao for her love, understanding and continuing support to bring this research work into success.

Mr. Witchapong Sochu

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Nomenclature

AH	ammonium hydroxide
ASTM	American Society for Testing and Materials
a	crack length
α	hardening coefficient
B	thickness of T-WOL specimen
B_n	net thickness of T-WOL specimen
BS	British Standards
CMOD	crack-mouth opening displacement
CTOD δ_5	crack-tip opening displacement defined for a gage length of 5 mm
CTOD 45°	crack-tip opening displacement defined as a distance between two intersections of $\pm 45^\circ$ lines from the crack tip with crack surfaces
CT specimen	compact-tension specimen
da/dt	crack propagation rate
ds	length increment along the path Γ
θ	polar coordinate at the crack tip
ΔK	stress intensity factor range
E	modulus of elasticity
EAC	environment-assisted cracking
EDM	electrical discharge machine
EDS	energy-dispersive X-ray spectroscopy
EPFM	elastic-plastic fracture mechanics
ε	strain
ε_{bf}	back-face strain
ε_{ij}	strain tensors
F	applied force
F_L	limit force corresponding to a fracture
FEA	finite element analysis

ISO	International Organization for Standardization
J -integral	release rate of the potential energy associated with crack propagation
J_c	critical J -integral
J_i	initial J -integral
J_{SCC}	threshold J -integral for susceptibility to stress corrosion cracking
K	stress intensity factors
K_I	stress intensity factor under opening mode
K_{SCC}	threshold stress intensity factor under opening mode for susceptibility to stress corrosion cracking
K_c	critical stress intensity factor
LEFM	linear-elastic fracture mechanics
MPC	multi-point constraint
μ	friction coefficient
NAB	nickel aluminum bronze
n_j	components of the unit vector normal to the path Γ
ν	Poisson's ratio
RSL test	rising step load test
R^2	coefficient of determination or R squared
r	size of element at crack tip
r_c	cyclic plastic zone at crack tip
SCC	stress corrosion cracking
SCCG curve	stress corrosion crack growth curve
SENB specimen	single-edge notch bending specimen
SW	artificial seawater
σ	normal stress
σ_{ij}	stress tensors
σ_n	normal stress
σ_Y	yield stress
T_i	components of the traction vector

t	time
T-WOL specimen	T-type wedge opening loaded specimen
τ	shear stress
τ_c	critical shear stress
u_i	components of the displacement vector
V	crack-mouth opening displacement on T-WOL specimen's face
W	distance from the load line to the back surface of T-WOL specimen
WES	Japan Welding Engineering Society Standard
w	strain energy density
4PBT	4-point bending technique



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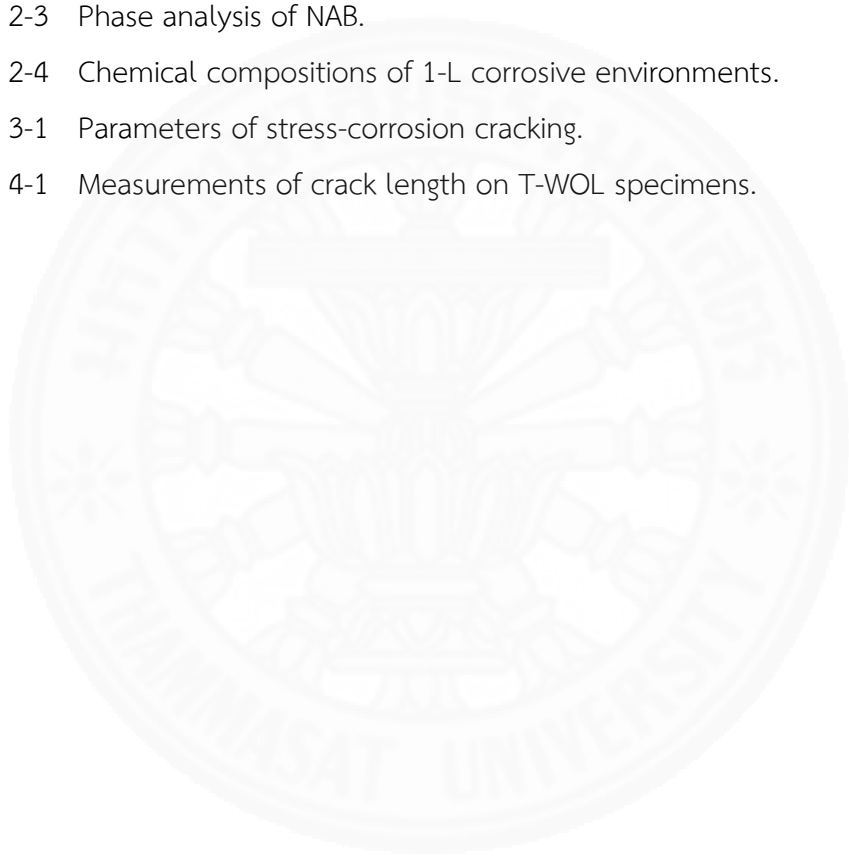
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Chapter 1

Introduction

1.1 Background

Nickel aluminium bronze (NAB) is a copper alloy that contains 8.5-9.5 wt.% aluminium, 4-5 wt.% nickel, and 3.5-4.5 wt.% iron. For these alloys, aluminium can improve the casting and hot working characteristics, as well as the strength; nickel can improve the corrosion resistance, yield strength, and microstructure stabilization; and iron assists in grain refinement and improves the tensile strength [1]. The corrosion resistance of NABs can be optimized by controlling their composition and by heat treatment [2]. Because both cast and wrought NABs have a good combination of mechanical and corrosion resistance properties, they have been used in various marine applications, e.g., valves, propeller blades, pumps, shafts, and heat exchangers [3].

With the growth of urbanization and industries, the seawater near coastal areas has become polluted due to the release of sewage, agricultural water, and industry wastewater into the water system. Among the various pollutants (e.g., ammonia, sulphides, nitrogen, and phosphorus), ammonia is a major concern for countries with agricultural activities. In addition to seawater being an aggressive corrosive environment, the NAB components around coastal areas are also affected by ammonia-containing seawater. As the gradual destruction resulting from the electrochemical reaction, corrosion is possible for NAB components used in service environments exposed to seawater.

Under the enhancement of service load to the corrosion, the damage process (i.e., crack initiation, steady-state crack growth, and final fracture) can occur easily. This damage is the stress corrosion cracking (SCC). Because SCC involves the mechanical action; the state of stress (i.e., plane stress and plane strain) of the engineering components influence the SCC process. The plane stress is likely to occur for thin component, where the resistance to the deformation in thickness direction is low. Thus, the stress in thickness direction is low, and only stress on the plane parallel to the applied load occurs, i.e., plane stress condition. On the other hand, for a thick component, the resistance to the deformation in thickness direction is high; thus, the

strain in thickness direction is low, and only strain on the plane parallel to the applied load occurs, i.e., plane strain condition [4]. For examples, SCC of NAB components (i.e., a ship propeller to represent thick component and a salt-water pump impeller [5] to represent thin component) are shown in Fig. 1-1. The diameter of ship propeller is approximately 4,000 mm, and has been operated under ammonia-polluted seawater (i.e., lower region of Chao Phraya river, Thailand). While the diameter of salt-water pump impeller is approximately 550 mm, and has been operated under seawater.

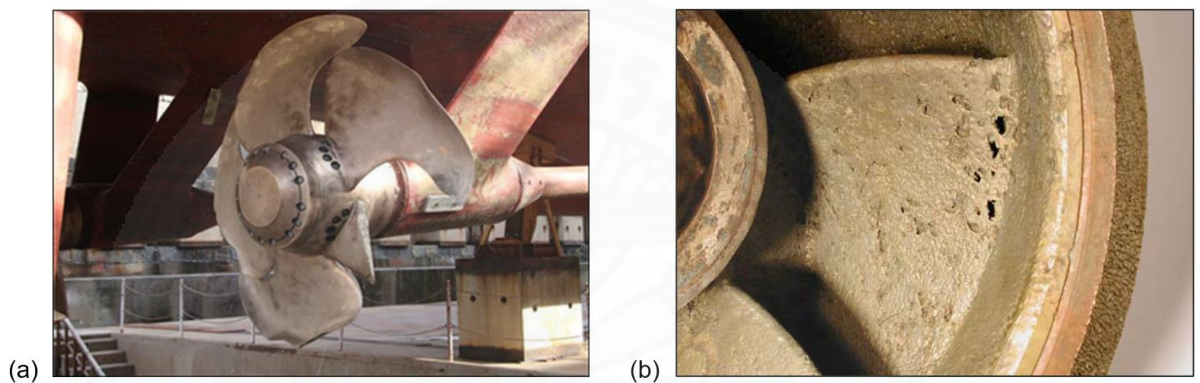


Fig. 1-1 SCC of NAB components: (a) a ship propeller to represent thick component, and (b) a salt-water pump impeller [5] to represent thin component.

1.1.1 Stress corrosion cracking under plane stress condition

It is known that engineering components may contain crack-like defects. These defects include the complex geometry of a component, as well as those arising from production and/or subsequent service [6]. Under the combination of a corrosive environment and service load, damage resulting from the growth of cracks from these defects is possible, i.e., stress corrosion cracking (SCC). If a defect of an engineering component is under the condition of small-scale yielding (i.e., plane strain condition), the stress intensity factor under opening mode (K_I) can be used as a linear-elastic fracture mechanics (LEFM) parameter and applied to characterize the severity at the crack tip. This K_I parameter is in a function of the crack size, geometry, and applied load. Above a certain level of K_I , SCC in a susceptible alloy will initiate and propagate,

whereas no measurable propagation can be observed below this level. This is defined as the threshold stress intensity factor for susceptibility to stress corrosion cracking (K_{ISCC}). Above K_{ISCC} , the crack propagation rate (da/dt) versus K_I is plotted as a SCC crack growth curve. Crack propagation rate (da/dt) increases with K_I and severity of corrosive environment. Therefore, K_{ISCC} and K_I vs da/dt are the important properties for the design, usage and improvement of engineering components under corrosive environment [7]. Although SCC tests have been successfully performed on various alloys, e.g., iron-based superalloys [8], bronzes [9], carbon steel [10], stainless steel [11], titanium alloy [12], and magnesium alloy [13, 14]; limited SCC studies, particularly determinations of K_{ISCC} using pre-cracked specimens, have been performed for NABs.

Moreover, the linear-elastic fracture mechanics (LEFM) parameter or K_{ISCC} becomes invalid if the size of the plastic zone at the crack tip is larger than the thickness, crack length, and/or remaining ligament, i.e., for the large-scale yielding condition (i.e., plane stress condition) [4]. Although many research studies of SCC have been conducted using the fracture mechanics approach, most of these studies were based on the assumption of linear-elastic fracture mechanics. For SCC of thin NAB components under corrosive environments, e.g., a vane of a salt-water pump impeller [5], it is likely that the crack-like defects are under the condition of large-scale yielding. Thus, the linear-elastic fracture mechanics (LEFM) parameter or K_{ISCC} may not be suitable for characterizing the severity at crack tips in these components. Under large-scale yielding conditions, the J -integral, or release rate of the potential energy associated with crack growth, can be used as an elastic-plastic fracture mechanics (EPFM) parameter to characterize the severity at the crack tip [15]. Accordingly, for thin NAB components under corrosive environments, a threshold J -integral for susceptibility to stress corrosion cracking (J_{ISCC}) is defined as a certain level of J -integral at which a crack in a susceptible alloy will initiate and propagate.

Because there are no published reports on the influence of the ammonia concentration in seawater on the SCC of NAB under plane stress condition; SCC experiments on NABs under laboratory air, artificial seawater, and ammonia-containing artificial seawater were conducted using a 4-point bending technique in the present work. The stress/strain state near the crack tip and the J -integral were numerically

calculated using finite element analysis (FEA). The J -integrals were correlated with the crack growth rates (da/dt) from the SCC experiments. Together with the analysis of crack propagation path and fracture surfaces, the SCC behaviour and mechanism are discussed. The finding can be applied for the prediction of plane-stress SCC of NAB.

1.1.2 Stress corrosion cracking under plane strain condition

According to ISO 7539-6 [7] and ASTM E1681 [16], SCC testing is recommended to be performed using a pre-cracked specimen under either a constant applied force or a fixed displacement. Under constant applied force, the plane-strain specimen (e.g., a compact-tension (CT) specimen) requires an external machine for the force application. On the other hand, the testing under constant applied displacement, the plane-strain specimen can be self-deformed using built-in loading bolt (e.g., a T-type wedge opening loaded (T-WOL) specimen). K_I can be calculated from force, geometry of specimen, and crack length, while da/dt can be calculated from crack length and time. Because SCC test is normally performed using a long period of time; the cost for a special loading equipment to generate a constant applied force on a pre-cracked specimen is significantly high. Therefore, to minimize the testing cost, SCC test may be performed using a T-WOL specimen, where a loading bolt is turned against a reaction pin to generate an opening of crack surfaces.

For a T-WOL specimen with built-in loading bolt, the force decreased with the extension of crack under corrosive environment. If the response force and crack-mouth opening displacement (CMOD) of a T-WOL specimen can be monitored; the crack length can be numerically calculated using finite element analysis (FEA). In the present work, the indirect crack length measurement technique using instrumented reaction pin (i.e., a support of the loading bolt) was developed to measure the crack length of a T-WOL specimen. The proper location of strain measurement in an instrumented reaction pin was determined using FEA. The instrumented reaction pin was calibrated using the tensile testing machine, and applied to measure the response force of a T-WOL specimen. Based on the three-dimensional elastic-plastic finite element analysis (3D elastic-plastic FEA) of a T-WOL specimen, the measured force as well as the measured CMOD were used as the input for the numerical calculation of

crack length. To validate this crack length measurement technique, the measured crack lengths of T-WOL specimens under ambient environment were compared with those measured using the visual inspection. This indirect crack length measurement technique is expected to be applied for the determination of crack length of T-WOL specimen during SCC experiment.

Although the corrosion resistance of alloys is enhanced from the formation of protective layer; the protective layer at crack tip can be damaged and loses its corrosion resistance under the combination between crack-tip opening and corrosive environment. Therefore, the crack-tip opening displacement (CTOD) of compact-tension specimen was successfully applied to characterize da/dt during SCC of high-strength steels with hydrogen embrittlement [17]. Without liquid environment around crack tip, CTOD can be directly measured on side surface of a specimen at 2.5-mm above and below a crack tip, i.e., CTOD δ_5 method [18]. Unfortunately, CTOD δ_5 cannot be accurately related to the damage of protective layer at crack tip because it does not represent the actual CTOD. As an alternative fracture mechanics parameter, CTOD 45° is defined as a distance between two intersections of $\pm 45^\circ$ lines from the crack tip with crack surfaces.

There are standard procedures for CTOD estimation of compact-tension specimen (e.g., ASTM E1290 [19], BS 7448-1 [20], WES 1108 [21]); however, no published report for CTOD estimation of T-WOL specimen with a built-in loading bolt is available. Moreover, the influence of side grooves (i.e., to constrain the crack-tip branching) on CTOD of T-WOL specimen is far from complete understanding. Accordingly, in the present work, CTOD δ_5 and CTOD 45° of T-WOL specimen with a built-in loading bolt was firstly calculated from the crack-opening force, load-line displacement, crack length, specimen geometry and material properties using three-dimensional elastic-plastic finite element analysis (FEA). Under ambient environment, CTOD δ_5 from experiment and FEA were compared for FEA validation. Validated FEA was used for the calculation of CTOD 45° . Influence of side grooves on CTOD 45° was evaluated and discussed. Suitable method for CTOD 45° determination was proposed. The finding can be applied for the determination of CTOD 45° of T-WOL specimen to characterize da/dt during plane-strain SCC experiment of engineering alloy.

1.2 Literature reviews

1.2.1 Nickel-aluminum bronze

Nickel aluminum bronze (NAB) has good corrosion resistance, fatigue resistance, and erosion resistance, which is considered as a suitable alloy for marine applications [22]. According to ASTM B148 [23], the chemical compositions of NABs (i.e., C95800 and C95820) are shown in Table 1-1.

Table 1-1 Chemical compositions of NABs [23].

NAB	Chemical composition (wt.%)				
	Ni	Al	Fe	Mn	Cu
C95800	4.0-5.0	8.5-9.5	3.5-4.5	0.8-1.5	79.0 min
C95820*	4.5-5.8	9.0-10.0	4.0-5.0	1.5 max	77.5 min

* Zinc shall be 0.2 max and tin 0.02 max.

Quaternary phase diagram of the Cu-Al-Ni-Fe system [24, 25] is shown in Fig.1-2. During solidification of a melted NAB with 9 wt.% of Al (i.e., a blue line of Fig. 1-2), a copper solid solution (i.e., β phase) is formed. Within the β phase, the other alloyings are dissolved. The cooling below 940 °C decomposes the β -phase into α -phase and κ -phase. Between 940 and 800 °C, a ternary phase (i.e., α , β , and κ phases) exists. While, below 800 °C, only α and κ phases are forms. The κ phases are the compositions between Fe_3Al and NiAl [26]. During quenching from 1,000 °C to room temperature, the formation of a martensitic phase (i.e., β' phase) occurs. However, after a subsequent heat treatment, the β' phase decomposes into fine-grained α and κ phases (Fig. 1-3).

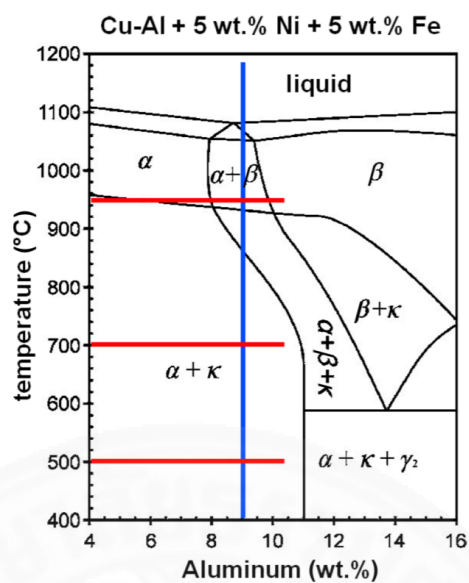


Fig. 1-2 Quaternary phase diagram of the Cu-Al-Ni-Fe system [24, 25].

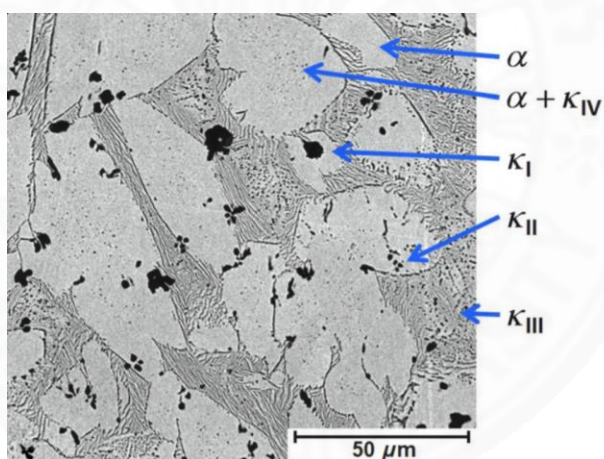


Fig. 1-3 As-cast NAB microstructure [25].

According to ASTM B148 [23], the mechanical properties of as-cast NABs (i.e., C95800 and C95820) should meet the requirements, as shown in Table 1-2.

Table 1-2 Mechanical properties of NABs [23].

NAB	Mechanical properties, min		
	Tensile strength (MPa)	Yield strength (MPa)	Elongation in 2 in. (%)
C95800	585	240	15
C95820*	650	270	13

1.2.2 Corrosion and stress corrosion cracking of nickel-aluminum bronze

Stress corrosion cracking (SCC) is interpreted to take place due to specific combinations of susceptible materials in suitable corrosion environments under adequate stress, specifically under constant stress (Fig. 1-4). However, the equivalent mechanism for crack growth under cyclic stresses is called corrosion fatigue, and crack growth here depends upon the stress ratio. In particular, when the stress ratio is high, CF becomes close to SCC; this type of loading is called ripple loading, which often simulates very well the loading situation during plant operations. Strain-induced corrosion cracking is used especially in Germany as a term for the situation, where cracking takes place under increasing load, which may not necessarily increase monotonically, but also some cyclic variations may be present as typically in start-up and shut-down situations of plants. Therefore, the last mechanism can be considered to be a special case of corrosion fatigue, when only a very small number of cycles are present. All these mechanisms can be described by a general term, namely, environment-assisted cracking (EAC).

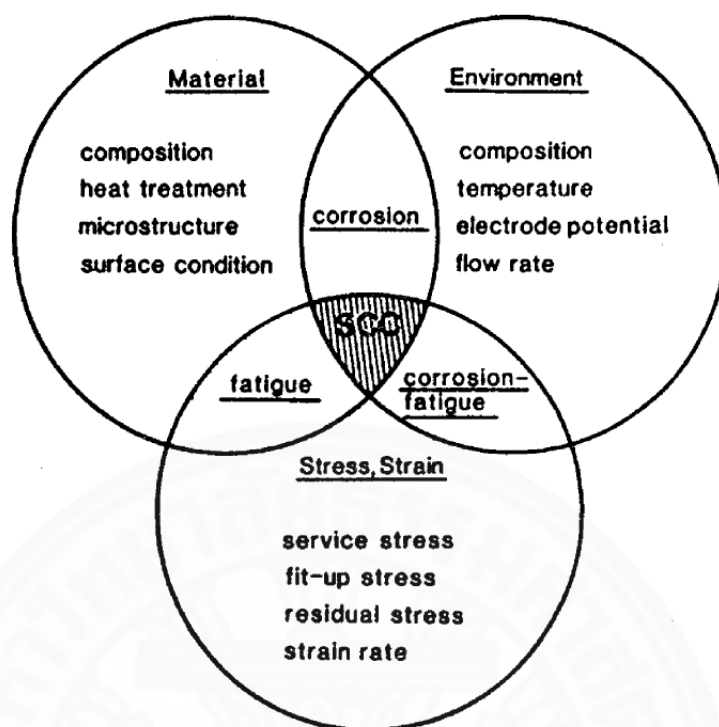


Fig. 1-4 Specific conditions for stress corrosion cracking [27].

SCC describes service failures in engineering materials that occur by slow time-dependent environmentally assisted crack growth (e.g., 10^{-6} m/s). The crack growth is a result of the combined and synergistic interaction of mechanical stress and corrosion reactions. In general, the stresses required to cause SCC are small, often below the macroscopic yield stress and they are tensile in nature, either external applied or residual fabrication stresses. Crack growth occurs perpendicular to the principal tensile stress direction. Crack branching is generally thought to be a positive indication of SCC. Compressive stresses can be imposed to prevent SCC. Static loading is usually considered to be responsible for SCC, but the corrosion fatigue and the strain-induced corrosion cracking are often considered to be subsets of SCC.

Stress corrosion cracks initiate at surface flaws that either are pre-existing or are formed during service by corrosion. The cracks grow with very little macroscopic evidence of mechanical deformation in metals and alloys that are normally very ductile. Crack growth can be either transgranular or intergranular, and sometimes mixed cracking can occur. Typically, fracture surfaces look very brittle and close

matching of the fracture surfaces is commonly observed, indicating minor loss of material by corrosion. SCC is mainly used to describe failures in metallic alloys. However, ceramics also exhibit environmentally assisted crack propagation and polymers exhibit craze cracking due to interaction of applied stress and environmental reactions [28]. The present review was focused on SCC of metallic materials and was not be concerned with ceramics and polymers.

The environments that cause SCC are usually aqueous solutions, but they can also be condensed as layers of moisture, and even water of crystallization of salts can induce SCC. Typically, SCC of a metal alloy is a result of the presence of some chemical species in addition to water in the environment [29]. For example, SCC of austenitic stainless steels and aluminum alloys is most commonly caused by chloride ions, and ammonia is the most common cause of SCC in copper alloys, traditionally referred to as season cracking. Variation in temperature, degree of aeration (corrosion potential), and concentration of ionic species may change an innocuous environment to cause SCC. Metallurgical conditions of alloys may also affect markedly SCC susceptibility.

In general, SCC is observed in alloy-environment combinations that result in the formation of a film on the metal surface, which reduces the rate of general and uniform corrosion [28]. However, materials like austenitic stainless steels and copper alloys also show cracking in active corrosion conditions [30], which naturally is not their normal operating condition in practice. These films may be passive, salt and tarnish films, or dealloyed layers. Thus, SCC is of greatest concern with the corrosion-resistant alloys in aggressive aqueous environments. The sequence of events involved in SCC is usually divided into three stages: (1) crack initiation, (2) steady-state crack growth, and (3) final failure. Distinction between these stages is often difficult because the transition occurs in a continuous manner. For example, the crack initiation stage has been considered to change to the crack growth phase when the size of the crack is 20-50 mm, i.e., about one grain diameter and greater [31]. The critical conditions for a SCC crack to grow are the environmental, mechanical, and electrochemical conditions of the incipient crack. However, in the engineering sense, the initiation takes place when

the cracks can be detected by normal nondestructive methods, i.e., crack size is greater than 1-10 mm, dependent on material and component geometry.

When SCC cracks start to grow from pre-existing flaws or localized corrosion sites, all the mechanisms of crack growth are localized at the crack tips, and it is not necessary for the entire component to become embrittled. There are a number of possible mechanisms that can describe SCC in a large variety of materials. The general mechanisms of EAC of materials are shown in Fig. 1-5. For EAC, either the slip-dissolution or hydrogen induced cracking mechanisms have been favored as reasonable working hypotheses. Under high-strain-rate conditions, when the crack tip is continuously maintained in a bare surface condition, the crack growth can be identified atomistically with the bare surface reaction rates for either dissolution or hydrogen evolution reaction. Under normal strain rate conditions, however, the crack tip surface is not maintained continuously in a bare condition. The crack growth is controlled by the oxide rupture rate, the repassivation rate, and the liquid diffusion rate at or near the crack tip [32].

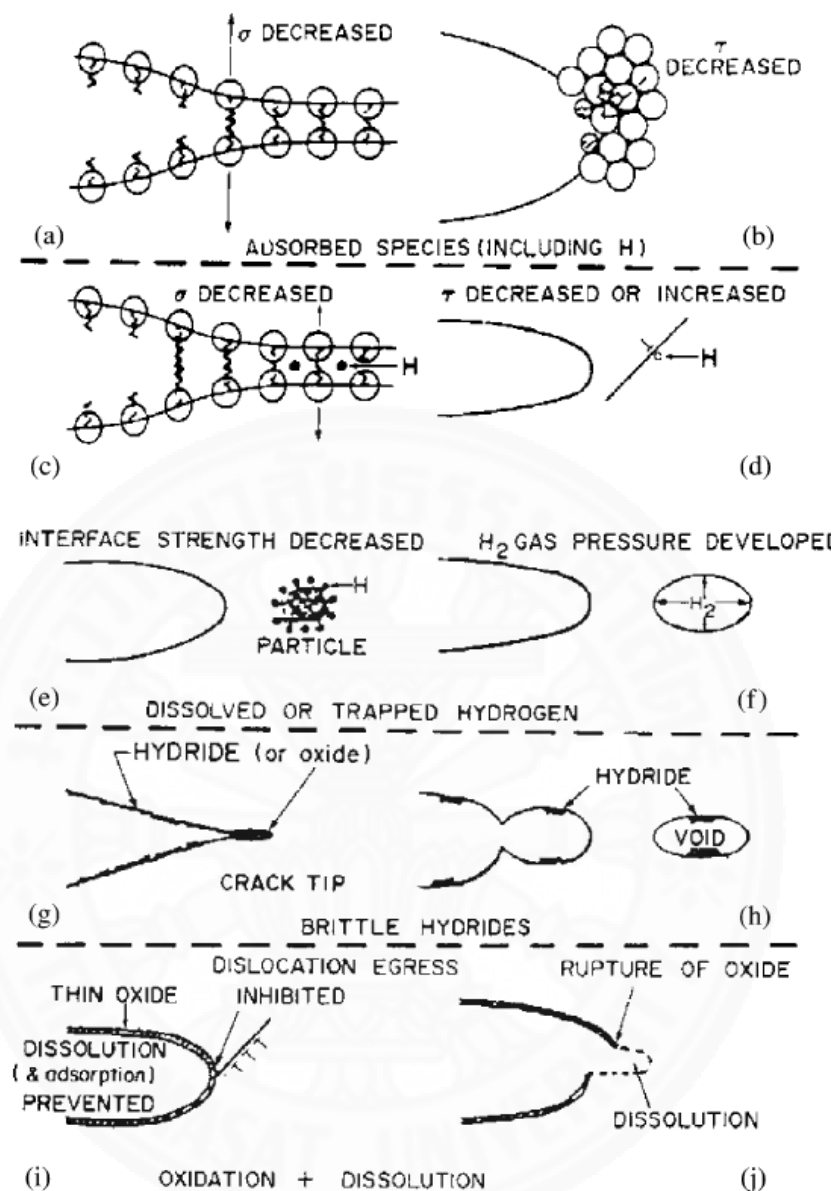


Fig. 1-5 Schematic presentation of possible effects of environment on fracture [33].

Adsorption of surface-active species other than hydrogen has been proposed to reduce the fracture stress in various embrittlement phenomena. In adsorption-related mechanisms, the influence of adsorption is limited to one or two atomic distances from the surface of the metal. Adsorption can thus only influence the tensile strength and the shear strength of the interatomic bonds at the crack tip (Fig. 1-6). A crack can grow rapidly by cleavage or slowly by slip, depending on whether

the tensile fracture stress for the interatomic bond or the shear stress to cause slip in a favorable slip system is achieved first. Hence, as the ratio of shear strength/tensile strength decreases, cleavage becomes more probable and, conversely, shear failure becomes more likely as the ratio increases [34].

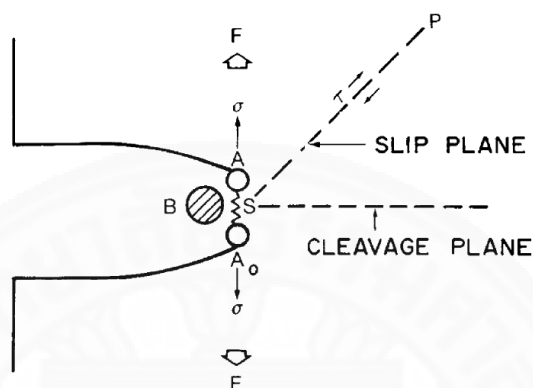


Fig. 1-6 Environment/metal interaction model [34].

For copper alloys, SCC occurs typically in moist air, where a condensed moisture layer collects ammonia and oxygen on the copper surface. In the condensed film, a high concentration of cupric complex ions forms rapidly. Tarnish film formation starts quickly and has been found to consist of Cu_2O . Most research on SCC of copper alloys is conducted in these conditions. Other environments, such as sulfates, nitrates, nitrites, chloride containing species, and even pure water, have also been identified as causing SCC of copper alloys. In addition to brass, other copper alloys including pure copper (in nitrite and acetate solutions) are also susceptible to SCC. Environmental conditions causing SCC in copper alloys have been reviewed in detail [35, 36]. For a brass exposed to various non-ammoniacal environments, cracking tends to occur where Cu_2O film is likely to form, thus revealing the importance of Cu_2O films in the SCC mechanism of copper alloys.

Temperature dependence of SCC in copper alloys appears to follow an Arrhenius type behaviour (i.e., the temperature dependence of reaction rates). It has been demonstrated that pure copper displays transgranular SCC in nitrite solutions in the same potential range as for copper alloys. In brasses, dealloying is involved in

transgranular SCC, but the cracking produced by the environments that cause the Cu_2O films is intergranular. Therefore, the mechanism of copper alloys must include both the effects of Cu_2O film and dealloying. Dealloying is not possible in pure copper and therefore rupture of a dealloyed layer does not explain transgranular cracking of pure copper [29]. The mechanism of intergranular SCC in copper alloys is considered to occur by a slip-dissolution/film rupture mechanism. Transgranular SCC, which occurs discontinuously and has a brittle cleavage-like appearance of the fracture surfaces, however, is thought to take place by discontinuous cleavage possibly triggered by an epitaxial film such as oxide or dealloyed layer at the crack tip.

Wharton et al. [37] evaluated the corrosion behaviours of cast and wrought NABs in seawater using short-term electrochemical techniques and longer-term immersion trials. They discovered that the corrosion rate depends on the surface films, composition of the seawater electrolyte, metallurgical composition, surface roughness, electrode/electrolyte geometry, electrolyte flow, erodent, and immersion time. Al-Hashem and Riad [38] investigated the effect of the NAB microstructure on the cavitation corrosion behaviour in seawater using a 20-kHz ultrasonic-induced cavitation system. They determined that the corrosion in quiescent seawater occurs at the interfaces between the α phase and intermetallic κ precipitates, whereas the κ precipitates and precipitate-free zones do not experience corrosion. They observed microcracks (5-10 μm in length) in the α phase adjacent to κ precipitates. They suggested that the selective phase corrosion and cavitation stresses could lead to cracking. Schüssler and Exner [39] studied the electrochemical corrosion of a cast NAB in synthetic seawater with and without sulphide pollution. They found that the protective layer primarily consists of aluminium oxide, which serves as a barrier for ionic transport across the protective layer. They proposed that the loss of passivity in sulphide-containing seawater due to the formation of copper sulphide in the corrosion layer could markedly accelerate the charge transfer kinetics of a high corrosion rate resulting from sulphide exposure. Koul and Gaies [9] performed SCC experiments on heat-treated NAB (C63200) under artificial seawater and ammonia-containing artificial seawater. They found that SCC is possible for NAB under ammonia-containing artificial seawater, i.e., $K_{\text{ISCC}} = 15 \text{ MPa}\cdot\text{m}^{1/2}$; however, SCC was not detected for NAB under

artificial seawater. Neodo et al. [40] investigated the corrosion properties of NAB in aqueous chloride media using linear potential sweep voltammetry and scanning electron microscopy. They found that NAB electrochemical behaviour depends on the solution pH. At solution pHs higher than 4.0, the NAB oxidation was driven by the dissolution of the copper-rich α -phase. While, at pHs lower than 4.0, its anodic behaviour was controlled by the oxidation of the iron-, nickel- and aluminium-rich κ_I -, κ_{II} - and κ_{IV} -phases. Fonlupt et al. [41] investigated the role of second phases in SCC (i.e., slow strain rate tensile tests) of a complex copper-aluminium alloy $\text{CuAl}_9\text{Ni}_3\text{Fe}_2$ in artificial seawater. They found that the cracking is mainly intergranular, especially at interface between the α matrix and the second phases.

Although there are many works related to the corrosion and SCC of Cu alloys; only a small number of works directly related to the corrosion and SCC of NAB (e.g. ref. [37-41]). Most of these works emphasized the SCC mechanism of NAB, while only Koul and Gaies [9] were applied fracture mechanics approach to characterize the SCC of NAB. Thus, the understanding of SCC of NAB is far from complete, and further investigation should be performed.

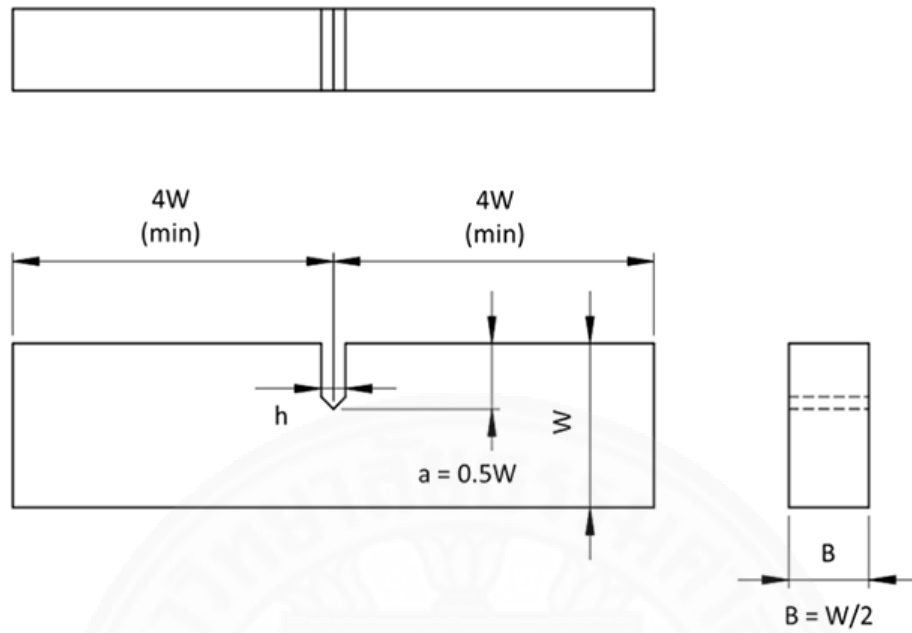
1.2.3 Stress corrosion cracking experiment

To understand the phenomena of SCC, the SCC experiment is recommended by ISO 7539-6 [7] and ASTM E1681 [16]. SCC experiment involves precracked specimen (e.g., single-edge notch bending specimen or SENB specimen, compact-tension specimen or CT specimen, T-type wedge opening loaded specimen or T-WOL specimen). The naturally-sharp precracking of specimen is performed via fatigue test. SENB specimen and CT specimen (Fig. 1-7) are tested under a constant force, i.e., they require an external machine for the force application. While T-WOL specimen (Fig. 1-8) is tested under a constant deformation using self-deformed mechanism. An environmental chamber is either attached to the crack tip of specimen, or the specimen is contained within the chamber. The prescribed environmental conditions should be maintained within the chamber during the test. K_I can be calculated from force, geometry of specimen, and crack length, while da/dt can be

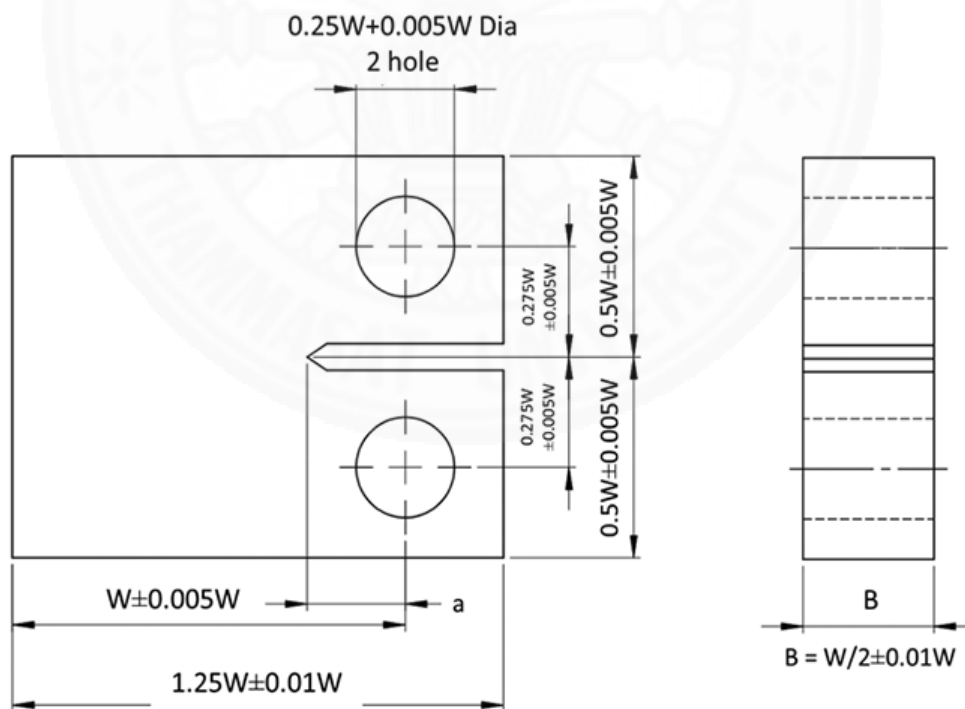
calculated from crack length and time. As a SCC crack growth curve, da/dt versus K_I is plotted, and K_{ISCC} can be determined.

Because SCC test is normally performed using a long period of time; the cost for a special loading equipment to generate a constant applied force on a pre-cracked specimen is significantly high. Therefore, to minimize the testing cost, SCC test may be performed using a T-WOL specimen, where a loading bolt is turned against a reaction pin to generate an opening of crack surfaces.





(a)



(b)

Fig. 1-7 Geometries of: (a) SENB specimen, and (b) CT specimen [16].

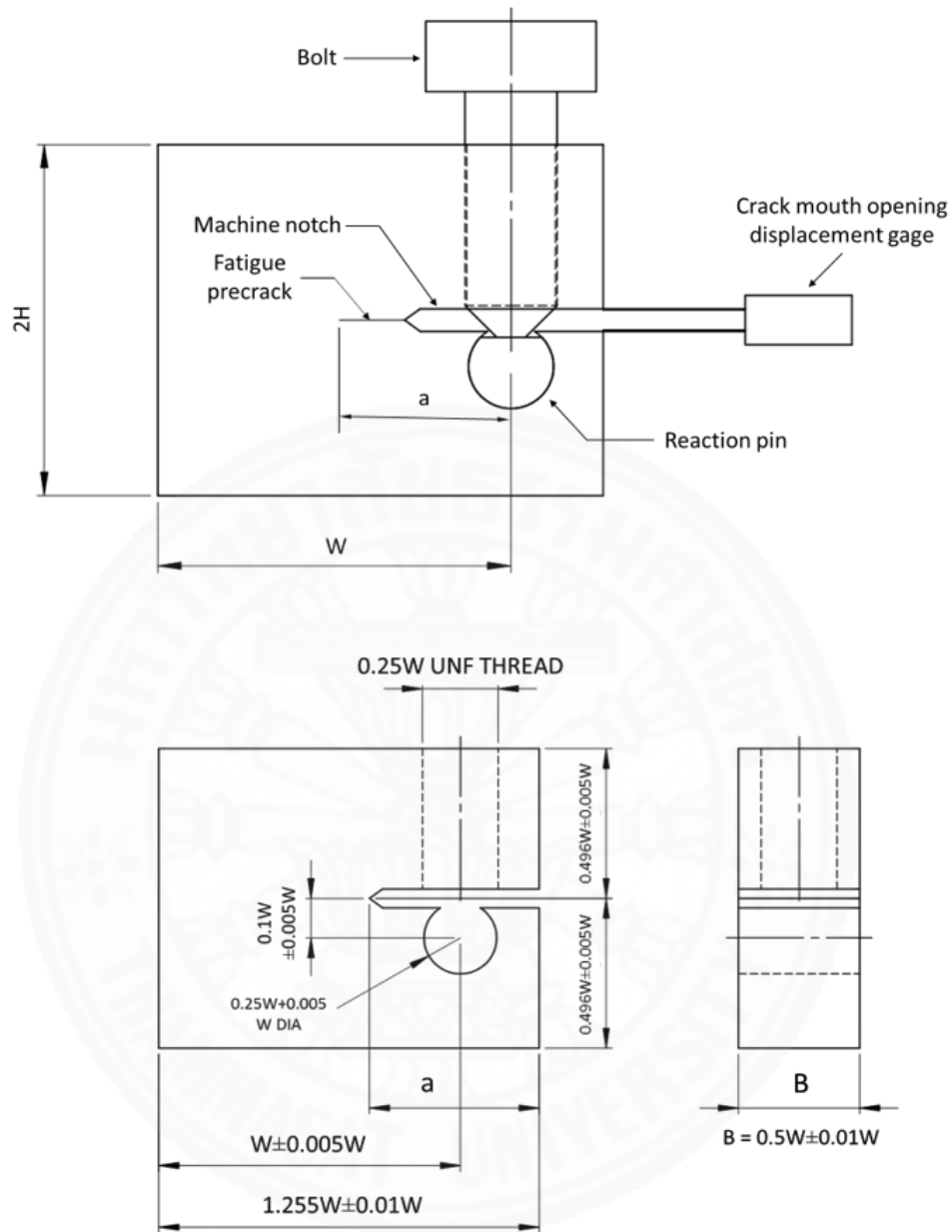


Fig. 1-8 Geometry of T-WOL specimen [16].

Although the geometry of T-WOL specimen recommended by ISO 7539-6 [7] and ASTM E1681 [16] are similar; the calculations of K_I are different. According to ISO 7539-6 [7], K_I is calculated from crack-mouth opening displacement (ν), i.e.,

$$K_I = (F / W a^{1/2}) f(a/W) \quad (1-1)$$

$$f(a/W) = [30.96(a/W) - 195.8(a/W)^2 + 730.6(a/W)^3 - 1186.3(a/W)^4 + 754.6(a/W)^5] \quad (1-2)$$

where, F is the applied force, W is the width of specimen, a is the crack length.

On the other hand, according to ASTM E1681 [16], K_I is calculated from crack-mouth opening displacement (δ), i.e.,

$$K_I = (\sqrt{VE/W^{1/2}})f(a/W) \quad (1-3)$$

$$f(a/W) = [1 - a/W]^{1/2}[0.654 - 1.88(a/W) + 2.66(a/W)^2 - 1.233(a/W)^3] \quad (1-4)$$

where, E is the modulus of elasticity.

It is important to underline that (i) the procedures for J -integral and CTOD have never been described in ISO 7539-6 [7] and ASTM E1681 [16], and (ii) the crack length measurement influences the accuracy of K_I and da/dt of SCC. In the previous works, various techniques, that have been successfully employed to monitor the crack length, can be the visual inspection technique [42], replica technique [43], electrical potential technique [44], acoustic emission technique [45], and compliance technique [46]. However, during the SCC test under liquid corrosive environment, the corrosion product can be presented at the crack tip, which is hard to directly monitor the subcritical crack growth using visual measurement and replica. The other indirect techniques, such as electrical potential technique, acoustic emission technique and compliance technique using back-surface strain, are difficult to performed under liquid corrosive environment. Moreover, the compliance technique required a force transducer, which is not available for a T-WOL specimen.

For a T-WOL specimen with built-in loading bolt, the force decreased with the extension of crack under corrosive environment. If the response force and crack-mouth opening displacement (CMOD) of a T-WOL specimen can be monitored; the crack length can be numerically calculated using finite element analysis (FEA). A force-measuring device for a T-WOL specimen (i.e., converting the strain of loading bolt to response force) was developed by Chung et.al. [47] and Vigilante et.al. [48]. The difference between these works is that Chung et.al. [47] used the strain on the longitudinal axis of loading bolt, while Vigilante et.al. [48] used the surface strain of

loading bolt. Based on the elastic compliance of specimen, they applied the measured force and measured CMOD to numerically estimate the crack length (i.e., 2D linear-elastic FEA performed by Newman, Jr. [49]). Unfortunately, the use of a loading bolt to measure the response force has some limitations, e.g., (i) the accuracy depends on the alignment and contact between the loading bolt and reaction pin, (ii) turning the loading bolt during SCC testing can affect the force measurement, and (iii) the elastic compliance is inaccurate for T-WOL specimen with long crack under large deformation (i.e., the plastic deformation at crack tip as well as the bending of T-WOL specimen can become the significant factors).

Because the protective layer at crack tip can be damaged and loses its corrosion resistance under the combination between crack-tip opening and corrosive environment; the CTOD of T-WOL specimen should be applied to characterize da/dt during SCC of NAB. As an alternative fracture mechanics parameter, CTOD 45° is defined as a distance between two intersections of $\pm 45^\circ$ lines from the crack tip with crack surfaces. In the previous works, CTOD 45° was numerically calculated from the force, load-line displacement, crack length, specimen geometry and material properties, and used for the determination of fracture toughness, i.e., ASTM E1290 [19], BS 7448-1 [20], WES 1108 [21] (hereinafter denoted as CTOD_{ASTM}, CTOD_{BS}, CTOD_{WES}, respectively). Because the estimation of CTOD_{ASTM} is based on J-integral method, while those of CTOD_{BS} and CTOD_{WES} are based on plastic hinge rotation method; CTOD_{ASTM} is smaller than CTOD_{BS} and CTOD_{WES}, especially for steels with low yield-to-tensile ratio (σ_y/σ_{ut}) [50]. Moreover, the estimation of CTOD also depends on the geometry of specimen, i.e., the calculation methods of CTOD using three-point bend, compact-tension, and arc-shaped bend specimens are not similar [19].

1.3 Objective

Stress corrosion cracking under plane stress condition

- To evaluate SCC behaviour and mechanism of NAB under plane stress condition using SENB specimen and 4PBT jig.
- To numerically calculate and apply J -integral (i.e., a fracture mechanics parameter for a large-scale yielding at crack tip) to characterize the crack growth rate of NAB.
- To give some guidelines for practitioners to avoid SCC and predict the crack growth rate (da/dt) of NAB under plane stress condition.

Stress corrosion cracking under plane strain condition

- To design, produce, and validate T-WOL specimen with an instrumented reaction pin (i.e., to measure crack-opening force) for SCC experiment under plane strain condition.
- To numerically calculate and validate the indirect crack length measurement (i.e., FEA using measured crack-opening force and measure CMOD) of T-WOL specimen.
- To numerically calculate and validate CTOD (i.e., a fracture mechanics parameter to characterize the damage of protective layer at crack tip) of T-WOL specimen, and investigate the influence of side grooves.
- To give some guidelines for practitioners to perform SCC experiment under plane strain condition using T-WOL specimen.

1.4 Scope

- Materials: NAB grade C95800 (i.e., ASTM B148 [23])
- Corrosive environment: artificial seawater, and ammonia-containing artificial seawater
- SCC experiment: as recommended by ISO 7539-6 [7] and ASTM E1681 [16]
- Engineering design: 4PBT jig for SENB specimen and instrumented reaction pin for T-WOL specimen
- Fracture mechanics parameter: K , J -integral, CTOD
- Metallurgical analysis: corrosion product, crack path and fracture surface
- Finite element analysis: commercial three-dimension finite element analysis software (i.e., ABAQUS [51])



Chapter 2

Stress corrosion cracking under plane stress condition: research procedure

Plane-stress SCC experiments on NABs under laboratory air, artificial seawater, and ammonia-containing artificial seawater were conducted using a 4-point bending technique in the present work. The stress/strain state near the crack tip and the J -integral were numerically calculated using finite element analysis (FEA). The J -integrals were correlated with the crack growth rates (da/dt) from the SCC experiments. Together with the analysis of crack propagation path and fracture surfaces, the SCC behaviour and mechanism are discussed. Details of the research procedures are described as follows.

2.1 Nickel-aluminum bronze

The material used in this study was a cast nickel aluminium bronze (NAB) alloy, which was supplied by the Royal Thai Navy. This NAB was sectioned from an intact blade of ship propeller. Its chemical composition was evaluated using an optical emission spectrometer (Thermo Scientific: ARL 3460). The result is summarized in Table 2-1, which corresponds to that of the ASM handbook [52]. Following ASTM recommendations, the tensile properties [53] and hardness [54] were performed using a universal testing machine (Instron: 5969), and a hardness machine (Nakai Precision: NBH-3), respectively. The stress-strain curve of NAB is shown in Fig. 2-1, while the mechanical properties are summarized in Table 2-2. The fracture toughness of NAB [55] is also listed in Table 2-2. Ramberg-Osgood relationship was applied to represent the elastic-plastic behaviour of NAB, as follows,

$$\varepsilon = \frac{\sigma}{E} + \alpha \frac{\sigma}{E} \left(\frac{\sigma}{\sigma_Y} \right)^{n-1}, \quad (2-1)$$

where, E is the modulus of elasticity, σ_Y is the yield stress, α and n are the hardening coefficient and hardening exponent, respectively. The hardening coefficient and hardening exponent are 1.1215 and 4.6, respectively.

Table 2-1 Chemical composition of NAB.

NAB	Chemical composition (wt.%)				
	Ni	Al	Fe	Mn	Cu
Present alloy	4.3	9.5	4.6	1.1	80.5
C95800*	4.0	9.0	4.0	1.5	81.5

*ASM handbook [52]

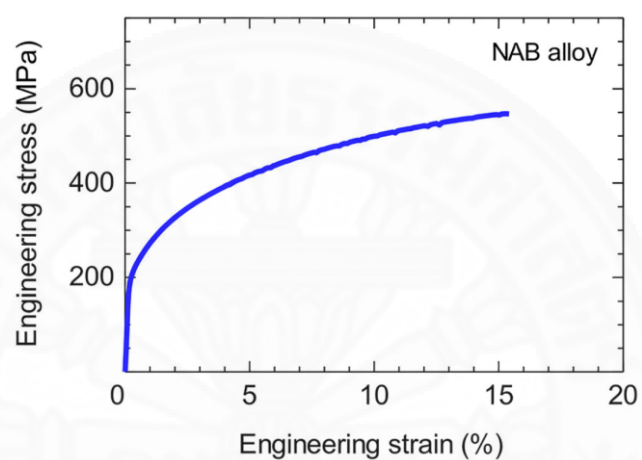


Fig. 2-1 Stress-strain curve of NAB.

Table 2-2 Tensile and hardness properties of NAB.

Tensile strength (MPa)	547
Yield strength at 0.2% strain offset (MPa)	214
Modulus of elasticity (GPa)	120
Elongation of 10-mm gage length (%)	30
Hardness (HB)	146
Fracture toughness (K_{IC} , MPa.m ^{1/2}) [55]	25

NAB was polished to a 1- μm diamond finish and etched with a mixture containing 1 g of FeCl_3 , 10 mL of HCl , and 100 mL of water. The etched NAB was examined using reflected light microscopy to observe the microstructure. The microstructure of NAB is shown in Fig. 2-2. Energy-dispersive X-ray spectroscopy (EDS) analysis shows five distinct phases, and the approximate compositions of these phases are shown in Table 2-3. It is noted that the EDS analysis of elemental composition has limited accuracy; therefore, the EDS technique was applied here for comparative analysis. The microstructural constituents can be described as follows: (i) α -phase: FCC copper-rich solid solution; (ii) κ_I -phase: globular or rosette shape of iron-rich (Fe_3Al); (iii) κ_{II} -phase: dendritic rosettes unevenly distributed within the α -phase; (iv) κ_{III} -phase: lamellae of nickel-rich (NiAl), which grows normal to the α -phase and around the κ_I -phase; and (v) κ_{IV} -phase: fine iron-rich precipitates within the α -phase [56].

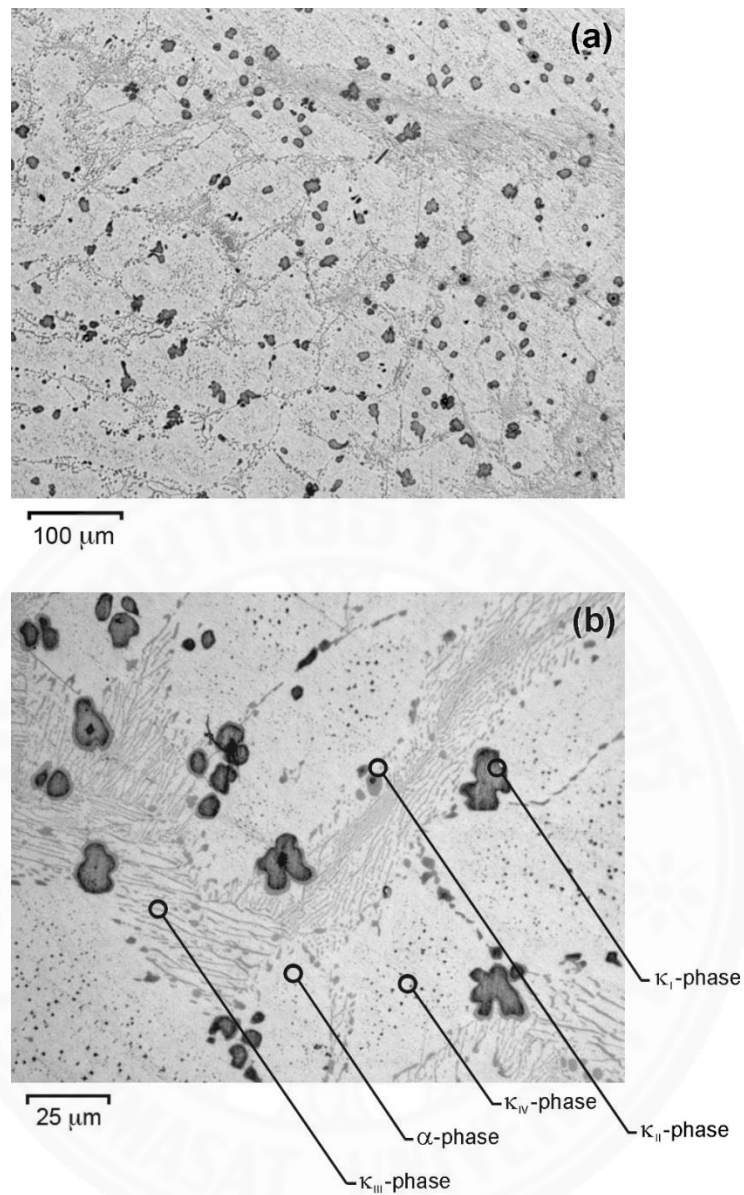


Fig. 2-2 Micrographs of NAB at (a) low magnification and (b) high magnification.

Table 2-3 Phase analysis of NAB.

Phase	Composition (wt. %)				
	Ni	Al	Fe	Mn	Cu
α	2.2 ± 0.2	6.0 ± 0.2	1.6 ± 0.1	0.9 ± 0.1	77.3 ± 0.6
κ_I	12.4 ± 0.3	11.0 ± 0.2	54.8 ± 0.4	1.8 ± 0.1	11.3 ± 0.3
κ_{II}	26.4 ± 0.4	17.3 ± 0.3	19.6 ± 0.3	1.9 ± 0.1	25.5 ± 0.4
κ_{III}	27.4 ± 0.3	22.8 ± 0.3	11.3 ± 0.2	1.4 ± 0.1	25.7 ± 0.4
κ_{IV}^*	4.0 ± 1.0	20.0 ± 3.0	62.0 ± 4.0	1.5 ± 0.3	13.0 ± 1.0

*Culpan and Rose [56]

2.2 Corrosive environment

Various environments were applied for the present SCC experiments, i.e., laboratory air, artificial seawater, and ammonia-containing artificial seawater. A 1-L glass container was used during the SCC experiments. The artificial seawater was prepared in accordance with the ASTM D1141 standard [57], and it contains inorganic salts (e.g., $MgCl_2$, $CaCl_2$, $SrCl_2$, KCl , $NaHCO_3$, and KBr) in proportions and concentrations that are representative of ocean water. The chemical composition of the 1 L of artificial seawater is shown in Table 2-4. The pH was adjusted to 8.2 using a 0.1 N sodium hydroxide (NaOH, 0.10 N) solution. This pH adjustment was performed immediately prior to use. It is noted that adding NaOH can adjusted the pH of artificial seawater for a short period of time. However, adding NaOH is not possible to adjust the pH to 8.2 throughout the SCC experiment because the artificial seawater evaporates, and its pH level significantly decreases. Accordingly, old artificial seawater is completely replaced by fresh artificial seawater every 5 to 7 day.

The ammonia-containing artificial seawater environments were mixtures of artificial seawater and ammonium hydroxide (SW:AH) in volume ratios of 10:1 and 5:1. The ammonium hydroxide was 25% NH_3 in H_2O . The mixtures were prepared immediately prior to use. The chemical compositions of the 1-L mixtures of artificial seawater and ammonium hydroxide in volume ratios of 10:1 and 5:1 are shown in Table 2-4. The pH of the mixtures of artificial seawater and ammonium hydroxide in

volume ratios of 10:1 and 5:1 are 9.5 and 10.5, respectively. These SW:AH volume ratios were selected to represent the moderate corrosiveness environment (pH of 9.5 to 10) and high corrosiveness environment (pH of 10 to 10.5) recommended by the ASTM B858 standard [58]. The liquid environments were circulated using a magnetic stirrer, and their temperatures were approximately 28 ± 1 °C during the SCC experiments.

Table 2-4 Chemical compositions of 1-L corrosive environments.

Composition	Corrosive environment		
	SW	10:1 / SW:AH	5:1 / SW:AH
NaCl (g/L)	24.534	22.304	20.445
Na ₂ SO ₄ (g/L)	4.094	3.722	3.412
MgCl ₂ .6H ₂ O (g/L)	11.111	10.101	9.259
CaCl ₂ (g/L)	1.159	1.054	0.966
SrCl ₂ .6H ₂ O (g/L)	0.042	0.038	0.035
KCl (g/L)	0.695	0.631	0.579
NaHCO ₃ (g/L)	0.201	0.183	0.167
KBr (g/L)	0.101	0.091	0.084
H ₃ BO ₃ (g/L)	0.027	0.025	0.023
NaF (g/L)	0.0030	0.0027	0.0025
ammonia hydroxide (mL)	-	90.9	166.7
pH	8.2	9.5	10.5

Note: These salts were diluted in deionized water to obtain a total volume of 1 L.

2.3 Stress corrosion cracking evaluation under plane stress condition

In the present section, SCC experiments on NABs under laboratory air, artificial seawater, and ammonia-containing artificial seawater were conducted using a 4-point bending technique. The stress/strain state near the crack tip and the J -integral were numerically calculated using finite element analysis (FEA). The J -integrals were correlated with the crack growth rates (da/dt) from the SCC experiments. Together with

the analysis of crack propagation path and fracture surfaces, the SCC behaviour and mechanism are discussed.

2.3.1 Single edge notched bending specimen

Single-edge notch bending (SENB) specimens were machined from NAB, and were used in the present SCC experiments. The geometry of the SENB specimen was in accordance with the ISO 7539-6 standard [7], as shown in Fig. 2-3. The use of SENB specimen has the advantage that it is possible to load the specimen to high stress levels due to its small cross-section and the application of a bending moment. The SENB specimens were machined using an electrical discharge machine (EDM) before pre-cracking. Because the specimen with pre-crack is used for the SCC experiment, the residual stress from EDM process can be neglected. Therefore, the annealing was not applied to specimen after EDM process. However, the EDM product on the surface of the SENB specimens was removed using abrasive paper, and then the SENB specimens were ultrasonically cleaned in acetone. The pre-crack was created by 4-point bending fatigue (Instron: ElectroPuls E10000) under an air environment, $R = 0.1$ stress ratio, and 20-Hz frequency. The pre-crack is a sharp crack with clean crack surfaces, which can be used for the SCC experiment.

The stress intensity factor (K) at the crack tip of the SENB specimen can be determined as follows [59];

$$K = F(\alpha) \frac{3P(S-d)}{2BW^2} \sqrt{\pi a}, \quad (2-2)$$

$$F(\alpha) = 1.122 - 1.121\alpha + 3.740\alpha^2 + 3.873\alpha^3 - 19.05\alpha^4 + 22.55\alpha^5, \quad (2-3)$$

$$\alpha = \frac{a}{W}, \quad (2-4)$$

where W is the width of the specimen, a is the average length of cracks on both sides of the specimen ($0.45 \leq a/W \leq 0.55$), P is the applied load, B is the thickness of the specimen, S is the lower span of 4-point bending, and d is the upper span of 4-point bending.

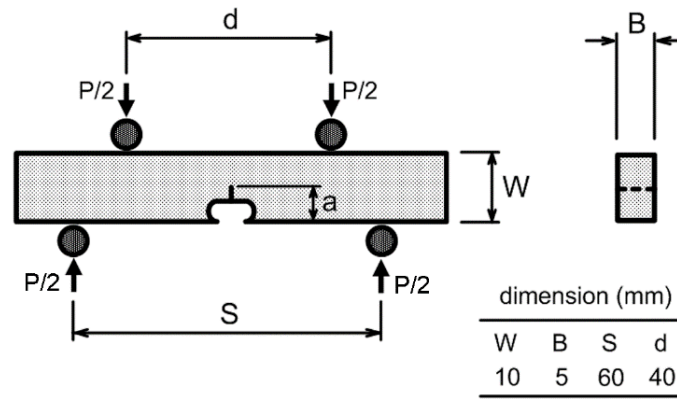


Fig. 2-3 Schematic illustration of a single-edge notch bending specimen.

Because the pre-cracking process is performed under cyclic loading, the plastic zone at maximum load is compressed by the elastic deformation during unloading. Therefore, the cyclic-plastic zone (r_c) is smaller than the monotonic-plastic zone. During the pre-cracking process, the size of the cyclic plastic zone (r_c) at the crack tip of the SENB specimen was maintained to be smaller than its thickness, crack length, and remaining ligament. The r_c can be estimated as follows [6]:

$$r_c(\theta) = \frac{\Delta K^2}{16\pi\sigma_Y^2} \left(\frac{3}{2} \sin^2 \theta + (1 - 2\nu)^2 (1 + \cos \theta) \right), \quad (2-5)$$

where ΔK is the stress intensity factor range, σ_Y is the yield stress, θ is the polar coordinate at the crack tip, and ν is Poisson's ratio ($\nu = 0.3$). In the present work, the pre-cracking process was performed with $\Delta K_f = 11 \text{ MPa}\cdot\text{m}^{1/2}$; therefore, the maximum r_c at $\theta = \pi/2$ was $87.3 \text{ }\mu\text{m}$. This cyclic plastic zone size is significantly smaller than the thickness, crack length, and remaining ligament of the SENB specimen; thus, it is confirmed that the crack tip is under the condition of small-scale yielding during the pre-cracking process.

2.3.2 Stress corrosion cracking test

The SCC experiment was conducted using a 4-point bending technique (4PBT), as shown in Fig. 2-4. This SCC experiment was performed in accordance with the ISO 7539-6 standard [7]. The 4PBT jig was composed of AISI 304 stainless steel. During the SCC experiment, the 4PBT jig (AISI 304 stainless steel) was exposed to the test environment, and was in contact with the NAB specimen. The contact between these alloys, which have different potentials in seawater, induces a flow of electrons from the electronegative alloy to the electropositive alloy, i.e., galvanic corrosion and crevice corrosion of the electronegative alloy are possible at the contact. According to the galvanic series in seawater [60], the potentials of AISI 304 stainless steel and NAB range from -30 to -90 mV and -140 to -210 mV, respectively; therefore, the NAB is the electronegative alloy. However, the difference in potentials is marginal, and the alloys were exposed to the test environments for short periods of time (less than 350 hours). Accordingly, the galvanic corrosion and crevice corrosion on the NAB specimen are insignificant, and the amount of corrosion product at the contacts between NAB specimen and 4PBT jig is not enough to be visually detected after the SCC experiment.

Deflection of the SENB specimen was achieved by moving a bolt. The load (P) on the SENB specimen was measured using full-bridge strain gauges (TML: FLA-03-11), which were calibrated with a 1-kN load cell (Kyowa: LMA-A-1kN). Following the ASTM recommendation [61], the 4PBT jig with the SENB specimen was periodically removed from the corrosive environment and observed under an optical microscope to measure the crack length. The corrosion products on the side surfaces of the SENB specimen were wiped using wet paper before the crack length measurement to obtain a clear observation. The crack length was measured by direct optical measurement with a resolution of 10 μm . During the crack length measurement, the corrosion product on the crack surfaces of the SENB specimen may come into contact with air, and its properties may change. To minimize this effect, the period of the measurement was controlled, i.e., less than 30 minutes. The compressive load was also removed prior to the crack length measurement. Therefore, the crack surfaces were closed, and the contact of the corrosion product around the crack tip with air was limited. The

crack length measurement was performed on both side surfaces of the SENB specimen, and the average crack length was determined.

During the SCC experiment, relaxation of the compressive load occurs due to crack propagation. Although relaxation of the compressive load was marginal, this behaviour should be considered. Thus, the loads and crack lengths before and after crack propagation were averaged and used for the FEA of the J -integral. While the crack lengths before and after crack propagation were used for determining the crack growth rate (da/dt).

The SCC experiment was divided into a J -decreasing experiment and a J -increasing experiment. Similar procedure of crack length measurement was applied for the J -decreasing and J -increasing experiments. For the J -decreasing experiment, deflection of the SENB specimen was achieved by turning the bolt on the 4PB jig, and then it was maintained constant. An initial J -integral (J_i), i.e., $J_i > J_{SCC}$, was then calculated from the initial load and initial crack length. As the crack propagated, the J and da/dt decreased with increasing crack length. The crack eventually arrested; thus, the J became J_{SCC} . Crack arrest is defined as the situation where the $da/dt \leq 3.6 \times 10^{-4}$ mm/hr [7].

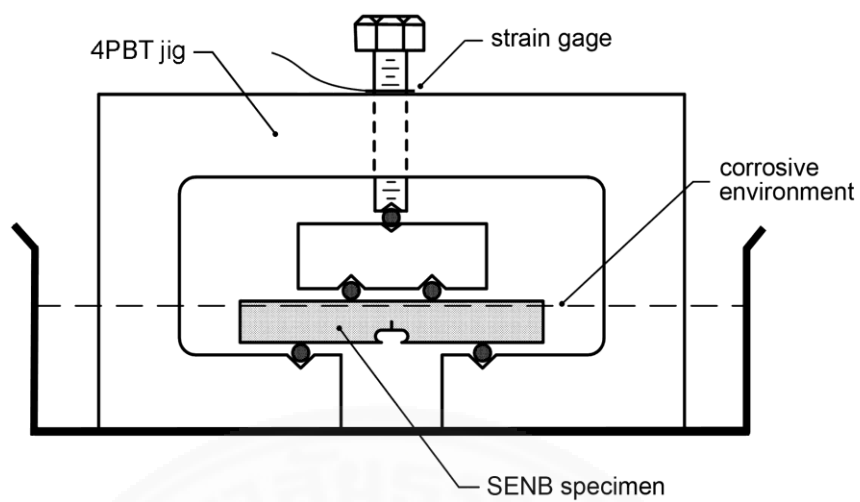


Fig. 2-4 Illustrations of stress corrosion cracking experiment.

After the crack arrested, the J -increasing experiment was performed on the SENB specimen to obtain the relationship between J and da/dt at higher stress levels. The applied load was periodically increased, which resulted in increases in J and da/dt . The applied load was measured using full-bridge strain gauges, which were attached on the 4PBT jig. The time interval between the load-increasing steps depended on the crack growth rate, e.g., approximately 25 hours at low da/dt and 10 hours at high da/dt . The increases in J and da/dt continued until complete fracture, i.e., J reached the critical J -integral or fracture toughness (J_c). After fracture, the side surfaces and fracture surfaces of the SENB specimens were observed by SEM. At each testing condition, SCC experiments were repeated twice, and the results were averaged.

2.4 Finite element analysis for J -integral

Although many research studies of SCC have been conducted using the fracture mechanics approach, the majority of these studies were based on the assumption of linear-elastic fracture mechanics. Unfortunately, a large plastic zone at the crack tip is possible for thin NAB components under corrosive environments, and thus, the linear-elastic fracture mechanics parameter (K_{ISCC}) may become invalid. Therefore, the elastic-plastic fracture mechanics (EPFM) parameter should be applied as an alternative. Based on finite element analysis (FEA), the stress/strain state near the crack tip and the J -integral were numerically calculated. The J -integrals were correlated with the crack growth rates (da/dt) from the SCC tests.

2.4.1 Modelling

Three-dimensional elastic-plastic FEA, i.e., ABAQUS [51], was used to determine the stresses and strains around the crack tip of the SENB specimen under 4-point bending, as shown in Fig. 2-5a. The Ramberg-Osgood relationship was used as the constitutive equation for the present FEA, and the mechanical properties of NAB determined from the tensile test (Table 2-2) were used as the material input for the FEA. The elements around the crack tip were quarter-point singular elements, and their nodes were allowed to move to provide an elastic-plastic response. Quadratic hexahedral elements were used elsewhere. Boundary conditions were applied on the

SENB specimen, as shown in Fig. 2-5b. Rotation around the z -axis was allowed at both lower supports. On the lower supports, the displacements in all directions were under constraint, except the displacement in the x direction of one of the lower supports was allowed to represent the bending of the SENB specimen. During the FEA, the SENB specimen was loaded by uniform compression forces. The dependence of the FEA results on the element size was minimized by varying the element size until the von Mises stress around the crack tip became stable. The FEA model had 44,250 elements and 189,907 nodes, and the smallest element at the crack tip was 50 μm .



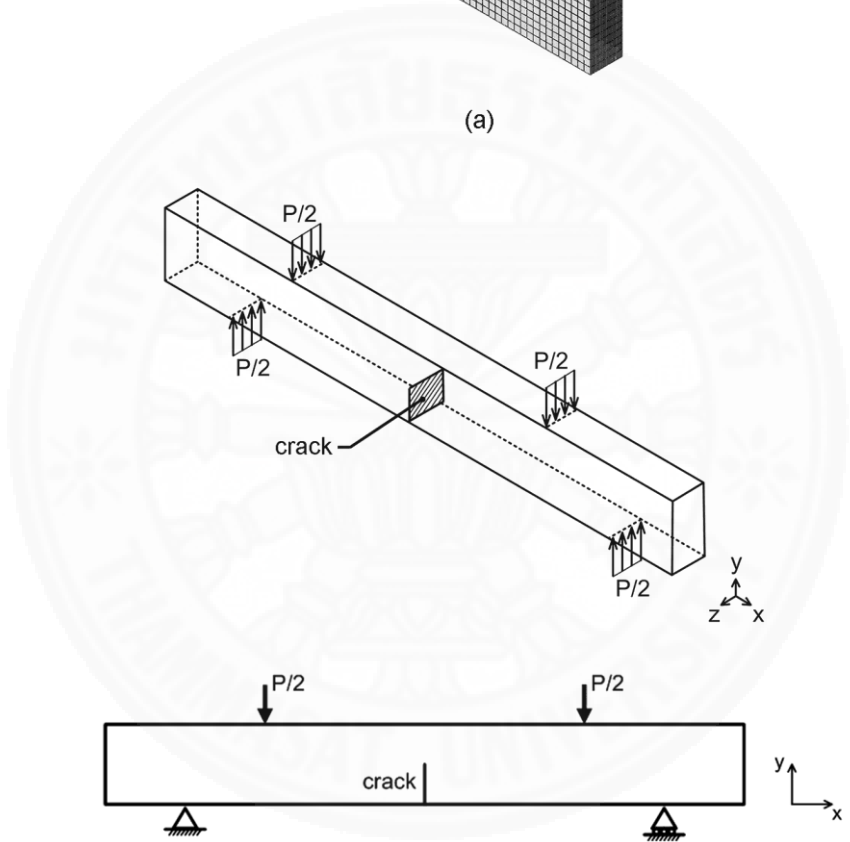


Fig. 2-5 Single-edge notch bending specimen: (a) FE model, (b) boundary condition.

2.4.2 Validation

Because J -integral at crack tip cannot directly measured from experiment; but the crack mouth opening displacement (i.e., a parameter represents the elastic-plastic strains of a single-edge notch bending specimen) can be accurately measured during experiment. Thus, it was selected as a parameter for the verification of the FEA. The crack mouth opening displacements (CMODs) of the SENB specimen ($a/W = 0.53$) calculated from FEA were compared to those measured from the experiment conducted under an air environment, as shown in Fig. 2-6. During the experiment, the crack did not propagate, and the CMOD was only a function of the applied load. The applied load was increased by turning a bolt of the 4PB jig, which resulted in the CMOD increasing. The CMOD was measured using a clip gauge with a resolution of 10 μm . In contrast, the load and crack length were used for the FEA of the CMOD. As observed in the figure, the calculated CMOD is in good agreement with the measured CMOD, and thus, the verification of the FEA is confirmed.

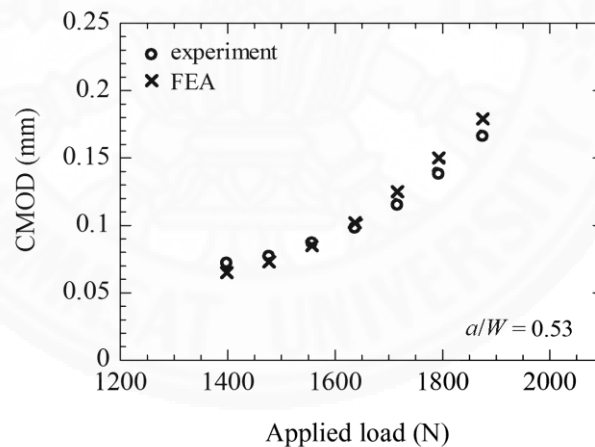


Fig. 2-6 Relationships between crack mouth opening displacement and applied load of SENB specimen under air environment ($a/W = 0.53$).

2.4.3 J -integral

To evaluate the size of plastic zone at crack tip, the distribution of von Mises stress on the mid-thickness plane of the SENB specimen ($a/W = 0.5$) under a 1,750-N load is shown in Fig. 2-7. It is observed that the plastic zone is significantly larger than the thickness, crack length, and remaining ligament, i.e., a large-scale yielding condition. It is known that the stress intensity factor (K), i.e., a linear-elastic fracture mechanics parameter, should not be applied to characterize the severity at the crack tip of an engineering component if the size of the monotonic plastic zone at the crack tip is larger than the thickness (B), crack length (a), and/or remaining ligament ($W-a$) [4],

$$B, a, (W - a) \leq 2.5 \left(\frac{K}{\sigma_Y} \right)^2 \quad (2-6)$$

where σ_Y is the yield strength. Based on the yield strength of the present NAB ($\sigma_Y = 214$ MPa) and the applied K at the crack tips of the SENB specimens during the SCC experiments, i.e., 12 to 25.5 MPa.m^{1/2}, the sizes of the monotonic plastic zones at crack tips were estimated to be 7.9 to 35.5 mm. These monotonic plastic zones are significantly larger than the thickness, crack length, and remaining ligament, which correspond to the FEA (Fig. 2-7). Accordingly, a linear-elastic fracture mechanics parameter (i.e., a stress intensity factor or K) is not justified to apply for the present SENB specimen. As an elastic-plastic fracture mechanics parameter, the J -integral should be applied to characterize the severity at the crack tip under a large-scale yielding condition.

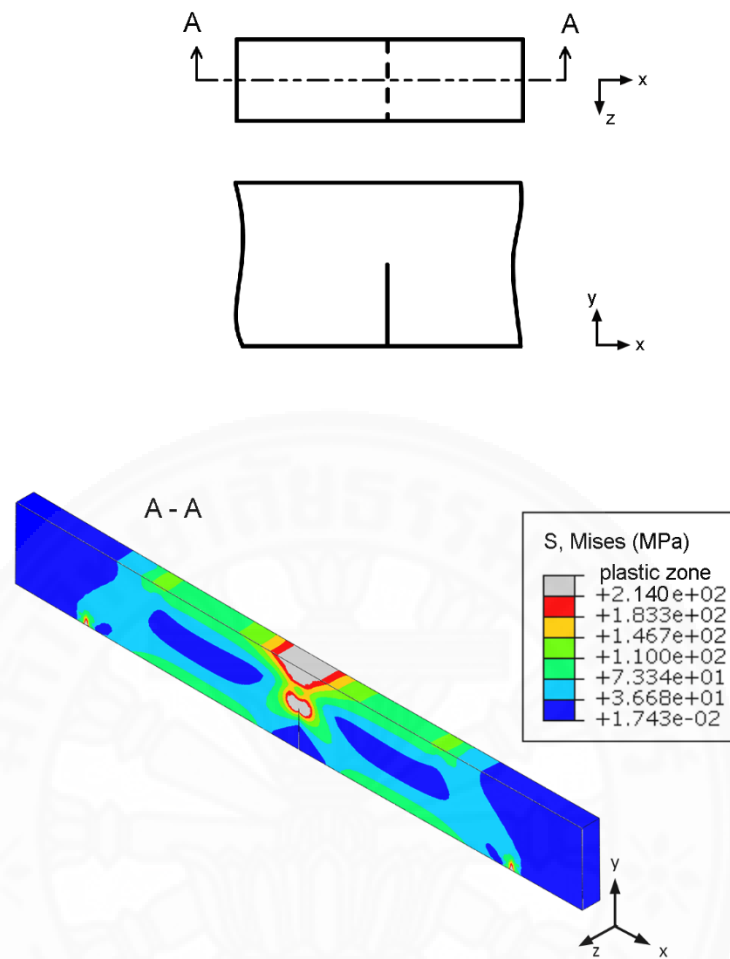


Fig. 2-7 Distribution of von Mises stress on the mid-thickness plane of SENB specimen ($a/W = 0.5$) under a 1,750-N load.

For an arbitrary counter-clockwise path Γ around the crack tip (Fig. 2-8), the J -integral is given by [15]:

$$J = \int_{\Gamma} \left(w dy - T_i \frac{\partial u_i}{\partial x} ds \right), \quad (2-7)$$

where w is the strain energy density, T_i are the components of the traction vector, u_i are the components of the displacement vector, and ds is the length increment along the path Γ . The strain energy density can be defined as,

$$w = \int_0^{\varepsilon_{ij}} \sigma_{ij} d\varepsilon_{ij}, \quad (2-8)$$

where σ_{ij} and ε_{ij} are the stress and strain tensors, respectively. The components of the traction vector can be defined as,

$$T_i = \sigma_{ij} n_j, \quad (2-9)$$

where n_j are the components of the unit vector normal to the path Γ .

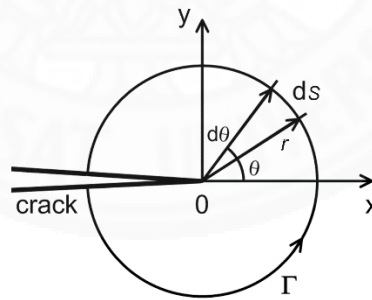


Fig. 2-8 Schematic illustration of the line integral of J along an arbitrary path Γ .

Three-dimensional elastic-plastic FEA, i.e., ABAQUS [51], was used to determine the J -integral of a SENB specimen under 4-point bending. Variation in the J -integral along the crack front of the SENB specimen is observed from the FEA because the plane stress condition occurs on both free surfaces of the SENB specimen.

Therefore, the state of stress is in two dimensions, and the J -integrals around these regions are lower than those around the mid-thickness region, where the state of stress is in three dimensions or plane strain. As an example, the J -integrals along the crack front of the SENB specimen with an arbitrary crack length ($a/W = 0.5$) under an arbitrary load (1,750 N) are shown in Fig. 2-9a. On the other hand, the J -integrals at various loads and crack lengths are shown in Fig. 2-9b. In the present work, the J -integral at the mid-thickness plane was used as an elastic-plastic fracture mechanics parameter to characterize the crack growth.

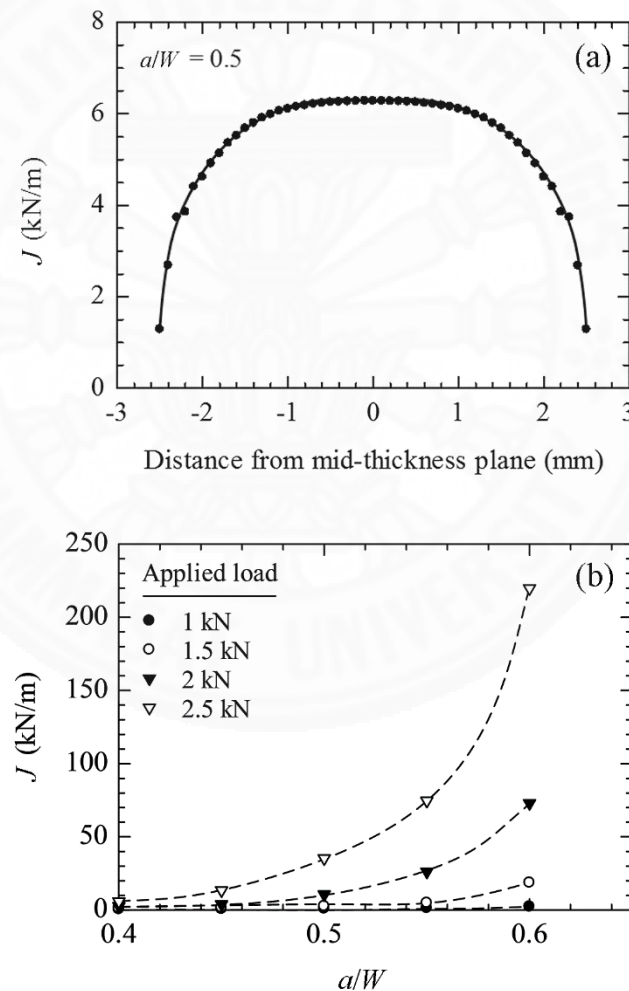


Fig. 2-9 (a) J -integrals along the crack front of SENB specimen ($a/W = 0.5$) under a 1,750-N load and (b) J -integrals at various loads and crack lengths.

Chapter 3

Stress corrosion cracking under plane stress condition: results and discussion

To evaluate the SCC under plane stress condition, the threshold J -integral, critical J -integral, SCC propagation curve, corrosion product, crack propagation path, and fracture surface were analyzed. SCC behaviour and mechanism are discussed. Detail of the discussions can be described as follows.

3.1 Threshold J -integral for susceptibility to stress corrosion cracking

The crack lengths and loads during the J -decreasing experiments of NABs under SW and SW:AH environments exhibited similar behaviour. As an example, the crack length and load as functions of time during the J -decreasing experiment of NAB under the 5:1 / SW:AH environment are shown in Fig. 3-1. According to the ISO 7539-6 standard [7], crack arrest is defined as the situation where $da/dt \leq 3.6 \times 10^{-4}$ mm/hr. During the J -decreasing experiment, the crack initially propagates at a high propagation rate (da/dt). The da/dt decreases with time, and it eventually becomes unmeasurable after 70 hours. The J -decreasing experiment is continued for approximately 200 hours to ensure the condition of unmeasurable crack propagation. Because the crack length is measured via a direct optical measurement with a resolution of 10 μm ; the unmeasurable crack propagation corresponds to a da/dt of $\leq 5 \times 10^{-5}$ mm/hr.

Propagation of crack involves damage in the vicinity of the crack tip, i.e., stress and corrosion. It is likely that the combination between stress and corrosion is high enough to propagate the crack during the first 70 hours of the J -decreasing experiment. Because the deflection of the SENB specimen is maintained constant during the J -decreasing experiment, the resistance load decreases with increasing crack length. Accordingly, the contribution of stress decreases with increasing crack length and causes a decrease in the crack propagation rate (da/dt) during the first 70 hours. After 70 hours, the combination between stress and corrosion is not sufficient to cause crack propagation.

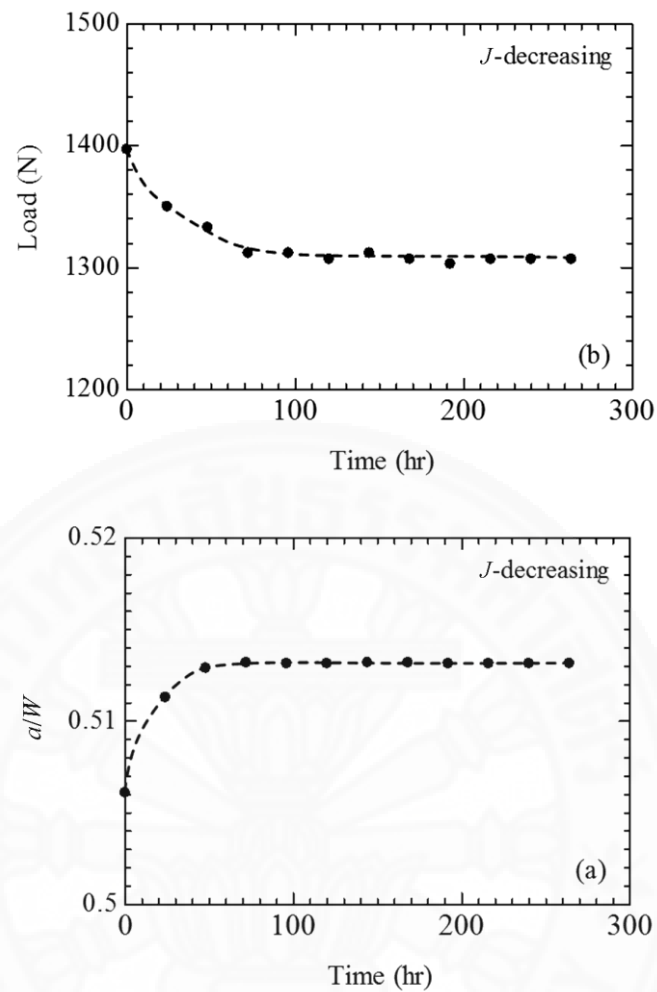


Fig. 3-1 J -decreasing stress-corrosion cracking experiment of NAB under the 5:1 / SW:AH environment: (a) load vs. time, and (b) crack length vs. time.

Although the crack stops propagating after 70 hours, deformation around the crack tip may continue, which causes a further reduction in the load until approximately 100 hours. In the present work, the crack length and load corresponding to a crack growth rate (da/dt) of 3.6×10^{-4} mm/hr are used for the FEA of the threshold J -integral for susceptibility to stress corrosion cracking, i.e., J_{SCC} . The J_{SCC} values of NABs under various corrosive environments are summarized in Table 3-1. Under high ammonia-containing artificial seawater (5:1 / SW:AH environment), the J_{SCC} is the lowest, followed by those under the 10:1 / SW:AH environment and the SW environment. The difference in J_{SCC} between experiment under 5:1 / SW:AH

environment and 10:1 / SW:AH environment is approximately 0.4 kN/m. The effect of this difference on SCC of NAB is systematically shown on the stress corrosion crack propagation curve, which is given in the following section. However, the present findings indicated that (i) SCC of NAB is possible under both seawater and ammonia-containing seawater and that (ii) the SCC of NAB is enhanced by the amount of ammonia present in seawater.

Table 3-1 Parameters of stress-corrosion cracking.

Corrosive environment	J_{SCC} (kN/m)	J_c (kN/m)
SW	3.57	22.66
10:1 / SW:AH	2.72	9.68
5:1 / SW:AH	2.35	8.18
Air	-	22.71

Koul and Gaies [9] evaluated the stress corrosion cracking of heat-treated NAB (C63200) under SW and 5:1 / SW:AH environments. The rising step load (RSL) tests were performed in their study, where a specimen was loaded to a target load, and held at constant displacement for 4 hours. If no crack growth was observed during the 4-h period, the next load step was applied. The crack length and load corresponded to the first observation of crack growth were used for the determination of J_{SCC} . Based on an assumption of small-scale yielding, K_{ISCC} was calculated from J_{SCC} . They observed a subcritical crack growth of 381 $\mu\text{m/hr}$ during the RSL test of NAB under the SW:AH environment. The subcritical load step and subcritical crack length from their test under the SW:AH environment were used for the determination of K_{ISCC} , i.e., 15 $\text{MPa.m}^{1/2}$. However, no subcritical crack growth was detected in their test under the seawater environment. This finding differs from the present result, in which SCC of NAB is possible under artificial seawater. The absence of K_{ISCC} under artificial seawater may relate to their loading profile, i.e., the rising step load (RSL) test with constant displacement period of 4 hours.

Moreover, their size of the monotonic plastic zone at the crack tip is approximately 2.2 mm, which was calculated using Eq. 2-5, the yield strength of their NAB ($\sigma_y = 510$ MPa), and the K_{ISCC} . Because they used a compact-tension (CT) specimen with a thickness of 18 mm, the size of plastic zone is smaller than the thickness of specimen and the crack tip is under the condition of small-scale yielding. Unlike the condition of small-scale yielding, severe deformation occurs at the crack tip in the present work, i.e., the condition of large-scale yielding. The severe deformation at the crack tip may enhance the corrosion process; thus, SCC of NAB is possible under artificial seawater. Unfortunately, the NABs investigated in the present work and in the work of Koul and Gaies [9] are different, i.e., their chemical compositions and mechanical properties differ; thus, additional studies are required to understand the influence of crack tip deformation on SCC of NAB.

3.2 Critical J -integral

After the J -decreasing experiment, the applied load was increased; then, the J -integral became higher than the J_{SCC} . At this crack-tip condition ($J > J_{SCC}$), the crack propagated again, and its da/dt increased as the J -integral increased, i.e., the J -increasing experiment. The loads and crack lengths during the J -increasing experiments of NABs under SW and SW:AH environments showed similar behaviour. As an example, the load and crack length as functions of time during the J -increasing experiment of NAB under the 5:1 / SW:AH environment are shown in Fig. 3-2. During the J -increasing experiment, the crack length increased with increasing applied load. The increase in the crack length with increasing applied load continued until fast fracture occurred.

Because the applied load and crack length are increased during the J -increasing experiment, the contribution of stress to the SCC process increases with applied load and crack length. Eventually, the damage process in the vicinity of the crack tip is dominated by the applied stress. If the severity of damage at the crack tip of NAB reaches the fracture condition (J_C), fast fracture occurs. The applied load and crack length at this situation can be used for the FEA of J_C .

The J_C values of NABs under various corrosive environments are summarized in Table 3-1. Under the artificial seawater environment, the J_C is similar to that under the

air environment. The J_c decreases as the amount of ammonia present in the artificial seawater increases. The present finding indicated that the fracture toughness of NAB under the artificial seawater environment is not influenced by the SCC process, whereas the fracture toughness of NAB under the ammonia-containing artificial seawater environment is significantly influenced by the SCC process. To understand the mechanism of SCC under the ammonia-containing artificial seawater environment, the corrosion product, crack path and fracture surface of specimen were evaluated, which are described in the following sections.

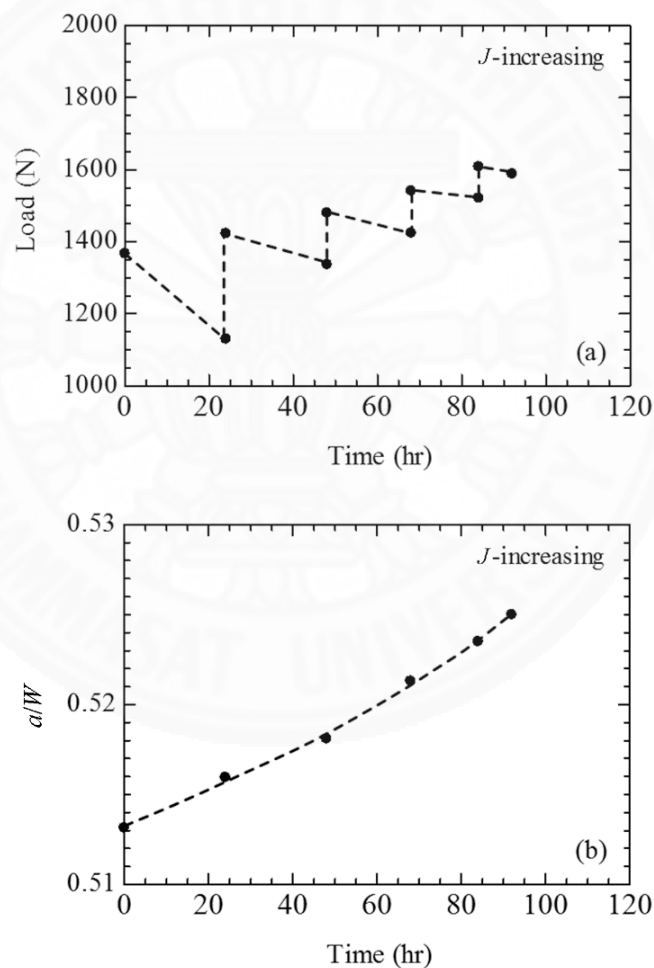
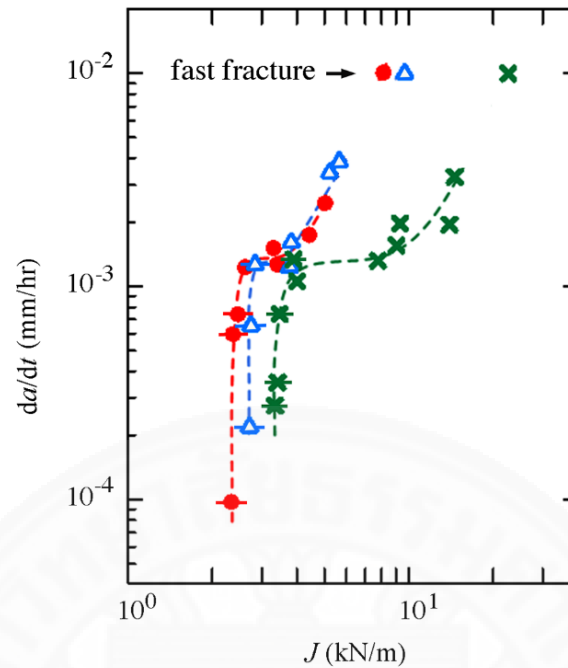


Fig. 3-2 J -increasing stress-corrosion cracking experiment of NAB under the 5:1 / SW:AH environment: (a) load vs. time, and (b) crack length vs. time.

3.3 Stress corrosion crack propagation curve

In the present work, the SCC experiments on NABs under SW and SW:AH environments were repeated twice. Differences between repeated experiments could not be identified when the J and da/dt were plotted on a log-log scale, i.e., the stress corrosion crack growth (SCCG) curve. However, the J and da/dt from repeated experiments were averaged and used for the stress corrosion crack growth (SCCG) curves, as shown in Fig. 3-3. The results from the J -decreasing experiment were plotted at the low da/dt region of the SCCG curve, whereas those from the J -increasing experiment were plotted at the high da/dt region of the SCCG curve. It is noted that the precision of crack measurement would cause the uncertainties of J and da/dt , i.e., approximately $\pm 2\%$ for the present J and da/dt . However, these uncertainties were marginal, and insignificant when the relationship between J and da/dt were plotted on a log-log scale.



SCC experiment	<i>J</i> -decreasing			<i>J</i> -increasing		
	symbol	<i>J</i> (kN/m)	<i>da/dt</i> (mm/hr)	symbol	<i>J</i> (kN/m)	<i>da/dt</i> (mm/hr)
SW	✱	3.3 – 3.6	$2.8 \times 10^{-4} - 7.4 \times 10^{-4}$	✱	4.0 – 22.7	$1.1 \times 10^{-3} - 10^{-2}$
10:1 / SW:AH	△	2.7 – 2.9	$2.1 \times 10^{-4} - 1.2 \times 10^{-3}$	△	2.9 – 9.7	$1.2 \times 10^{-3} - 10^{-2}$
5:1 / SW:AH	●	2.2 – 2.4	$1.0 \times 10^{-4} - 7.3 \times 10^{-4}$	●	2.6 – 8.2	$1.2 \times 10^{-3} - 10^{-2}$

Fig. 3-3 Relationships between *J*-integral and crack growth rate.

During the *J*-decreasing experiment, the *J* and *da/dt* decrease with crack growth until crack arrest occurs, i.e., the *J* becomes *J*_{SCC}. In contrast, the crack propagates and the *da/dt* rapidly increases during the *J*-increasing experiment until it reaches approximately 1.2×10^{-3} mm/hr, where the SCCG curve exhibits a plateau. It is believed that the corrosive environment plays a dominant role in the initial stage of crack propagation, i.e., $da/dt < 1.2 \times 10^{-3}$ mm/hr. However, the contribution of the corrosive environment decreases with the increasing *J*, and eventually, the corrosive environment is not severe enough to accelerate the crack growth rate. Therefore, the SCCG curve exhibits a plateau of *da/dt* at approximately 1.2×10^{-3} mm/hr during the *J*-increasing experiment. As the increase of *J* continues, the driving force for crack propagation is dominated by mechanical loading. Eventually, the *J* is high enough to

accelerate the crack growth rate again, and thus, the da/dt increases until fast fracture occurs, i.e., J becomes J_c . In the present work, fast fracture is defined as $da/dt \geq 10^{-2}$ mm/hr.

SCCG curves of NABs under the SW and SW:AH environments show similar behaviour. However, the influence of the corrosive environment is greater for NAB under the ammonia-containing seawater environments; therefore, their J_{SCC} , J_c and J at plateau are lower than those of NAB under artificial seawater. It is noted that SCC of NAB under SW:AH environments between SW to 5:1 may be estimated for the present experimental results. However, under the SW:AH environments above 5:1, more experiments are required for SCC behaviours.

3.4 Mechanisms of stress corrosion cracking

To evaluate the SCC mechanisms of NAB, the corrosion product, crack propagation path, and fracture surface were analyzed and related to J_{SCC} , J_c and J vs. da/dt , as follows.

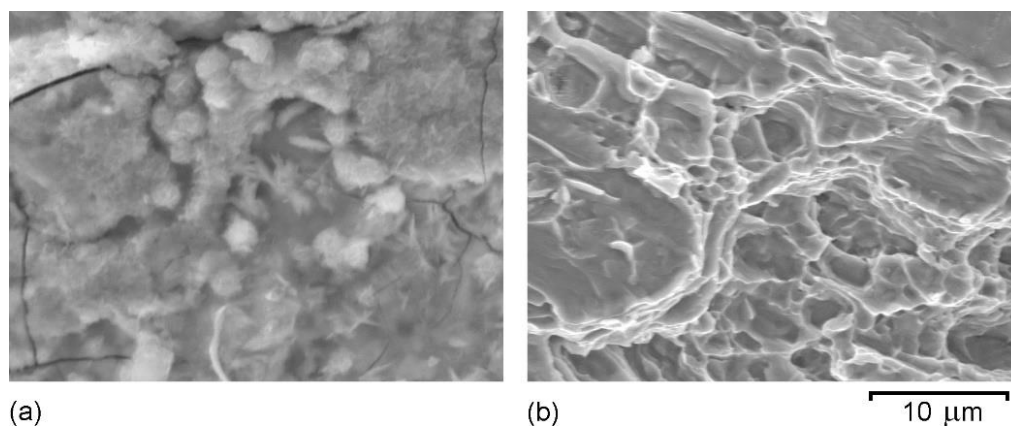
3.4.1 Corrosion product

The corrosion product on the fracture surface was evaluated by an energy-dispersive X-ray spectroscopy (EDS). Note that EDS analysis of elemental composition has limited accuracy; therefore, the EDS technique was applied only for comparative analysis. Because the EDS analysis of corrosion products from the SCC experiments under the SW:AH (5:1 and 10:1) environments showed similar characteristics, only the EDS analysis results of the corrosion product from the SCC experiment under the 5:1 / SW:AH environment are presented here.

EDS analyses and micrographs of the SCC and overload fracture regions of NAB under the 5:1 / SW:AH environment are compared in Fig. 3-4. The lengths of the pre-crack and SCC crack were used to identify the SCC region on the fracture surface. The corrosion product can be clearly observed on the suspected SCC fracture surface (Fig. 3-4a), whereas ductile fracture without corrosion product is a characteristic of the overload fracture region (Fig. 3-4b). On the SCC region, significant amounts of Cl and O

were found, whereas neither of these elements could be detected in the overload fracture region.

Under the present corrosive environments, the corrosion process of NAB involves the anodic reaction of copper ions from the α phase on the crack surfaces, whereas the predominant cathodic reaction is the oxygen reduction reaction. Via a dissolution process, the cuprous oxides (Cu_2O and/or CuCl_2^-) can form on the crack surfaces [62]. On the other hand, the dissolution of the α phase in ammonia solution is a corrosion process, which give the corrosion product of $\text{Cu}(\text{NH}_3)_4^{2+}$ [63]. Because CuCl_2^- is a soluble species, it is believed that the corrosion products on the crack surface may be other species containing chloride, such as $\text{Cu}_2(\text{OH})_3\text{Cl}$. This corrosion product is consistent with the EDS analysis, i.e., the presence of Cu, O, and Cl on the SCC region (Fig. 3-4a). Moreover, $\text{Cu}(\text{NH}_3)_4^{2+}$ can be dissolved into the corrosive environment, i.e., the mixture of artificial seawater and ammonium hydroxide. The dissolution of the corrosion product is confirmed by; (i) the lower wt% of Cu on SCC region of fracture surface than that on overload fracture region, and (ii) the change in colour of the corrosive environment from white (i.e., the SW:AH environment) to blue (i.e., the SW:AH environment and $\text{Cu}(\text{NH}_3)_4^{2+}$ during the SCC experiment, whereas this change in colour was not observed for the case of NAB under artificial seawater.



Region	Composition (wt. %)						
	Ni	Al	Fe	Mn	O	Cl	Cu
SCC	0.8±0.1	1.2±0.1	0.8±0.1	-	14.7±0.3	7.3±0.1	67.7±0.5
Overload fracture	7.1±0.2	8.6±0.2	4.0±0.1	1.2±0.1	-	-	77.2±0.5

Fig. 3-4 EDS analyses and micrographs of fracture surface of NAB under the 5:1 / SW:AH environment: (a) SCC region and (b) overload fracture region (crack propagated from bottom to top).

3.4.2 Crack propagation path

During the SCC experiment, the 4PBT jig with the SENB specimen was periodically removed from the corrosive environment and observed under an optical microscope. During the observation of the crack on the side surfaces of the SENB specimen, the propagation of crack out of plane, i.e., crack kinking, within the SCC region could be observed for the NABs tested under the ammonia-containing artificial seawater environments (5:1 / SW:AH and 10:1 / SW:AH). In contrast, the crack of NAB tested under the artificial seawater environment appeared in a regular manner, i.e., perpendicular direction to the applied bending stress.

To obtain details of the crack propagation path within the SCC region, the failed SENB specimen was sectioned on the mid-thickness plane and observed by SEM. Although the corrosive environment exhibited a dominant role in the initial stage of crack propagation of NAB under the artificial seawater environment (i.e., a plateau of

SCCG curve in Fig. 3-3), the crack propagation path within the SCC region exhibited a ductile fracture appearance without clear evidence of any corrosion process. However, the crack propagation paths of NABs tested under the ammonia-containing artificial seawater environments (5:1 / SW:AH and 10:1 / SW:AH) showed a similar combination between ductile fracture and the SCC process, i.e., the appearance of blunted secondary cracks. As examples, the crack propagation paths of the SENB specimens tested under the artificial seawater environment and 10:1 / SW:AH environment are shown in Fig. 3-5a and 3-5b, respectively. Under the 10:1 / SW:AH environment, the orientation of the crack within the SCC region changes during propagation, i.e., the dependence of the crack propagation path on the microstructure. However, the dependence of the crack propagation path within the pre-cracking region is unlikely, i.e., the orientation of the crack is in the perpendicular direction to the cyclic stress during the pre-cracking process.

In the SCC region (area A of Fig. 3-5b), the crack propagates mainly through the α -phase and lamellar κ_{III} -phase and along the interface between the α -phase and globular κ_I -phase. The crack orientation is generally in the $\pm 45^\circ$ direction. Evidence of crack arrest followed by a change in crack orientation can be observed along the crack propagation path. Blunted secondary cracks with a tunnel-like appearance are found to propagate within the α -phases and arrest at lamellar κ_{III} -phases, as shown in area B of Fig. 3-5b.

It is known that the stress is significantly high in the vicinity of the crack tip, which induces the generation and movement of slip dislocations [4]. Although the corrosion resistance of NAB under seawater has been attributed to the formation of a protective layer, the protective layer loses its corrosion resistance under the influence of ammonium hydroxide. Accordingly, the corrosive environment is likely to attack at regions of high dislocation density and interaction. The combination between corrosion and stress can cause corrosion tunnels at dislocation slips. During this process, the corrosion tunnels grow in size and length. Because the dislocation slip involves shear stress, the crack propagation is in the direction of the maximum shear stress, i.e., the $+45^\circ$ and -45° directions to the applied stress.

Although the deformation at the crack tip is high enough to cut through the lamellar κ_{III} phases in some locations, it is believed that the deformation at the crack tip and the crack propagation can be limited by the presence of the lamellar κ_{III} phases in some directions. Thus, evidence of crack arrest at the lamellar κ_{III} phases and a change in the crack propagation path into another maximum shear stress direction are observed in Fig. 3-5b. However, the shape, size, and distribution of globular κ_I phase are not suitable to arrest a crack; therefore, the crack propagates along the interface between α -phase and globular κ_I -phase (area A of Fig. 3-5b). Note that the possible influence of secondary phases on crack propagation is based on the comparison of the crack path within the α -phase region to that in the κ -phase region. However, the influence of secondary phases on crack propagation under the SCC process is complicated and involves other parameters, e.g., electrochemical reactions, fracture mechanics, and mechanical properties. Additional studies are necessary to understand the influence of secondary phases on crack propagation under the SCC process.

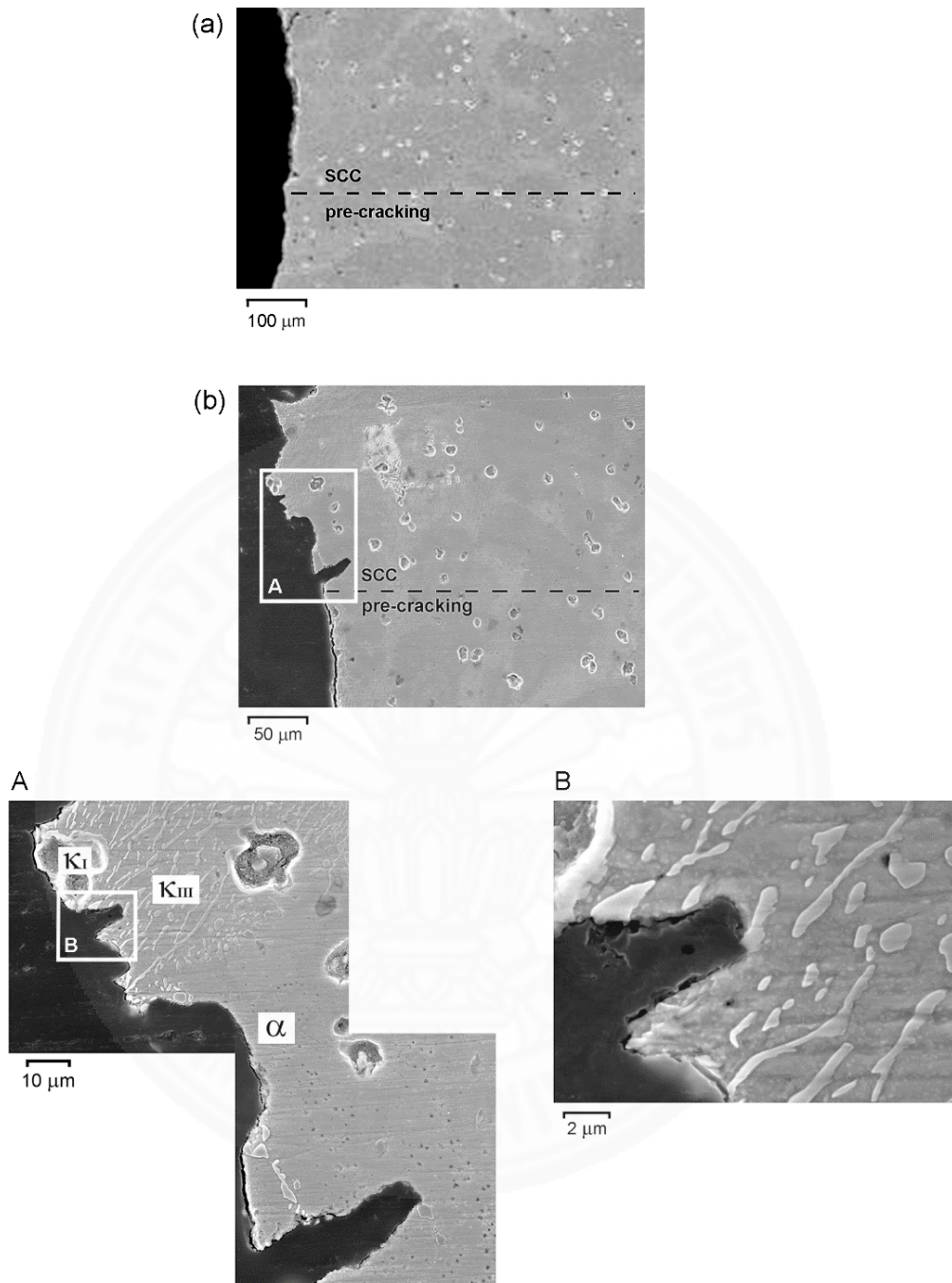


Fig. 3-5 Micrographs of crack propagation paths of: (a) NAB under the artificial seawater environment, and (b) NAB under the 10:1 / SW:AH environment (crack propagated from bottom to top).

3.4.3 Fracture surface

Before observing the fracture surface, the corrosion product was removed by a solution containing 100 mL of H_2SO_4 and 1,000 mL of distilled water. The fracture surfaces of NABs under the 5:1 / SW:AH and 10:1 / SW:AH environments showed a similar appearance. As examples, the fracture surfaces of NABs under the artificial seawater environment and 10:1 / SW:AH environment are shown in Fig 3-6a and 3-6b, respectively. For NAB under the 10:1 / SW:AH environment, the regions of pre-cracking, SCC, and overload fracture are indicated in the figure of the full fracture surface (Fig. 3-6b). The regions of pre-cracking, SCC, and overload fracture were identified based on the crack length measurement during SCC experiment. The fracture surface shows an approximately 0.5 mm deep SCC region. The crack propagation rate in the SCC region varied from 2.1×10^{-4} to 10^{-2} mm/hr (Fig. 3-3); thus, the period of crack propagation in the SCC region was approximately 250 hours. Evidence of corrosion tunnels was observed in the SCC region, whereas no such tunnels could be found in the pre-cracking and overload fracture regions. The severe plastic deformation (i.e., voids or dimples) was observed in overload fracture region, while more brittle fracture with less dimples was observed in pre-cracking region. For NAB under the artificial seawater environment, the evidence of corrosion tunnels could not be found in the SCC region (Fig. 3-6a). This finding corresponds to the observation of the crack propagation paths (Fig. 3-5).

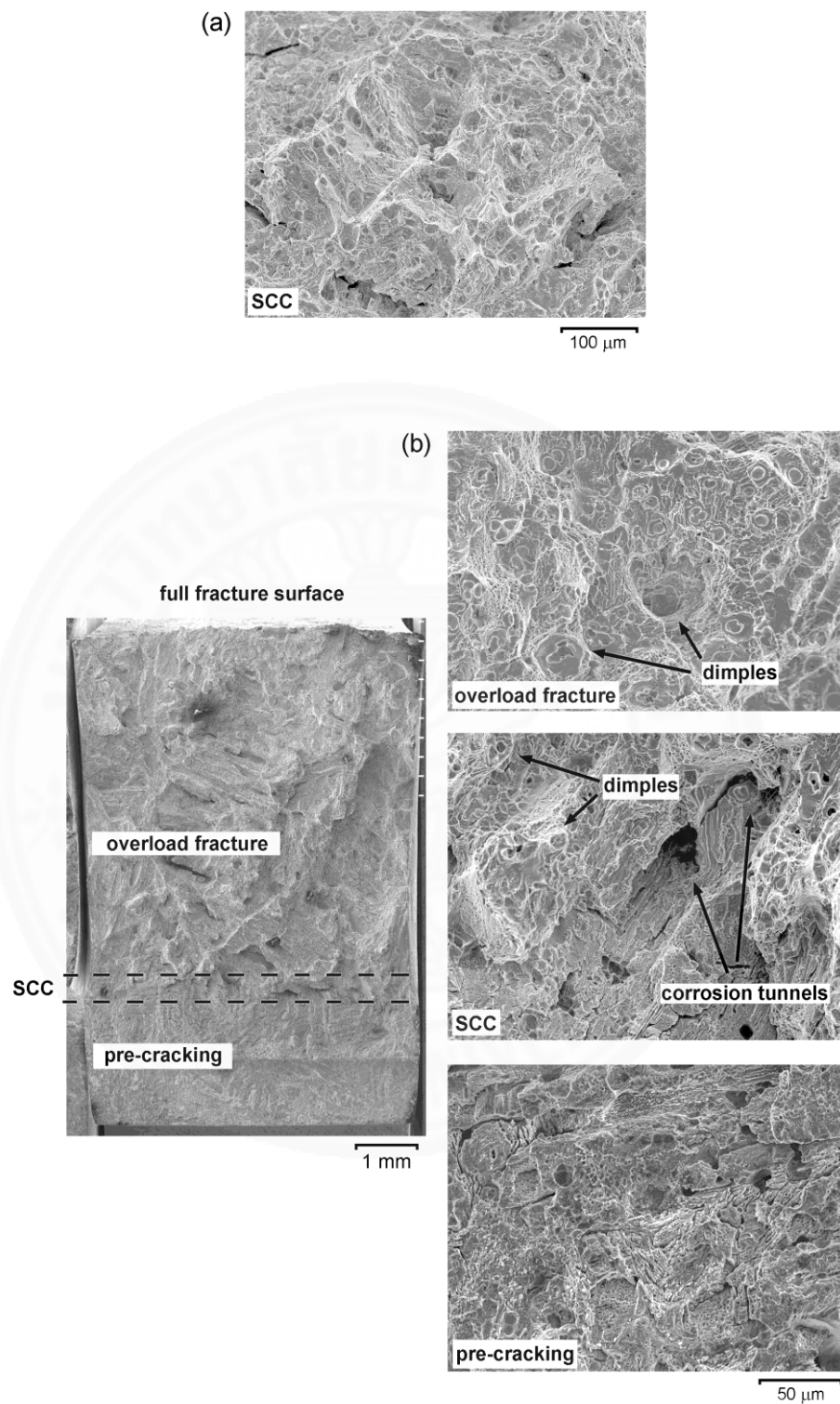


Fig. 3-6 Micrographs of fracture surfaces of: (a) NAB under the artificial seawater environment, and (b) NAB under the 10:1 / SW:AH environment (crack propagated from bottom to top).

3.5 Conclusion

Stress corrosion cracking (SCC) experiments on nickel aluminium bronze (NAB) under artificial seawater and ammonia-containing artificial seawater were conducted using a 4-point bending technique. The ammonia-containing artificial seawater environments were mixtures of artificial seawater and ammonium hydroxide (SW:AH) in volume ratios of 10:1 and 5:1. The stress/strain states near the crack tip and the elastic-plastic fracture mechanics parameter (J -integral) were evaluated using finite element analysis (FEA). The results are summarized as follows.

- During the SCC experiment, the plastic deformation at the crack tip was significantly large; thus, the crack tip was under a large-scale yielding condition. The elastic-plastic fracture mechanics parameter (J -integral) successfully characterized the severity at the crack tip.
- SCC was possible for NABs under both artificial seawater and ammonia-containing artificial seawater. Under a high concentration of ammonium hydroxide (5:1 / SW:AH environment), the threshold J -integral for susceptibility to SCC (J_{SCC}) and fracture toughness (J_C) were the lowest, followed by those under the 10:1 / SW:AH and SW environments.
- For NABs under artificial seawater and ammonia-containing artificial seawater, the SCC process occurred mainly in the copper-rich solid solution phase (α -phase). Both chloride and ammonium were the main causes of the SCC process for NABs under the ammonia-containing artificial seawater environments. In contrast, chloride was the main cause of the SCC process for NAB under the artificial seawater environment.
- For NABs under the ammonia-containing artificial seawater environments, tunnel-like cracks propagated in the maximum shear stress direction through the α -phase and lamellar κ_{III} -phase and along the interface between the α -phase and globular κ_I -phase. Crack arrest was observed at the lamellar κ_{II} -phases before the crack changed its path into another maximum shear stress direction. Although crack growth was possible for NAB under artificial seawater, there was no clear evidence of a corrosion process on its fracture surface, i.e., the fracture surface exhibited a ductile fracture appearance.

- The present findings gave some guidelines for practitioners to, e.g., avoid SCC by maintaining J below J_{SCC} , and predict the crack growth rate (da/dt) and the J for fast fracture (J_C) using the stress corrosion crack growth (SCCG) curve.



Chapter 4

Stress corrosion cracking under plane strain condition: crack length measurement

Under plane strain condition (i.e., a thick specimen with small plastic zone at crack front), a large SENB specimen and a large 4PBT jig are required for SCC experiment. Unfortunately, SCC experiment with a large SENB specimen and a large 4PBT jig is difficult and expensive to perform. Thus, T-WOL specimen is used for SCC under plane strain condition. The indirect crack length measurement technique using instrumented reaction pin (i.e., a support of the loading bolt) was developed to measure the crack length of a T-WOL specimen. The proper location of strain measurement in an instrumented reaction pin was determined using FEA. The instrumented reaction pin was calibrated using an electric fatigue machine, and applied to measure the crack-opening force of a T-WOL specimen. Based on the three-dimensional elastic-plastic finite element analysis (3D elastic-plastic FEA) of a T-WOL specimen, the measured force as well as the measured CMOD were used as the input for the numerical calculation of crack length. Detail of the SCC experiment using T-WOL specimen with instrumented reaction pin are given as follows.

4.1 T-type wedge opening loaded specimen and stress corrosion cracking test

Stress corrosion cracking experiment using T-WOL specimen with loading bolt and reaction pin is shown in Fig. 4-1. T-WOL specimen, loading bolt and reaction pin were produced from a cast nickel aluminum bronze (NAB). NAB was supplied by the Royal Thai Navy, and its mechanical properties are summarized in Table 2-2. The geometry of T-WOL specimen (Fig. 4-2) is in accordance with ISO 7539-6 [7].

T-WOL specimen is self-loaded by tightening a bolt in the upper half against a reaction pin at the lower half to generate an opening of crack surfaces. An environmental chamber is either attached to the crack tip of specimen, or the specimen is contained within the chamber. CMOD is measured by a displacement gage. According to ISO 7539-6 [7], the valid range of crack length (a) of T-WOL specimen for SCC experiment is $0.3 \leq a/W \leq 0.8$, where W is the distance from load line to back

surface. To facilitate the pre-cracking process, a chevron notch with 120° was machined on a T-WOL specimen using an electrical discharge machine (EDM). Fatigue crack growth experiment with stress ratio of 0.1 was applied to create a sharp pre-crack. Although the fatigue crack has a curved crack front; the crack is assumed to be a straight crack front (i.e., as recommended by ISO 7539-6 [7]). The initial crack length is the average of crack lengths on both sides of a T-WOL specimen, which was measured by direct optical measurement with a resolution of $10\text{ }\mu\text{m}$.

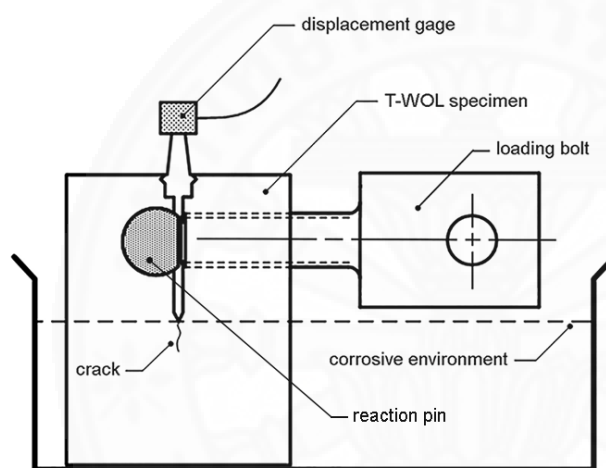


Fig. 4-1 Stress corrosion cracking experiment using T-WOL specimen with loading bolt and reaction pin.

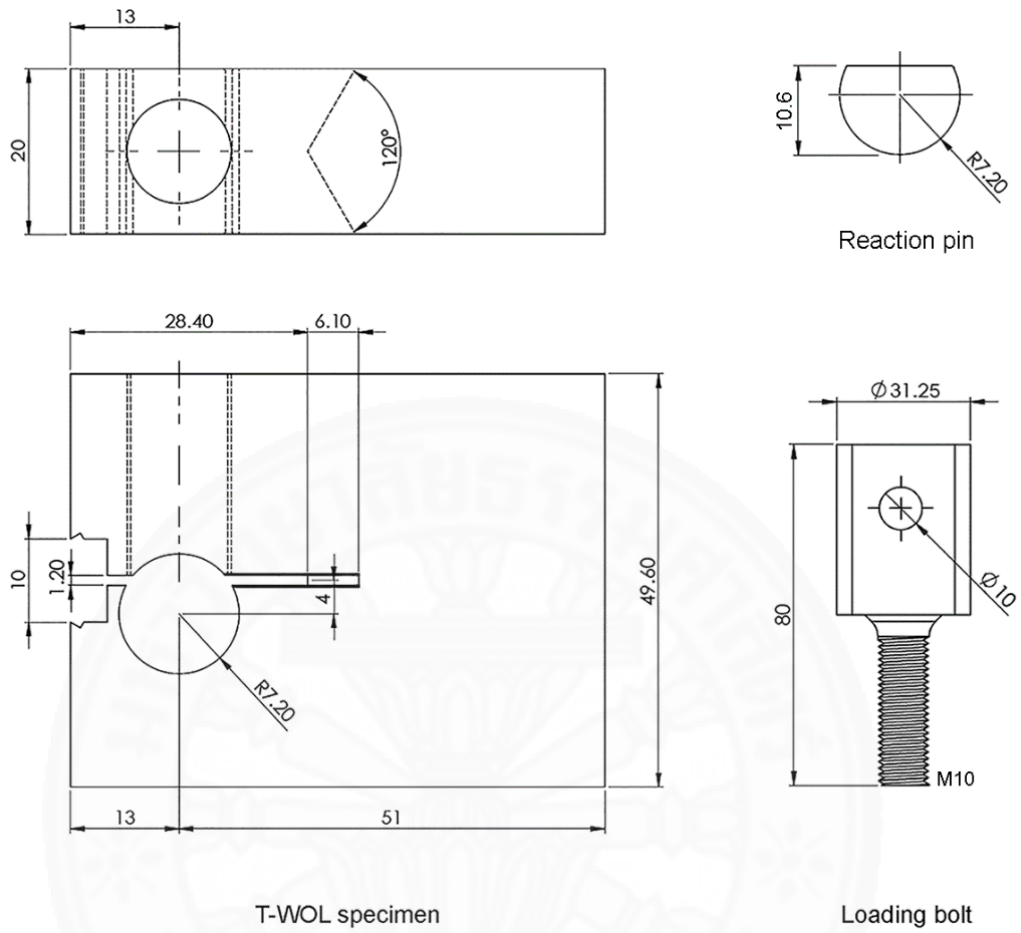


Fig. 4-2 Geometries of T-WOL specimen, loading bolt, and reaction pin (dimension in mm).

For crack tip under the condition of small-scale yielding, K_I of a T-WOL specimen can be determined as follows [7],

$$K_I = \frac{YF}{B\sqrt{a}} \quad (4.1)$$

$$Y = 30.96 \left(\frac{a}{W}\right) - 195.8 \left(\frac{a}{W}\right)^2 + 730.6 \left(\frac{a}{W}\right)^3 - 1186.3 \left(\frac{a}{W}\right)^4 + 754.6 \left(\frac{a}{W}\right)^5 \quad (4.2)$$

where, B is the thickness of a T-WOL specimen, and F is the crack-opening force.

4.2 Instrumented reaction pin for crack-opening force measurement

To measure applied compressive force from loading bolt, the instrumented reaction pin was drilled from the center of curve surface (Fig. 4-3), and a strain gage was applied to measure the normal strain in vertical direction. A special strain gage for longitudinal strain measurement of bolt (TML: BTM-1C) was used for an Instrumented reaction pin. After inserts a strain gage in a hole, the strain gage adhesive was injected into a hole and completely covered the body of strain gage. The measured strain was converted to an applied compressive force using a calibration factor. The instrumented reaction pin should have a linear relationship between the applied compressive force and the measured normal strain. Moreover, the maximum compressive stress of strain gage adhesive should be lower than its compressive strength. To achieve these criteria, the relationship between the normal strain in vertical direction and applied compressive force was numerically evaluated. Under the expected crack-opening force, the normal stress of strain gage adhesive was also numerically calculated to confirm its structural integrity.

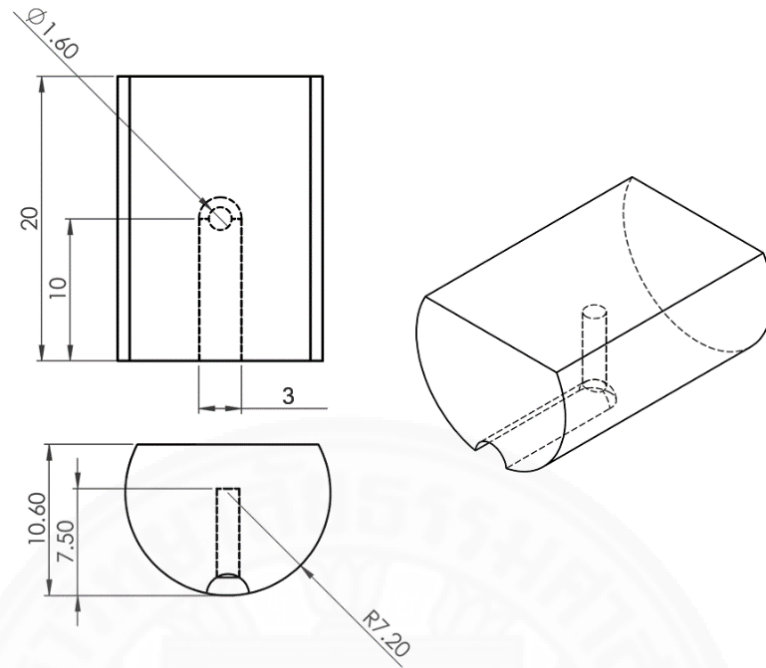


Fig. 4-3 Geometries of instrumented reaction pin (dimension in mm).

4.2.1 Modelling

Three-dimensional linear-elastic FEA, i.e., ABAQUS [51], was used to determine the normal stress and normal strain of the instrumented reaction pin. To reduce the computation cost, the assembly of instrumented reaction pin with a partial loading bolt and a partial T-WOL specimen (i.e., support block) were used for FEA (Fig. 4-4a). Because the assembly of FE models is symmetry on the mid-thickness plane, FEA was performed using half of the assembly. The quadratic hexahedral elements were used for all components of FE model (Fig. 4-4b).

Mechanical properties of NAB are used for the instrumented reaction pin, loading bolt and support block (Table 2-2), while the 3.1-GPa elastic modulus and 38-MPa compressive strength were used for the epoxy-based strain-gage adhesive. The tied contact was assumed along the contact between strain gage adhesive and instrumented reaction pin, while the frictional contact, master-slave algorithm, and penalty method were applied to other contacts [51]. The slip of frictional contact is assumed to occur when the shear stress (τ) is greater than the critical shear stress (τ_c), i.e.,

$$\tau \geq \tau_c = \mu\sigma, \quad (4-3)$$

where, σ and μ are the normal stress and the friction coefficient, respectively. For the contact between NAB components in air, the friction coefficient is 0.50 [64].

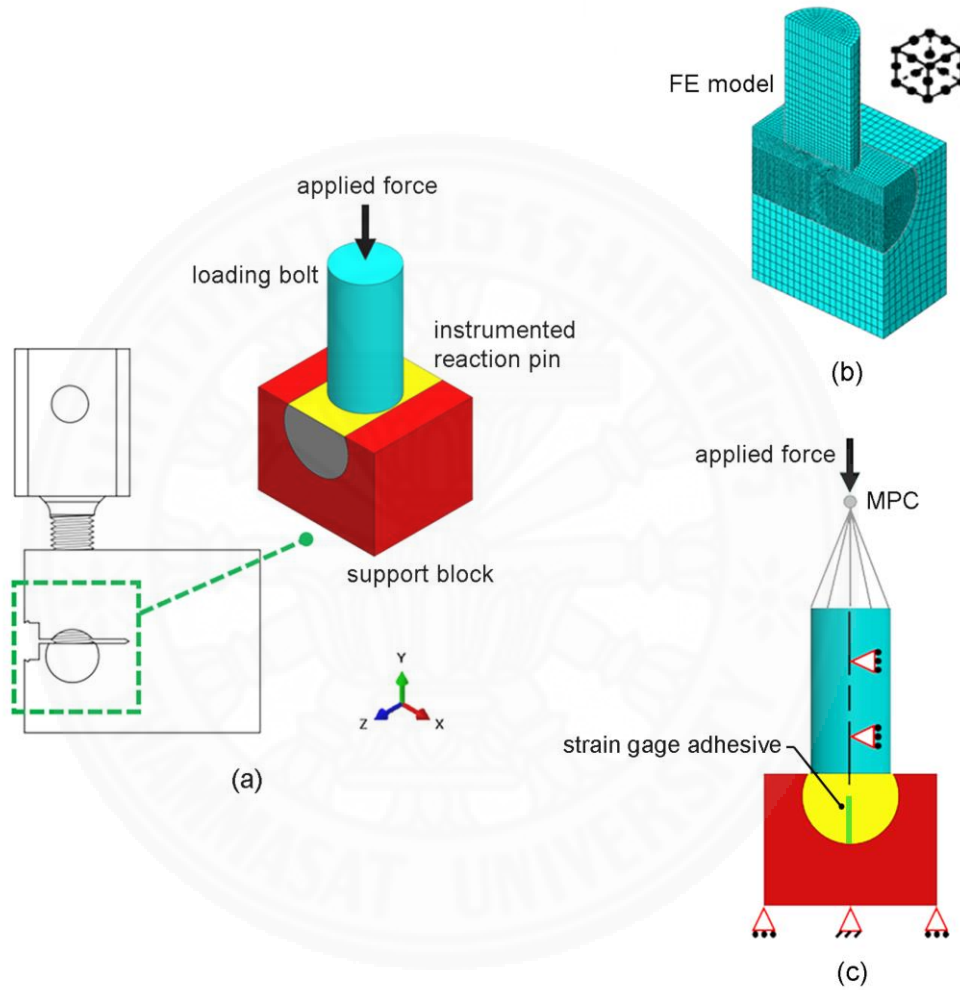


Fig. 4-4 FE model: (a) assembly of instrumented reaction pin with a partial loading bolt and a partial T-WOL specimen, (b) mesh configuration, and (c) boundary condition.

During FEA, the compressive force was applied on the upper part of loading bolt using multi-point constraint (MPC) method. Boundary conditions on the loading bolt and support are shown in Fig. 4-4c. Based on the fracture toughness of NAB (i.e.,

$K_{IC} = 25 \text{ MPa}\cdot\text{m}^{1/2}$) and the shortest crack length of present T-WOL specimen ($a/W = 0.4$), the maximum crack-opening force measuring by an instrumented reaction pin is approximately 14 kN. Therefore, during FEA, the applied compressive force was varied with the intervals of 100 steps from zero to 14 kN. The dependence of FEA results on element size was minimized by varying element size until the maximum von Mises stress on each component became stable. The total numbers of elements and nodes of FE model was 37,973 elements and 168,075 nodes, and the sizes of elements are in the range of 0.06 to 1.9 mm.

4.2.2 Stress and strain of instrumented reaction pin

The calculated von mises stress (σ_v) and normal stress (σ) in the vertical direction of an instrumented reaction pin under the applied compressive force of 14 kN are shown in Fig. 4-5a and 5b, respectively. The maximum von mises stress (i.e., approximately 170 MPa) occurs on the top surface of instrumented reaction pin, which is lower than the yield stress of NAB. Accordingly, an instrumented reaction pin deforms elastically during the crack-opening force measurement. On the other hand, the maximum compressive stress in the strain gage adhesive is approximately -10 MPa, which is lower than its compressive strength. Thus, during the force measurement, the failure of strain gage adhesive is unlikely, and the structural integrity of an instrumented reaction pin is maintained.

The calculated normal strain (ϵ) along the 7.5-mm depth of hole are shown in Fig. 4-6. The 1-mm strain gage is also shown in the figure for comparison. According to the geometry of the strain gage, the 1-mm gage length is located at 5 to 6 mm from the bottom of instrumented reaction pin. The calculated normal strains in the region corresponding to the gage length were averaged, and compared with those from the strain gage measurement to validate the FEA of an instrumented reaction pin.

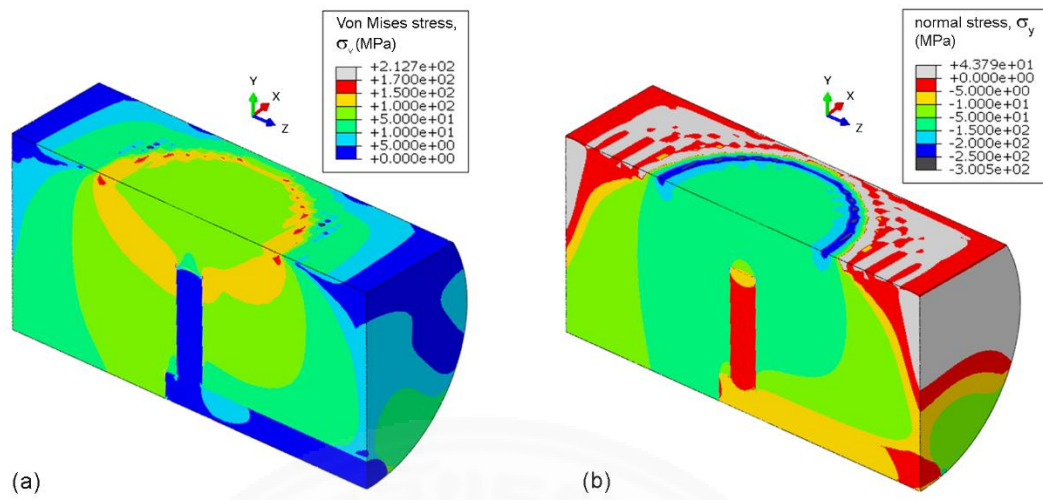


Fig. 4-5 Instrumented reaction pin under an applied compressive force of 14 kN:
(a) von Mises stress, and (b) normal stress.

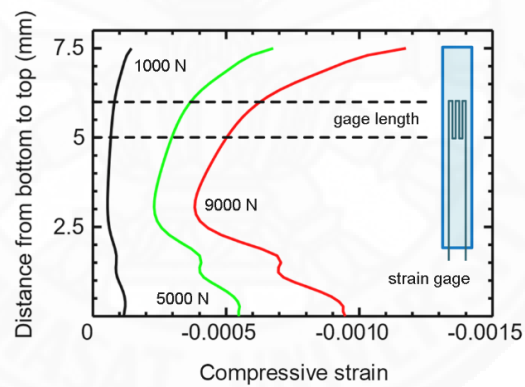


Fig. 4-6 Compressive strains in a function of distance from bottom to top of an instrumented reaction pin, and location of strain gage.

4.2.3 Calibration of instrumented reaction pin

Instrumented reaction pin was produced and tested to ensure the applicability of crack-opening force measurement. It was installed on an electric fatigue machine (Instron: ElectroPuls E10000 with 10 kN load cell) to determine the relationship between compressive force and compressive strain. The experimental setup is shown in Fig. 4-7a. Because of the limitation of testing machine's load cell, the applied compressive forces up to 9 kN were applied on an instrumented reaction pin during experiments. The average results from three repeated tests are compared with the numerical result, as shown in Fig. 4-7b. The results from experiment and numerical calculation are in good agreement. The compressive force vs. compressive strain shows linear behaviour, i.e., R^2 of 0.999 for both experiment and numerical calculation. It is confirmed that the relationship between compressive force and compressive strain from experiment can be used as a calibration curve for the force measurement.

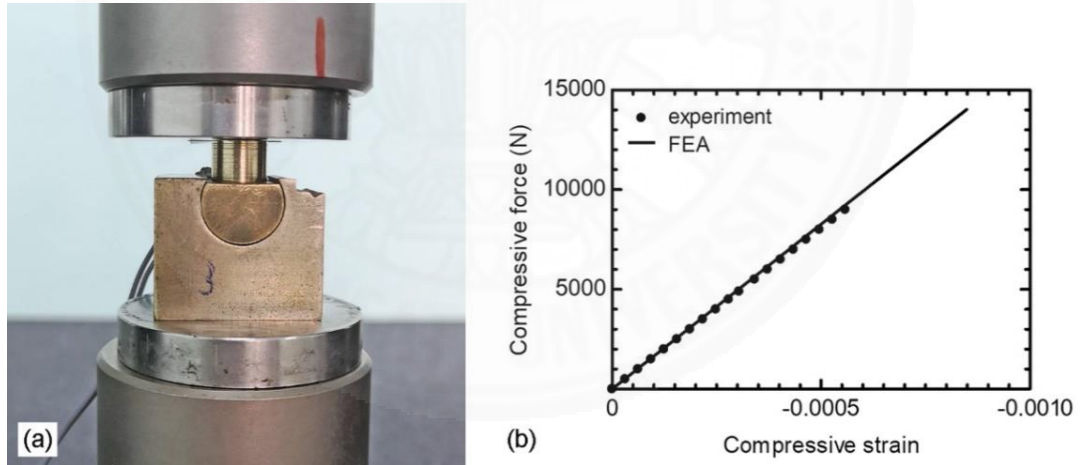


Fig. 4-7 (a) Calibration setup of instrumented reaction pin, and (b) Relationship between compressive force and compressive strain.

4.3 Indirect crack length measurement

4.3.1 Modelling

Three-dimensional elastic-plastic FEA, i.e., ABAQUS [51], was used to determine the relationship between crack-opening force and CMOD of T-WOL specimens with various crack lengths (a/W). To reduce the computational cost, half of an assembly of a T-WOL specimen, an instrumented reaction pin and a loading bolt was used for the present FEA. Straight crack front is assumed for FEA. Geometry and mesh configuration of FE model is shown in Fig. 4-8. The quarter-point singular elements were used around crack front, while the quadratic hexahedral elements were used elsewhere. To provide the deformation around crack tip, the node movement of elements on crack front was allowed.

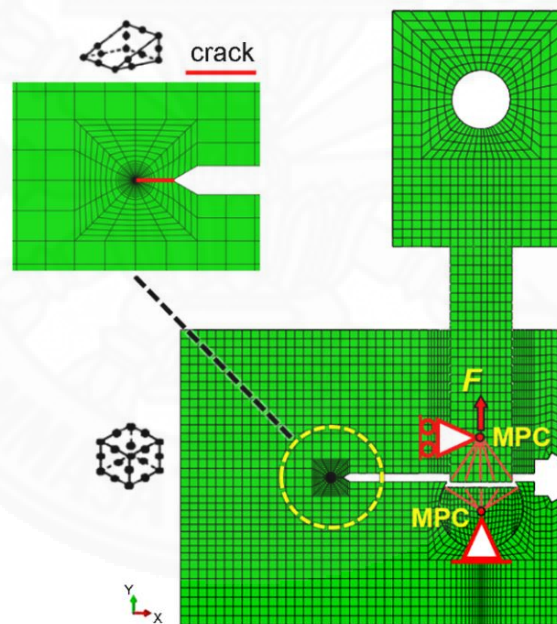


Fig. 4-8 Geometry and mesh configuration of T-WOL specimen with an instrumented reaction pin and a loading bolt.

Elastic-plastic behaviour of NAB (Table 2-2) is used as the mechanical properties of T-WOL specimen, instrumented reaction pin, and loading bolt. The frictional contact was assumed at the contact between T-WOL specimen vs. instrumented reaction pin, while the tied contact was assumed at the contact between

T-WOL specimen vs. loading bolt. Boundary conditions were applied on loading bolt and instrumented reaction pin using multi-point constraint (MPC) method, as shown in Fig. 4-8. On the instrumented reaction pin, the displacement in all directions were under constrain, while only displacement in vertical direction (i.e., y -axis) was allowed for the loading bolt. Based on the crack length and fracture toughness of NAB, the limited load of T-WOL specimen can be determined from Eq. 4-1 and 4-2. During FEA, the crack-opening force was varied with intervals of 100 steps from zero to the maximum force, i.e., limited load of T-WOL specimen. The dependence of FEA results on elements size was minimized by varying elements size until the von Mises stress around crack front became stable. FE model has approximately 55,000 elements and 220,000 nodes, and the sizes of element at crack tip are in the range of 0.08 to 0.2 μm .

4.3.2 Crack-opening force vs. Crack-mouth opening displacement

To investigate the plastic zone size around crack tip at limited load, the von Mises stress in the vicinity of crack tip at mid-thickness plane of a T-WOL specimen ($a/W = 0.65$) was numerically calculated, as shown in Fig. 4-9a. The gray color is defined as the stress greater than the yield strength (i.e., the plastic zone). The plastic zone size is approximately 2.5 mm, which is significantly smaller than the geometry of T-WOL specimen (Fig. 4-2); thus, the condition of small-scale yielding around crack tip is satisfied, and K_I can be applied to characterize the severity at crack tip of the present T-WOL specimen.

Relationship between crack-opening force and CMOD of a T-WOL specimen ($a/W = 0.65$) from FEA was compared with that from experiment, as shown in Fig. 4-9b. The differences between the numerical and experimental results are lower than 1%. Therefore, the FE model is validated, and can be applied to estimate the crack length. Relationships between crack-opening force and CMOD of T-WOL specimens with various crack lengths are shown in Fig. 4-10a. Numerical calculation was performed until the crack-opening force equals to the limited load of T-WOL specimen. The linearity relationship between crack-opening force and CMOD can be observed, which can be applied for the determination of crack length using the measured force (i.e.,

from an instrumented reaction pin) and the measured CMOD (i.e., from a displacement gage).

Because the interpolation of plots between crack-opening force and CMOD (Fig. 10a) to obtain a/W is not convenient; the numerical result at each a/W were arranged, as follows,

$$Z = \frac{1}{(EBV/F)^{1/2}}, \quad (4-4)$$

where, V is the crack-mouth opening displacement on T-WOL specimen's face. Relationship between Z and a/W is shown in Fig. 10b, which can be represented by the following equation,

$$a/W = 1.087 - 8.902Z + 96.674Z^2 - 846.807Z^3 + 2639.150Z^4. \quad (4-5)$$

To determine the crack length of a T-WOL specimen, Z is calculated from the measured force and the measured CMOD, and used for the determination of a/W .

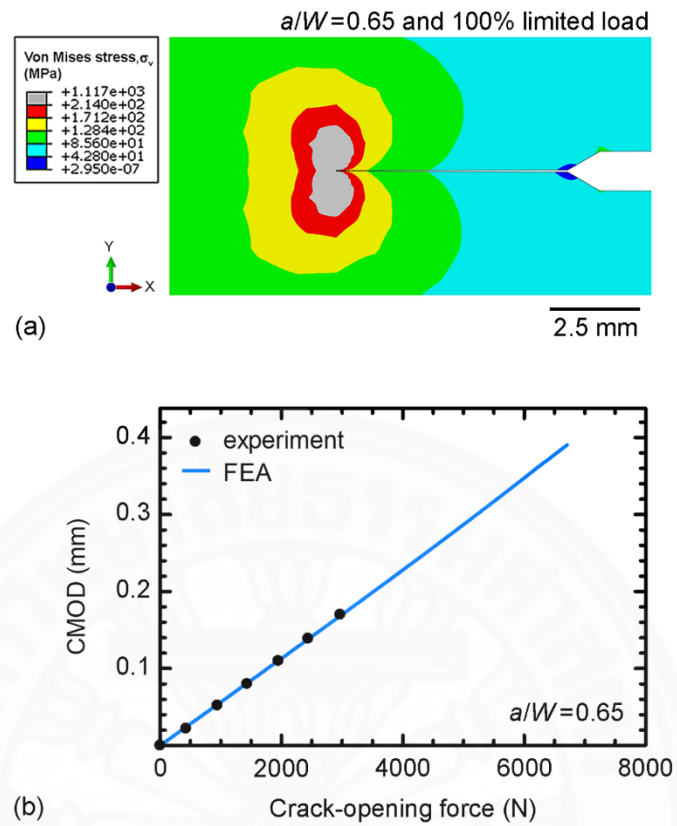


Fig. 4-9 (a) Von Mises stresses in the vicinity of crack tip at mid-thickness plane of a T-WOL specimen, and (b) Relationship between crack-opening force and crack-mouth opening displacement of a T-WOL specimen.

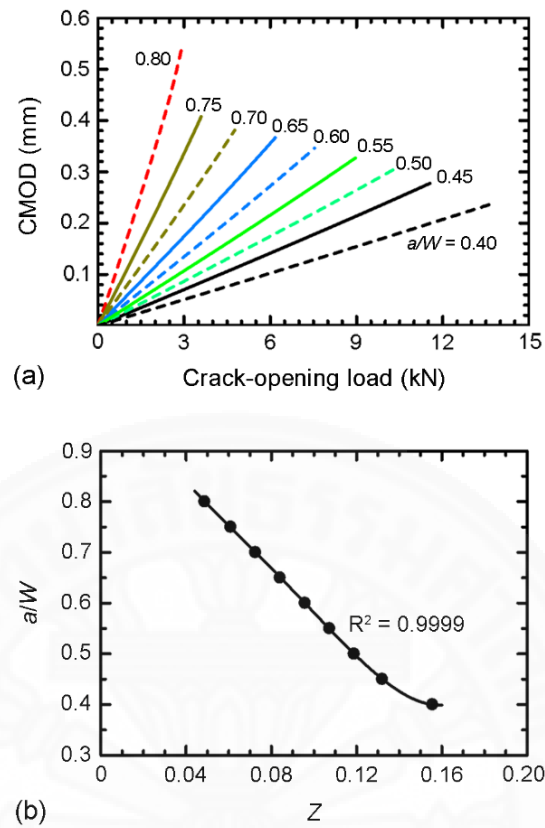


Fig. 4-10 (a) Relationships between crack-opening force and crack-mouth opening displacement of T-WOL specimens with various a/W , and (b) Relationship between Z and a/W .

4.3.3 Validation

T-WOL specimens with different crack length were used for the validation of the present indirect crack length measurement technique. Initially, the displacement of T-WOL specimen was applied by the loading bolt. Then, the crack length (a/W) was measured by a digital microscope with image analysis software, i.e., the direct crack length measurement. Simultaneously, the crack-opening force was measured by an instrumented reaction pin, while CMOD was measured by a displacement gage, and used for the indirect crack length measurement (Eq. 4-4 and 4-5). Both direct crack length measurement and clip gage have resolutions of 10 μm .

Crack lengths from indirect crack length measurement are in good agreement with those from direct crack length measurement, as shown in Fig. 4-11. The differences between a/W from direct and indirect crack length measurement are approximately less than 2%. Therefore, the present indirect crack length measurement technique by instrumented reaction pin can be used for the crack length measurement of T-WOL specimen during SCC experiment.

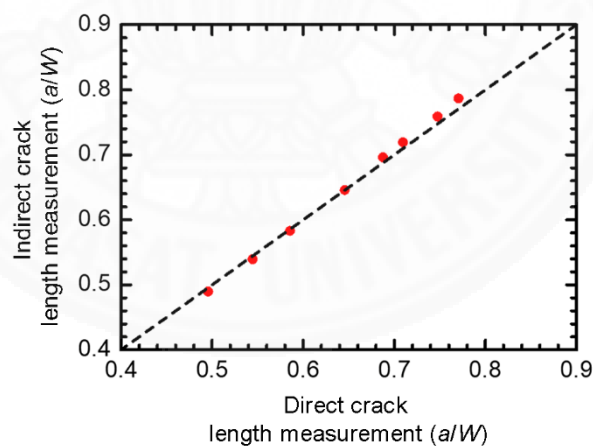


Fig. 4-11 Relationships between direct crack length measurement and indirect crack length measurement of T-WOL specimens with various a/W .

4.4 Applicability of the indirect crack length method

Previously, ASTM E1681 [16] and Vigilante et.al. [48] suggested the indirect crack length measurement using the similar expression (i.e., Eq. 4-5). The empirical equation from ASTM E1681 [16] is

$$a/W = 1 - 3.19Z - 4.66Z^2 + 32.03Z^3. \quad (4-6)$$

While, the empirical equation from Vigilante et.al. [48] (i.e., using an instrumented loading bolt to measure crack-opening force) is

$$a/W = 1 - 3.91Z - 4.77Z^2 + 32.03Z^3. \quad (4-7)$$

On the other hand, Chung et.al. [47] estimated the crack length from the measured crack-opening force (i.e., from an instrumented loading bolt) and measured CMOD using the different expression, i.e.,

$$36.845(a/W)^4 - 64.677(a/W)^3 + 42.314(a/W)^2 - 8.097(a/W) + 3.453 = \ln(EBV/F). \quad (4-8)$$

To investigate the applicability of the available indirect crack length method, the crack lengths measured from direct crack length measurement (i.e., using a digital microscope) are compared with those measured from indirect measurement; (i) the present method, (ii) ASTM E1681 method [16], (iii) Vigilante et.al. method [48], and (iv) Chung et.al. method [47], as listed in Table 4-1. The differences between crack lengths ($0.496 \leq a/W \leq 0.771$) from the direct crack length measurement and the present indirect crack length measurement are in the range of -1.4% to 1.9%, which are lower than those of ASTM E1681 method [16], Vigilante et.al. method [48], and Chung et.al. method [47]. The differences between crack lengths from the direct crack length measurement and the indirect crack length measurement using ASTM E1681 method [16] are significantly large, i.e., in the range of 7.1% to 21.2%. Similar finding also reported by Nibur et al. [65], who investigated the threshold stress intensity factors for steels in high-pressure hydrogen gas. Based on their physical measurements of crack length, they found that the relationship between crack length and force (i.e., at fixed

crack-mouth opening displacement) provided in ASTM E1681 [16] was inaccurate. The error was typically 25% or greater. Because the present crack length measurement is validated; it is expected to be applied for the determination of crack length of T-WOL specimen during SCC experiment.

4.5 Conclusion

The indirect crack length measurement technique using an instrumented reaction pin was developed to accurately measure the crack length of a T-WOL specimen. Using the finite element method, the instrumented reaction pin was designed, and its ability of crack-opening force measurement was checked and calibrated using an electric fatigue machine. Based on the elastic-plastic behaviour of a T-WOL specimen, the measured crack-opening force as well as the measured CMOD were used as the input for the numerical calculation of crack length. The crack lengths measured by the present indirect crack length measurement were in good agreement with the direct crack length measurement using a digital microscope. Because the present crack length measurement is validated; it is expected to be applied for the determination of crack length of T-WOL specimen during SCC experiment of engineering alloy.

Table 4-1 Measurements of crack length on T-WOL specimens.

Measurement method	Crack length (a/W)															
	a/W	% Diff.	a/W	% Diff.	a/W	% Diff.	a/W	% Diff.	a/W	% Diff.	a/W	% Diff.	a/W	% Diff.	a/W	% Diff.
Direct measurement	0.496	-	0.545	-	0.586	-	0.646	-	0.688	-	0.710	-	0.748	-	0.771	-
ASTM E1681 [16]	0.601	21.17	0.637	16.88	0.668	12.27	0.714	10.53	0.752	9.30	0.772	8.73	0.804	7.49	0.826	7.13
Vigilante et.al. [48]	0.512	3.23	0.557	2.20	0.595	1.51	0.652	0.93	0.699	1.60	0.722	1.69	0.761	1.74	0.789	2.33
Chung et.al. [47]	0.504	1.61	0.553	1.47	0.596	1.71	0.657	1.70	0.705	2.47	0.728	2.53	0.764	2.14	0.789	2.33
Present work	0.489	-1.41	0.539	-1.10	0.582	-0.68	0.645	-0.15	0.696	1.16	0.719	1.27	0.758	1.34	0.786	1.95

Chapter 5

Stress corrosion cracking under plane strain condition: crack-tip opening displacement

Crack-tip opening displacement (CTOD) can be directly measured on side surface of a specimen at 2.5-mm above and below a crack tip, i.e., CTOD δ_5 method. Unfortunately, CTOD δ_5 cannot be accurately related to the damage of protective layer at crack tip because it does not represent the actual CTOD. As an alternative fracture mechanics parameter, CTOD 45° (i.e., a distance between two intersections of $\pm 45^\circ$ lines from the crack tip with crack surfaces) was therefore purposed for the evaluation of SCC. In the present work, CTOD δ_5 and CTOD 45° of T-WOL specimen with a built-in loading bolt was firstly calculated from the crack-opening force, load-line displacement, crack length, specimen geometry and material properties using three-dimensional elastic-plastic finite element analysis (FEA). Under ambient environment, CTOD δ_5 from experiment and FEA were compared for FEA validation. Validated FEA was used for the calculation of CTOD 45° . Influence of side grooves on CTOD 45° was evaluated and discussed. Suitable method for CTOD 45° determination was proposed. The finding can be applied for the determination of CTOD 45° of T-WOL specimen to characterize da/dt during plane-strain SCC experiment of engineering alloy. Detail of the procedure to determine CTOD are given as follows.

5.1 T-type wedge opening loaded specimen with side grooves

Under ambient environment, CTOD δ_5 of a T-WOL specimen was measured during experiment, and used for FEA validation. Material used for a T-WOL specimen was a cast nickel aluminium bronze (NAB), which was supplied by the Royal Thai Navy. Mechanical properties are summarized in Table 2-2. T-WOL specimen with a chevron notch was machined from NAB using an electrical discharge machine (EDM). Geometry and dimension of T-WOL specimen (Fig. 5-1) was in accordance with the ISO 7539-6 [7]. Because the corrosive environment is likely to attack at the location of the maximum shear stress at $\pm 45^\circ$ to the applied force (i.e., the region of high dislocation density) and at the grain boundaries; the crack branching may occur during SCC

experiment. According to ISO 7539-6 [7], the side grooves was introduced on the side surfaces of T-WOL specimen (Fig. 5-1) to constrain the crack branching.

Pre-crack of T-WOL specimen was created by a fatigue machine (Instron: ElectroPuls E10000 with 10-kN load cell) under an air environment, $R = 0.1$ stress ratio, and 20-Hz frequency. During pre-cracking, a built-in loading bolt was attached to upper clevis grip of fatigue machine, while the lower half of T-WOL specimen was attached to lower clevis grip of fatigue machine (Fig. 5-2). Crack length (a) was measured using an optical microscope with a resolution of 10 μm . Stress intensity factor (K) at crack tip of T-WOL specimen can be determined from Eq. 1-1 and 1-2, where W is the width of specimen (51 mm), B_n is the net thickness of specimen (20 mm), a is the average length of cracks on both sides of specimen ($0.3 \leq a/W \leq 0.8$), and F is the applied force.

It is known that the crack has a curved crack front during fatigue crack growth experiment, and CTOD at mid-thickness plane is slightly different from those at side surfaces [66]. However, the determination of crack front curvature from fracture surface after SCC experiment is not practical and accurate because of the presence corrosion product on fracture surface. As recommended by ISO 7539-6 [7], the crack is assumed to be a straight crack front, and the crack length (a) is the average of crack lengths on both sides of T-WOL specimen.

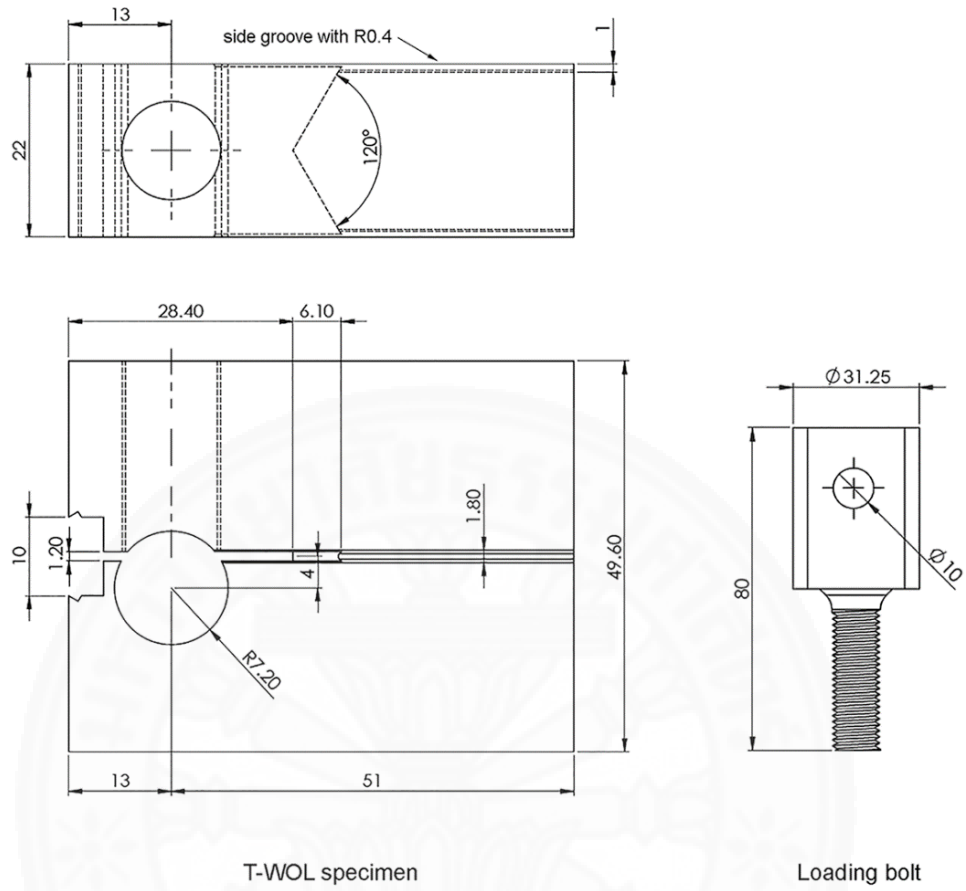


Fig. 5-1 Geometries of T-WOL specimen and loading bolt (dimension in mm).

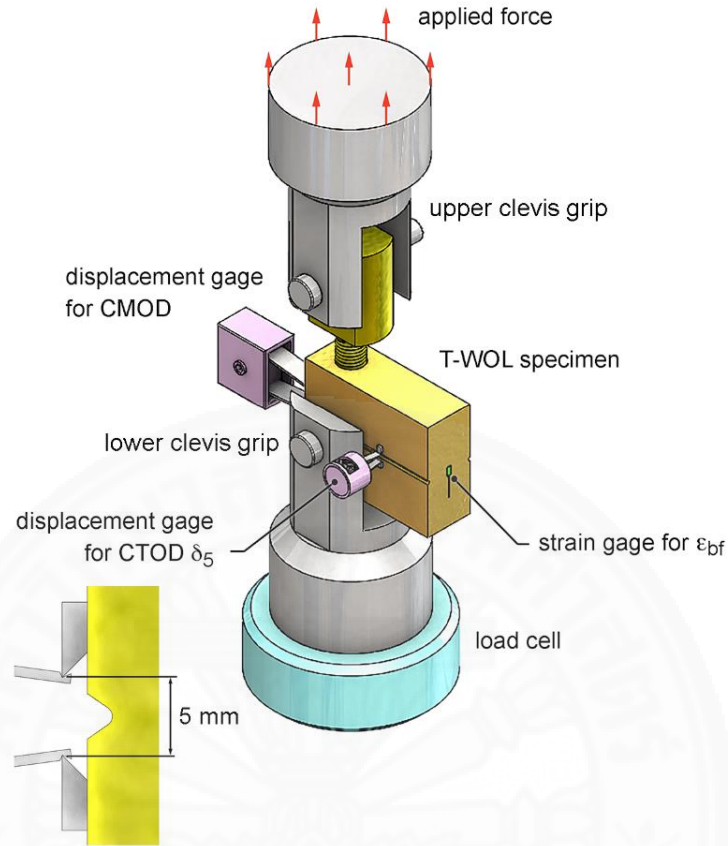


Fig. 5-2 Schematic drawing of the pre-cracking experiment, and the measurements of back-face strain (ϵ_{bf}), crack-mouth opening displacement (CMOD), and crack-tip opening displacement (CTOD δ_5) on T-WOL specimen.

During the pre-cracking, the size of the cyclic plastic zone (r_c) at crack tip of T-WOL specimen was maintained to be smaller than its thickness, crack length, and remaining ligament. The r_c can be estimated as follows [6]:

$$r_c(\theta) = \frac{\Delta K^2}{16\pi\sigma_Y^2} \left(\frac{3}{2} \sin^2 \theta + (1 - 2\nu)^2 (1 + \cos \theta) \right), \quad (5-1)$$

where ΔK is the stress intensity factor range, σ_Y is the yield stress, θ is the polar coordinate at crack tip, and ν is Poisson's ratio ($\nu = 0.3$). The final stage of pre-cracking ($a/W = 0.47$) was performed with $\Delta K = 11 \text{ MPa}\cdot\text{m}^{1/2}$; therefore, the maximum r_c at $\theta = \pi/2$ was $87 \text{ }\mu\text{m}$. This cyclic plastic zone size is significantly smaller than the thickness,

crack length, and remaining ligament of T-WOL specimen. Thus, the crack tip is under the condition of small-scale yielding.

5.2 Measurements of back-face strain, CMOD, and CTOD

In the present work, the K_I , back-face strain (ε_{bf}), CMOD, CTOD δ_5 , and CTOD 45° of T-WOL specimen were numerically determined from the geometry of specimen, crack length, mechanical properties, and applied force. To validate the numerical calculation, the tensile force was applied to T-WOL specimen under ambient environment using a fatigue machine (Instron: ElectroPuls E10000 with 10-kN load cell); then ε_{bf} , CMOD and CTOD δ_5 were measured (Fig. 5-2), while K_I was also calculated using Eq. 1-1 and 1-2 (i.e., ISO 7539-6 [7]). Based on the fracture toughness of NAB ($K_{IC} = 25 \text{ MPa}\cdot\text{m}^{1/2}$) and the crack length of T-WOL specimen ($a/W = 0.47$), the limit force (F_L) corresponding to a fracture of T-WOL specimen is approximately 11,800 N. During experiment, the tensile force (F) was applied with the loading rate of 100 N/min to the maximum force of 5,000 N (i.e., F/F_L is approximately 0.43). Results from FEA and experiment were compared for FEA validation. Validated FEA will be used for the calculation of CTOD 45° .

As the strain at mid-height location on back face of T-WOL specimen, ε_{bf} was measured using a strain gage with 1-mm gage length (TML: FLA-1-17), while CMOD and CTOD δ_5 were measured using the double-cantilever clip-in displacement gages. Because the knife edges cannot be machined on T-WOL specimen, two small knife edges were attached at 2.5-mm above and below a crack tip of T-WOL specimen, as shown in Fig. 5-2. Both double-cantilever clip-in displacement gages for the measurements of CMOD and CTOD δ_5 were designed, constructed, and calibrated in accordance with ASTM E399 [67].

5.3 Finite element analysis for CTOD

5.3.1 Modelling

Three-dimensional FEA, i.e., ABAQUS [51], was used to determine the plastic zone, K_I , ε_{bf} , CMOD, CTOD δ_5 , and CTOD 45° of T-WOL specimen. Because T-WOL specimen is symmetry on mid-thickness plane, FEA was performed using half of T-WOL specimen. Mechanical properties of T-WOL specimen are assumed to be homogeneous and isotropic, as shown in Table 2-2. For the calculation of plastic zone and K_I , the linear-elastic behaviour was used as a constitutive equation. On the other hand, the elastic-plastic behaviour was used as a constitutive equation for the calculations of ε_{bf} , CMOD, and CTOD δ_5 , and CTOD 45° .

Because the CTOD 45° is defined as a distance between two intersections of $\pm 45^\circ$ lines from the crack tip with crack surfaces (Fig. 5-3); CTOD 45° is extremely small. Therefore, the size of elements around crack front and groove tip should be significantly small to obtain the accurate CTOD 45° . To reduce the calculation time while maintain the accurate result, CTOD 45° of T-WOL specimen was numerically investigated using sub-modeling technique. The boundary that cut through the model with coarse elements (i.e., global model) is defined as boundary of the model with fine elements (i.e., local model). Displacements on the cut boundary of global model are used as the applied mechanical action for local model. In the present investigation, T-WOL specimen was used as a global model (Fig. 5-4a), while the cylindrical volume around crack front of T-WOL specimen was used as a local model (Fig. 5-4b).

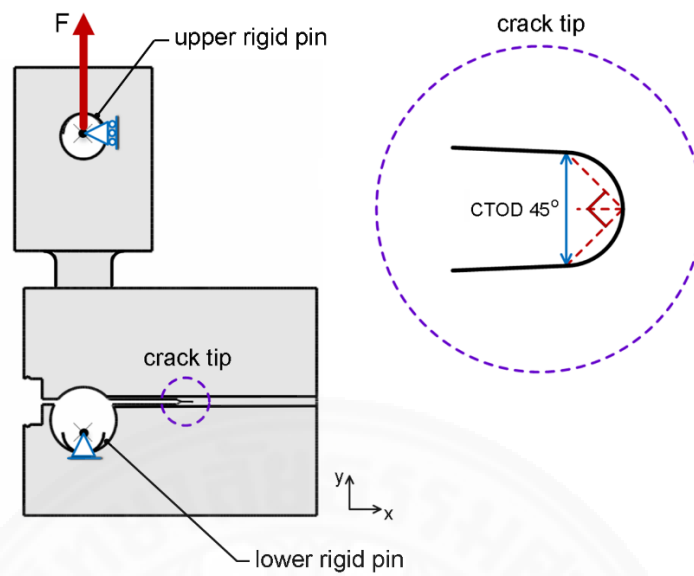


Fig. 5-3 Schematic drawing of CTOD 45° , and boundary conditions of FEA.

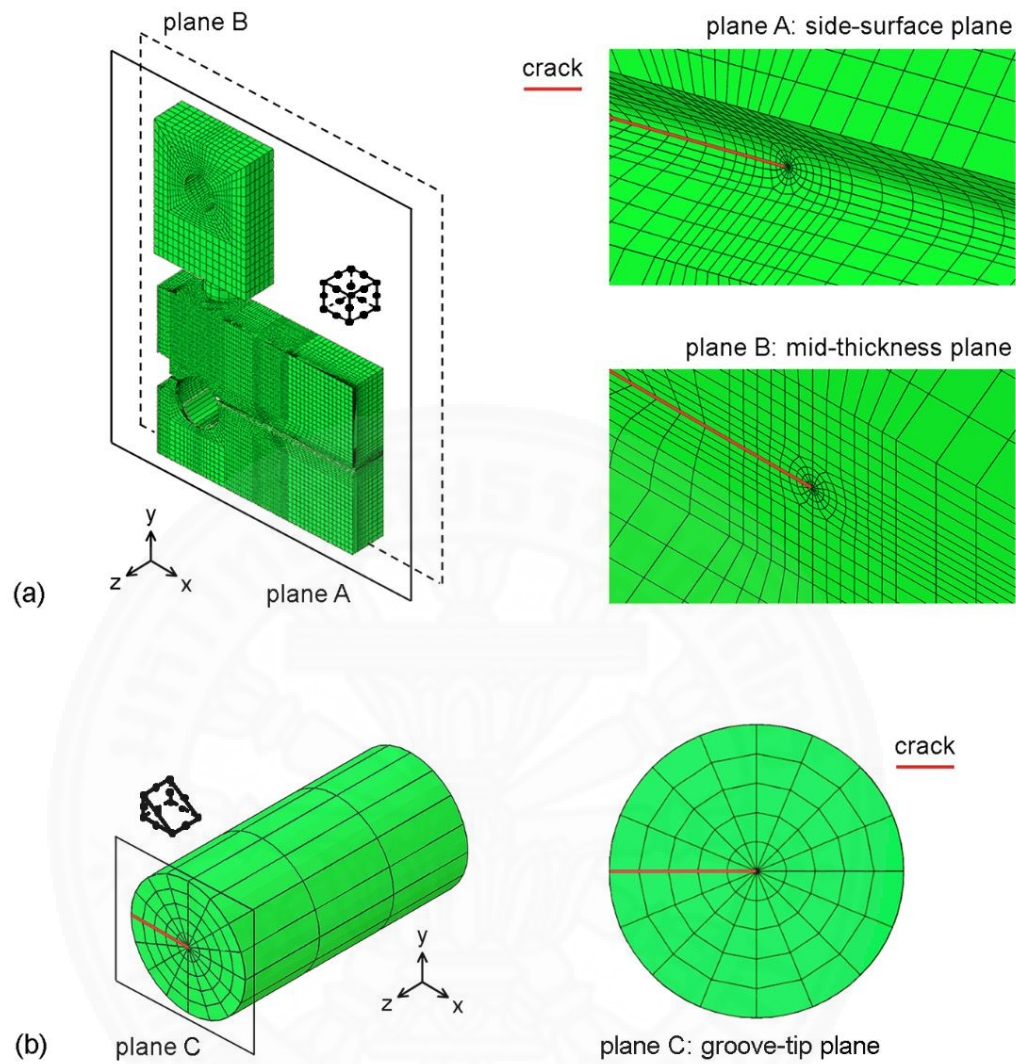


Fig. 5-4 FE model of T-WOL specimen: (a) global model, and (b) local model.

Straight crack front is assumed for FEA. The quarter-point singular elements were used around crack front, while the quadratic hexahedral elements were used elsewhere (Fig. 5-4). To provide the deformation around crack front, the node movement of elements on crack front was allowed during FEA. Frictional contact, master-slave algorithm, and penalty method [51] were applied to the contact. The slip of contact is assumed to occur when the shear stress is greater than the critical shear stress (τ_c), i.e., Eq. 4-3. For the contact between NAB components, the friction coefficient is 0.50 [64].

During FEA, the tensile force was applied at the center of upper rigid pin (which is inserted into the hole of loading bolt), while the center of lower rigid pin (which is inserted into the hole of T-WOL specimen) was fixed. Boundary conditions were applied on T-WOL specimen with a loading bolt, as shown in Fig. 5-3. Rotation around z -axis was allowed at both upper and lower rigid pins. On the lower rigid pin, the displacements in all directions were under constraint. While, the displacements in x and z directions were under constraint on the upper rigid pin, i.e., only displacement in y direction was possible. Applied force (F) was varied with intervals of 100 steps from zero to 5,000 N (i.e., $F/F_L = 0.43$). The dependence of FEA results on element size was minimized by varying element size until the von Mises stress around crack front became stable. Global model has 105,900 elements and 455,300 nodes, and the sizes of radial, angular, though-thickness elements along crack front are in the range of $5 \times 10^{-5} W$ to $10^{-3} W$. On the other hand, the local model has 102,400 elements and 493,000 nodes, and the sizes of radial, angular, though-thickness elements along crack front are in the range of $10^{-6} W$ to $5 \times 10^{-5} W$.

5.3.2 Validation

To validate the numerical calculation, ε_{bf} and CMOD of T-WOL specimen ($a/W = 0.47$) in a function of applied force (F/F_L) calculated from FEA were compared to those measured during experiment, as shown in Fig. 5-5a and 5-5b, respectively. It is noted that the engineering strains at back face of T-WOL specimen measured from experiment were converted to true strains before the comparison with those from FEA. Experiments were repeated five times, and the variations of ε_{bf} and CMOD are lower

than 3%. At an arbitrary crack length, ε_{bf} decreases with applied force, while CMOD increases with applied force. As observed in the figure, the calculated ε_{bf} and CMOD are in good agreement with the experimental results, thus the present FEA is validated.

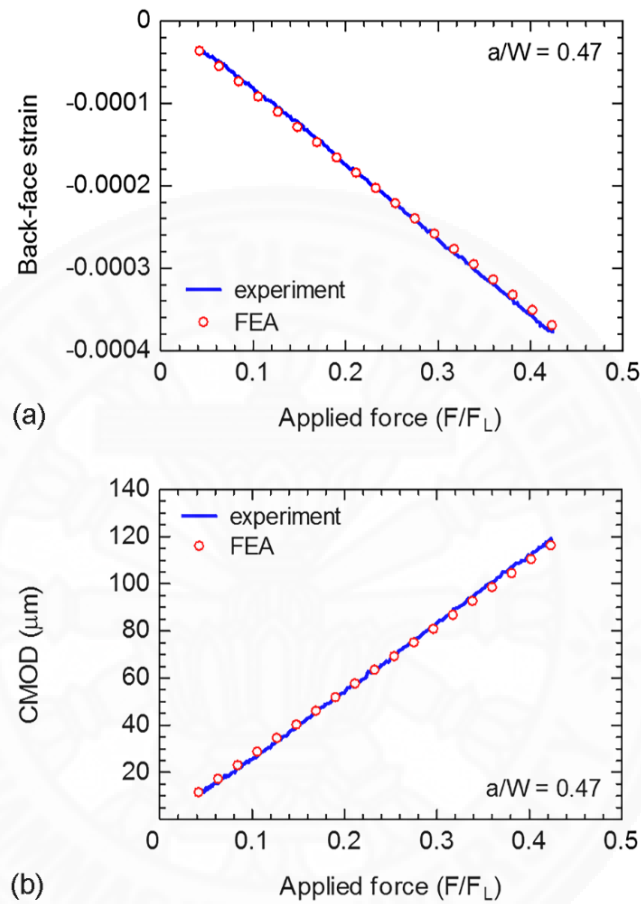


Fig. 5-5 (a) Back-face strain in a function of applied force, and (b) Crack-mouth opening displacement (CMOD) in a function of applied force.

5.4 Plastic zone and stress intensity factor

Distributions of von Mises stress on the plane perpendicular to thickness of T-WOL specimen ($a/W = 0.47$) under $F/F_L = 0.43$ are shown in Fig. 5-6. Plastic zone is the area where von Mises stress is higher than yield stress (i.e., grey area in Fig. 5-6). With the introduction of side grooves on T-WOL specimen, the stresses around crack tip at groove-tip plane is higher than those at mid-thickness plane. Therefore, the larger plastic zone at groove-tip plane ($z/B_n = 0.5$) is expected. The influence of side groove decreases with the distance from groove tip, i.e., the plastic zone size at 500 μm from groove-tip plane ($z/B_n = 0.475$) is almost identical to that at mid-thickness plane ($z/B_n = 0$). On the middle part of T-WOL specimen ($-0.475 < z/B_n < 0.475$), the plastic zone sizes along crack front are smaller than the thickness, crack length and remaining ligament, i.e., a small-scale yielding condition. Accordingly, the stress intensity factor (K_I) can be applied to characterize the severity at crack front of T-WOL specimen.

With the introduction of side grooves on T-WOL specimen, the state of stress around crack tip near groove-tip plane is complex than that on mid-thickness plane. K_I along crack front of T-WOL specimen ($a/W = 0.47$) under an applied force (F/F_L) of 0.43 are shown in Fig. 5-7a. K_I is small on groove-tip planes ($z/B_n = \pm 0.5$), increases with the distance from groove-tip plane, and becomes the highest at $z/B_n = \pm 0.498$. Subsequently, it decreases with the distance from groove-tip plane, and eventually becomes stable at $-0.45 < z/B_n < 0.45$. For sharp groove, the Hutchinson, Rice and Rosengren (HRR) singularity near the groove tip is expected, i.e., K_I at the tip of sharp groove is significantly large, and decrease with the distance from tip of sharp groove. However, with the introduction of groove-tip radius, the stress and K_I near the tip of groove deviate from HRR singularity [68], i.e., their maximum values are not at the groove-tip plane but slightly moves away from groove-tip plane. Similar effect of side groove on K_I was reported by Sobotka and Dodds [69].

Although the high and complex K_I is observed near side groove; the variation in K_I along crack front of T-WOL specimen becomes marginal at $-0.45 < z/B_n < 0.45$. K_I at mid-thickness plane of T-WOL specimen ($a/W = 0.47$ and $z/B_n = 0$) in a function of applied force (F/F_L) calculated from FEA were compared to those obtained from ISO

7539-6 [7], as shown in Fig. 5-7b. K_I linearly increases with applied force, and K_I from FEA and ISO 7539-6 are in good agreement.

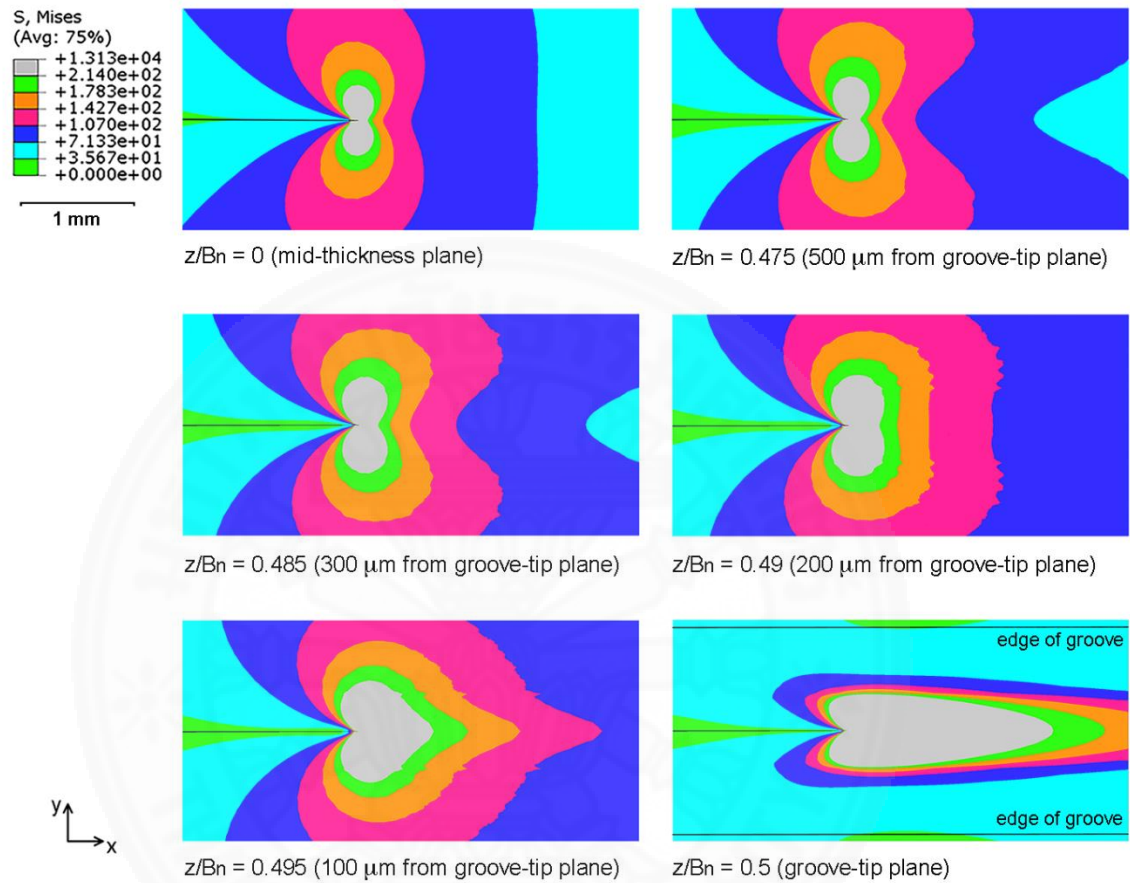


Fig. 5-6 Plastic zones along crack front of T-WOL specimen ($a/W = 0.47$) under an applied force ($F/F_L = 0.43$).

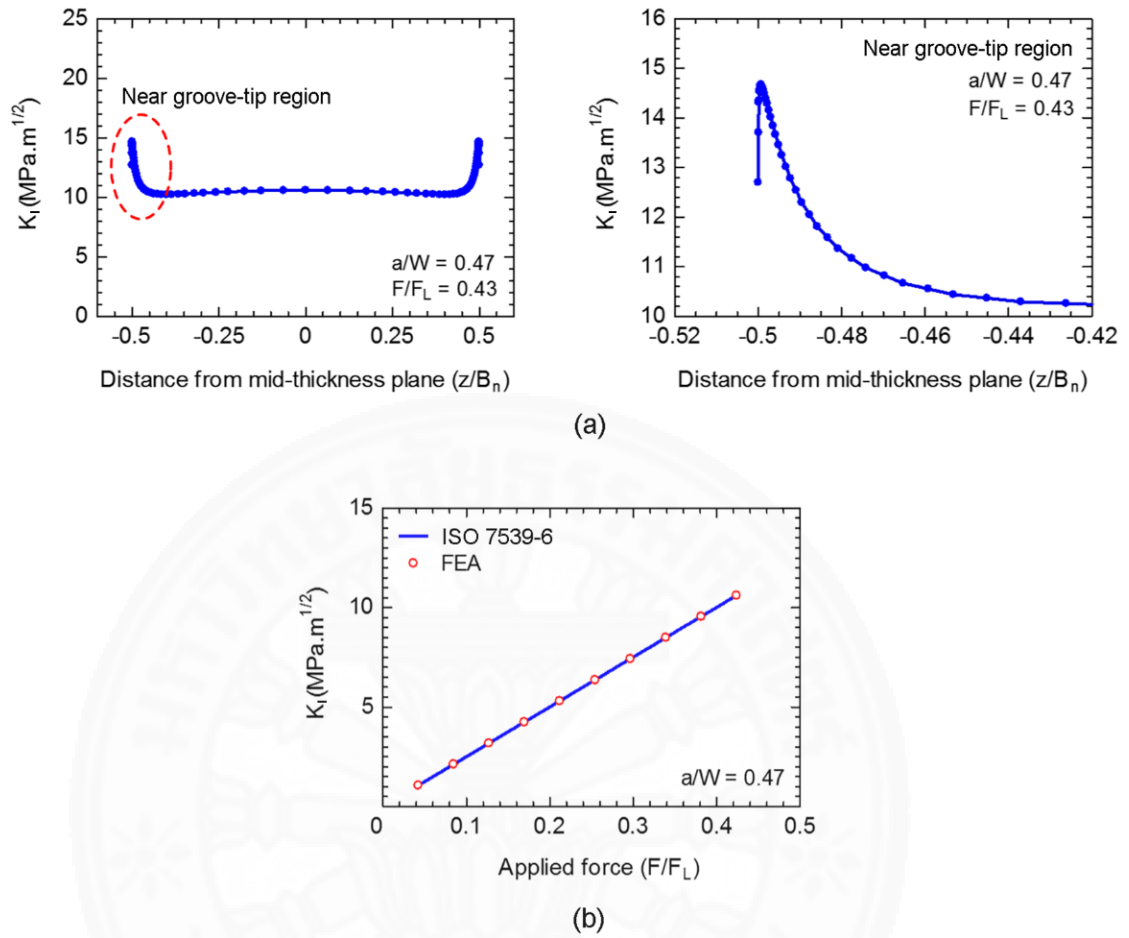


Fig. 5-7 (a) Stress intensity factor (K_I) along crack front of T-WOL specimen, and (b) Stress intensity factor (K_I) at mid-thickness plane of T-WOL specimen in a function of applied force (F/F_L).

5.5 Crack-tip opening displacement

On side surface of T-WOL specimen ($a/W = 0.47$ and $z/B_n = 0.55$), the crack-tip opening displacements at 2.5-mm above and below a crack front (i.e., CTOD δ_5) in a function of applied CMOD were numerically calculated and compared to those obtained from experiment, as shown in Fig. 5-8. Experiments were repeated five times, and the variation of CTOD δ_5 is lower than 5%. CTOD δ_5 on side surface linearly increases with CMOD, and CTOD δ_5 from FEA and experiment are in good agreement. Therefore, the FEA for CTOD δ_5 is validated, and the present FEA can be used for the calculation of CTOD 45° .

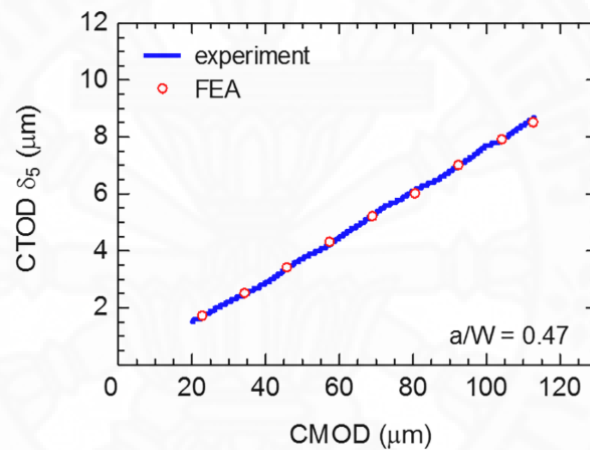


Fig. 5-8 CTOD δ_5 on side surface of T-WOL specimen in a function of applied CMOD.

As a distance between two intersections of $\pm 45^\circ$ lines from the crack tip with crack surfaces, CTOD 45° represents the severity at crack tip under corrosive environment, i.e., large CTOD 45° means more attack from corrosive environment at crack tip. Thus, CTOD 45° should be applied as an alternative parameter to characterize da/dt during SCC experiment. Unfortunately, CTOD 45° of T-WOL specimen is very small, and cannot be accurately measured, especially at groove tip of T-WOL specimen. Accordingly, CTOD 45° of T-WOL specimen with a built-in loading bolt was calculated using FEA. As examples, CTOD 45° of T-WOL specimen ($a/W = 0.47$) under $F/F_L = 0.41$ on groove-tip plane and mid-thickness plane are shown in Fig. 5-9. On the other hand, the variation of CTOD 45° along the crack front of T-WOL specimen (a/W

= 0.47) under $F/F_L = 0.41$ is shown in Fig. 5-10a. It is found that the variation of CTOD 45° along the crack front of T-WOL specimen is similar to that of K_I (Fig. 5-7a). CTOD 45° is small on groove-tip planes ($z/B_n = \pm 0.5$), increase with the distance from groove-tip plane, and becomes the highest at $z/B_n = \pm 0.498$. Then, CTOD 45° decreases with the distance from groove-tip plane, and eventually becomes stable at $-0.45 < z/B_n < 0.45$.

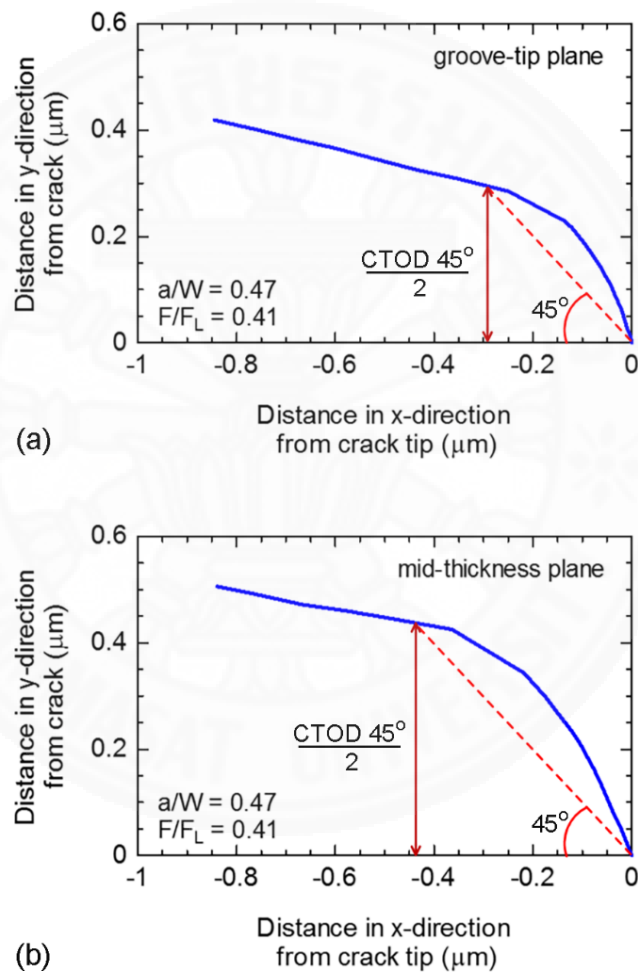


Fig. 5-9 CTOD 45° of T-WOL specimen ($a/W = 0.47$) under $F/F_L = 0.41$,
(a) groove-tip plane, and (b) mid-thickness plane.

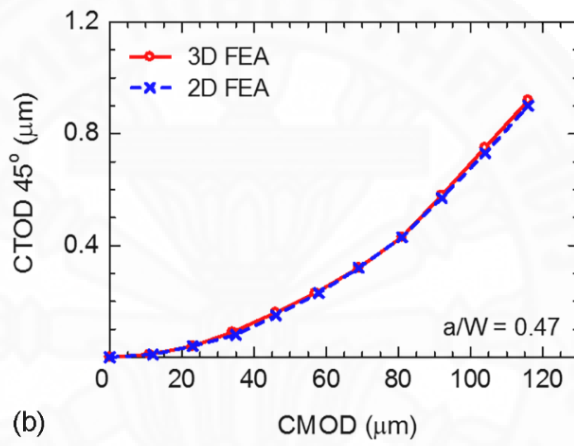
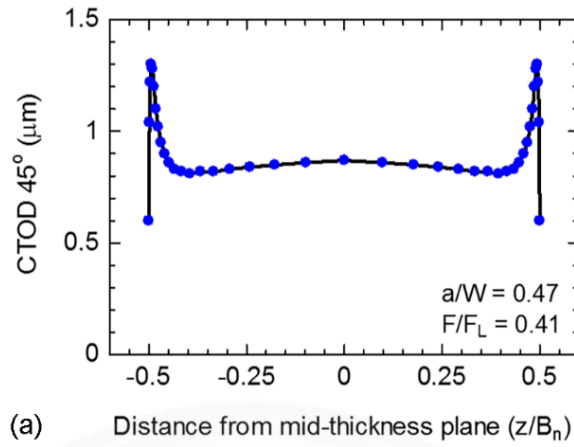


Fig. 5-10 (a) Variation of CTOD 45° along the crack front of T-WOL specimen ($a/W = 0.47$) under $F/F_L = 0.41$, and (b) CTOD 45° on mid-thickness plane of T-WOL specimen ($a/W = 0.47$) in a function of applied CMOD.

In the previous work, Wang et al. [70] numerically investigated the influence of sharp groove on CTOD of single-edge tension (SE(T)) specimen. Although their side grooves have a finite tip radius; the side groove was modelled as a sharp V-notch with an opening angle of 45° to simplify the mesh configuration of numerical model. They found that, for SE(T) specimen, CTOD 45° significantly increases in the region close to the tip of side groove. Their finding is analogical to the Hutchinson, Rice and Rosengren (HRR) singularity near the tip of sharp crack. However, to accurately determine CTOD 45° of T-WOL specimen, the tip radius was modelled in the present FEA. With groove-tip radius, the stress near groove-tip plane deviates from HRR singularity [68], therefore the highest CTOD 45° is not at the groove-tip plane but slightly moves away from groove-tip plane (Fig. 5-10a). Although, the measured CTOD 45° at groove-tip plane may become less accurate because of the additional stress concentration introduced by side groove. It is found that CTOD 45° is still valid at the region slightly away from the groove-tip plane, i.e., the variation of CTOD 45° along $> 90\%$ of crack front is less than 5% (Fig. 5-10a). Accordingly, it is justified to apply CTOD 45° at mid-thickness plane of T-WOL specimen to characterize da/dt during SCC experiment.

However, the accurate evaluation of CTOD 45° using elastic-plastic 3D FEA requires long calculation time. Calculation time may be reduced, when the plane-strain elastic-plastic 2D FEA (i.e., 4,650 elements and 14,540 nodes) is applied for the evaluation of CTOD 45° at mid-thickness plane of T-WOL specimen. Thus, CTOD 45° on mid-thickness plane of T-WOL specimen ($a/W = 0.47$ and $z/B_n = 0$) in a function of CMOD was numerically calculated using elastic-plastic 3D FEA, and compared to those obtained from plane-strain elastic-plastic 2D FEA, as shown in Fig. 5-10b. CTOD 45° increases with CMOD, and CTOD 45° from 3D FEA and 2D FEA are in good agreement. With shorter calculation time, 2D FEA is recommended for the determination of CTOD 45° of T-WOL specimen with side groove during SCC experiment.

For T-WOL specimen with the present geometry and material, CTOD 45° at mid-thickness plane in a function of CMOD at various a/W ($0.4 < a/W < 0.8$) are shown in Fig. 5-11. Numerical calculation was performed until F/F_L becomes nearly 1. Because of the plastic deformation around crack front, the nonlinearity relationship between CTOD 45° and CMOD can be observed. During SCC experiment, the measured CMOD

and crack length (a/W) are used for the determination of CTOD 45° . Therefore, the relationship between CTOD 45° and CMOD at various a/W (Fig. 5-11) can be applied for the determination of CTOD 45° at mid-thickness plane of T-WOL specimen.

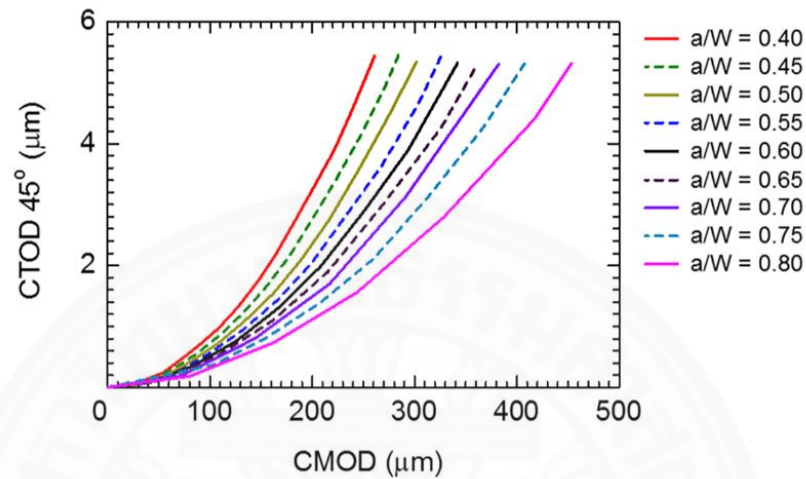


Fig. 5-11 CTOD 45° at mid-thickness plane in a function of CMOD at various a/W ($0.4 < a/W < 0.8$).

Although the numerical method for the determination of CTOD 45° of T-WOL specimen was preliminarily developed, and expected to be applied for the characterization of da/dt during SCC experiment of engineering alloy. Some improvements should be taken into consideration for more accurate result in the future work. Firstly, the direct measurement technique, which can accurately measure CTOD 45° in the order of 0.5 to 1.5 μm , should be developed, and used for the validation of numerical CTOD 45° on groove tip of T-WOL specimen. Secondly, the fine elements (element size $< 1 \mu\text{m}$) were applied along crack front to numerically calculate CTOD 45° , while the mechanical properties were assumed to be homogeneous and isotropic. It is known that the localized mechanical properties of alloy are not homogeneous and isotropic, but depend on the microscopic characteristics, such as crystal direction, alloying elements, dislocation density, grain boundary. Thus, more accurate CTOD 45° may be achieved if the localized mechanical properties can be determined and used for multi-scale modeling. Lastly, the crack was assumed to be a straight crack front for the numerical calculation of CTOD 45° ,

however the actual crack has a curved crack front. Unfortunately, the determination of crack front curvature from fracture surface after SCC experiment is not practical and accurate because of the presence corrosion product on fracture surface. If the shape of crack front can be accurately measured, and introduced into FE model; it is believed the accuracy of numerical calculation of CTOD 45° should be improved.



5.6 Conclusion

As an elastic-plastic fracture mechanics parameters for stress corrosion cracking (SCC), the crack-tip opening displacement (CTOD) of a T-type wedge opening loaded (T-WOL) specimen with a built-in loading bolt was firstly calculated using finite element analysis (FEA). Influence of side grooves on CTOD 45° was evaluated and discussed. The results are summarized as follows.

- Crack-tip opening displacements at 2.5-mm above and below a crack front (i.e., CTOD δ_5) in a function of crack-mouth opening displacement (CMOD) were numerically calculated using elastic-plastic FEA, and compared to those obtained from experiment. Both FEA and experimental results were in good agreement. Thus, the validation of FEA was achieved.
- As a distance between two intersections of $\pm 45^\circ$ lines from the crack tip with crack surfaces, CTOD 45° method could be numerically calculated from force, crack length, specimen geometry, and material properties. Because of the additional stress concentration introduced by side groove, CTOD 45° was small on groove-tip plane, increased with the distance from groove-tip plane, and became the highest. Then, it decreased with the distance from groove-tip plane, and eventually became stable. The stable CTOD 45° occurred on the majority (> 90%) of thickness of T-WOL specimen.
- CTOD 45° was less accurate at groove-tip plane; however, CTOD 45° at mid-thickness plane was still valid, and could be applied for SCC experiment. To reduce the calculation time for CTOD 45° , the plane-strain elastic-plastic 2D FEA could be applied for the evaluation of CTOD 45° at mid-thickness plane of T-WOL specimen, i.e., CTOD 45° from 3D FEA and 2D FEA are in good agreement.

Chapter 6

Summary and future work

6.1 Stress corrosion cracking under plane stress condition

To understand SCC of NAB under plane stress condition; SCC experiments on NABs under laboratory air, artificial seawater, and ammonia-containing artificial seawater were conducted using a 4-point bending technique. The stress/strain state near the crack tip and the J -integral were numerically calculated using finite element analysis (FEA). The J -integrals were correlated with the crack growth rates (da/dt) from the SCC experiments. Together with the analysis of crack propagation path and fracture surfaces, the SCC behaviour and mechanism are discussed.

SCC was possible for NABs under both artificial seawater and ammonia-containing artificial seawater. Under a high concentration of ammonium hydroxide (5:1 / SW:AH environment), the threshold J -integral for susceptibility to SCC (J_{SCC}) and fracture toughness (J_C) were the lowest, followed by those under the 10:1 / SW:AH and SW environments. The present findings gave some guidelines for practitioners to, e.g., avoid SCC by maintaining J below J_{SCC} , and predict the crack growth rate (da/dt) and the J for fast fracture (J_C) using SCCG curve.

For NABs under artificial seawater and ammonia-containing artificial seawater, the SCC process occurred mainly in the copper-rich solid solution phase (α -phase). Both chloride and ammonium were the main causes of the SCC process for NABs under the ammonia-containing artificial seawater environments. In contrast, chloride was the main cause of the SCC process for NAB under the artificial seawater environment. The fracture surfaces of for NABs under the ammonia-containing artificial seawater environments showed the tunnel-like cracks, which propagated in the maximum shear stress direction through the α -phase and lamellar κ_{III} -phase and along the interface between the α -phase and globular κ_I -phase. Crack arrest was observed at the lamellar κ_{III} -phases before the crack changed its path into another maximum shear stress direction. Although crack growth was possible for NAB under artificial seawater, there was no clear evidence of a corrosion process on its fracture surface, i.e., the fracture surface exhibited a ductile fracture appearance.

6.2 Experimental procedure for stress corrosion cracking under plane strain condition

Under plane strain condition, a self-deformed specimen (i.e., T-WOL specimen with built-in loading bolt) is recommended for SCC experiment under constant applied displacement. During SCC experiment, the force decreased with the extension of crack under corrosive environment. If the crack-opening force and CMOD of a T-WOL specimen can be monitored; the crack length can be numerically calculated using FEA. Therefore, the indirect crack length measurement technique using instrumented reaction pin (i.e., a support of the loading bolt) was developed to measure the crack length of a T-WOL specimen.

Using an instrumented reaction pin, the measured crack lengths of T-WOL specimens under ambient environment were compared with those measured using the visual inspection. The crack lengths measured by the present indirect crack length measurement (i.e., an instrumented reaction pin) were in good agreement with the direct crack length measurement using a digital microscope. Because the present crack length measurement is validated; it is expected to be applied for the determination of crack length of T-WOL specimen during SCC experiment of engineering alloy.

Because the protective layer at crack tip can be damaged and loses its corrosion resistance under the combination between crack-tip opening and corrosive environment; CTOD should be applied to characterize SCCG. Thus, CTOD δ_5 and CTOD 45° of T-WOL specimen with a built-in loading bolt was firstly calculated from the crack-opening force, load-line displacement, crack length, specimen geometry and material properties using 3D FEA. Under ambient environment, CTOD δ_5 from experiment and FEA were compared for FEA validation. Validated FEA was used for the calculation of CTOD 45° . Influence of side grooves on CTOD 45° was evaluated and discussed.

As a distance between two intersections of $\pm 45^\circ$ lines from the crack tip with crack surfaces, CTOD 45° method could be numerically calculated from force, crack length, specimen geometry, and material properties. Because of the additional stress concentration introduced by side groove, CTOD 45° was small on groove-tip plane, increased with the distance from groove-tip plane, and became the highest. Then, it

deceased with the distance from groove-tip plane, and eventually became stable. The stable CTOD 45° occurred on the majority ($> 90\%$) of thickness of T-WOL specimen. CTOD 45° was less accurate at groove-tip plane; however, CTOD 45° at mid-thickness plane was still valid, and could be applied for SCC experiment. To reduce the calculation time for CTOD 45° , the plane-strain elastic-plastic 2D FEA could be applied for the evaluation of CTOD 45° at mid-thickness plane of T-WOL specimen, i.e., CTOD 45° from 3D FEA and 2D FEA are in good agreement. The finding can be applied for the determination of CTOD 45° of T-WOL specimen to characterize da/dt during SCC experiment of engineering alloy.

6.3 Guidelines to minimize SCC

Based on the present work, it is suggested that SCC of NAB components can be minimized; if the combination between the concentration of ammonia-containing seawater and the stress is controlled. For a thin component, the J -integral at the critical location of component should be determined, and compared with the J_{SCC} at service environment. SCC is unlikely to occur; if J -integral $< J_{SCC}$. On the other hand, if J -integral $> J_{SCC}$; SCC is possible. The stress corrosion crack growth (SCCG) curve can be applied for the life prediction as well as the maintenance planning. The J_{SCC} and SCCG curve given here can be applied for SCC of NAB under SW to 5:1 SW:AH environment. However, more experiments are required for SCC behaviours of NAB under higher concentration of ammonia-containing seawater.

For a thick component, the experimental procedure for the determination of K_{ISCC} and SCCG curve using T-WOL specimen is proposed here. The engineers and researchers can use this procedure to obtain K_{ISCC} and SCCG curve under their service environments. Then K at the critical location of component is determined. If $K < K_{ISCC}$, SCC can be avoided. On the other hand, if $K > K_{ISCC}$, SCC is possible. The stress corrosion crack growth (SCCG) curve can be applied for the life prediction as well as the maintenance planning. Moreover, as a fracture mechanics parameter to relate the damage of protective layer at crack tip, the procedure to experimentally and numerically evaluate the CTOD 45° of T-WOL specimen was proposed. As an

alternative parameter, the engineers and researchers can apply CTOD 45° to gain better understanding of SCC.

SCC of NAB can be further minimized; if the K and J -integral at the critical location are decreased, while the K_{SCC} and J_{SCC} are increased. The K and J -integral at the critical location can be decreased by redesigning the component as well as specifying the limited load during service. On the other hand, K_{SCC} and J_{SCC} can be increased by modifying the chemical composition, microstructure, and surface treatment.

6.4 Future work

Diagram of SCC of NAB under both plane stress and plane strain conditions is shown in Fig. 6-1. The yellow blocks represent the scope; the green blocks represent the accomplishment; and the orange blocks represent the future work. For SCC of NAB under plane stress condition, CTOD at crack front of SENB specimen should be numerically calculated and validated. Subsequently, the application of CTOD to characterize da/dt during SCC experiment should be investigated. On the other hand, under plane strain condition, SCC of NAB should be performed. Then the application of K and CTOD to characterize da/dt during SCC experiment as well as the metallurgical analysis of corrosion product and fracture surface after SCC experiment should be investigated.

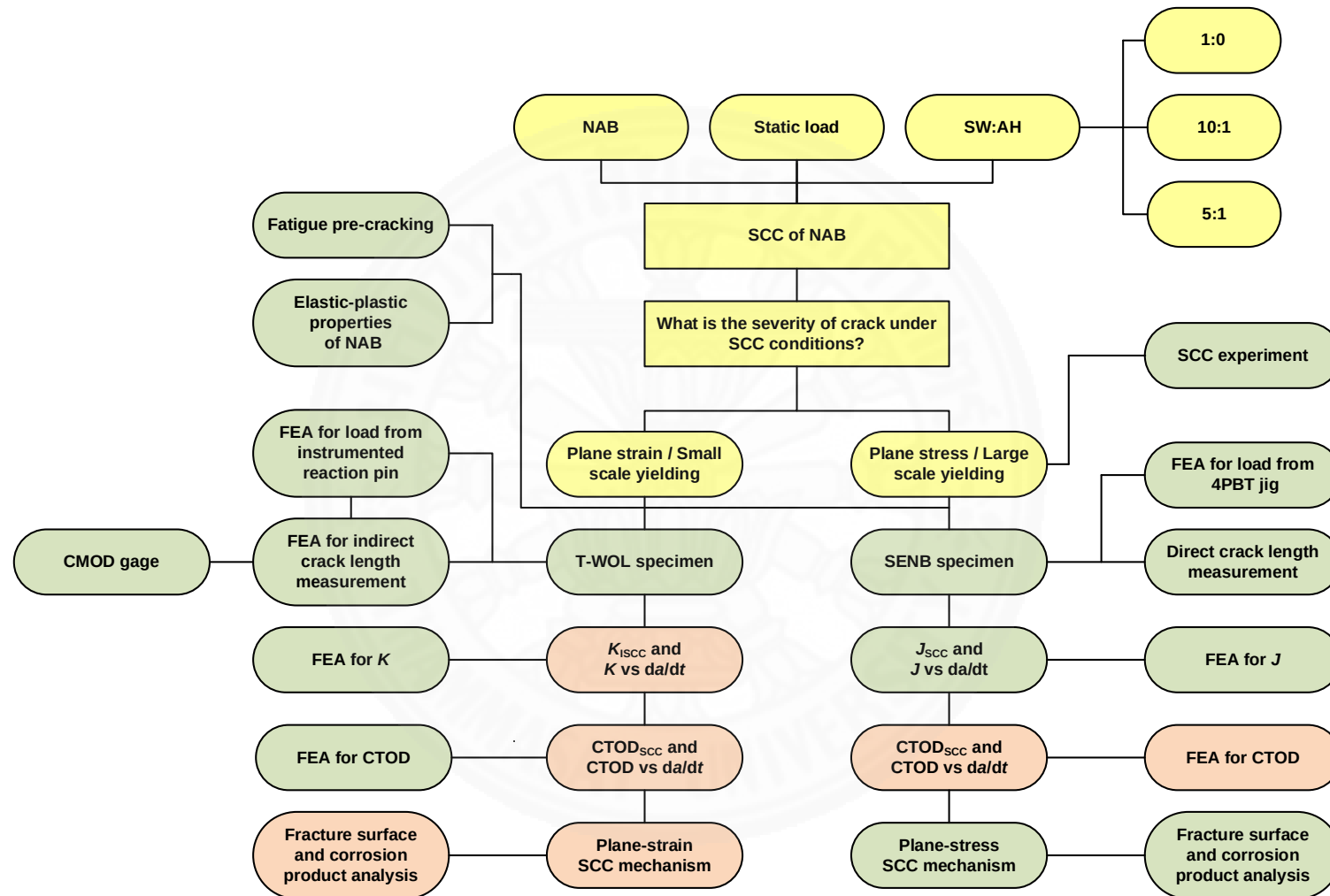


Fig. 6-1 Diagram of SCC of NAB under both plane stress and plane strain conditions.

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Appendix A

Design and calibration of 4-point bending jig

This appendix contains the detail of design, construction, and calibration of 4-point bending jig (4PB jig). The 4PB jig is an apparatus used for SENB specimen, i.e., plane-stress SCC experiment. It can generate various levels of bending stress to SENB specimen during SCC experiment. The 4PB jig was made from stainless steel grade 304 to minimize the effect of corrosion from ammonia-containing artificial seawater. The geometry of 4PB jig is shown in Fig. A-1.

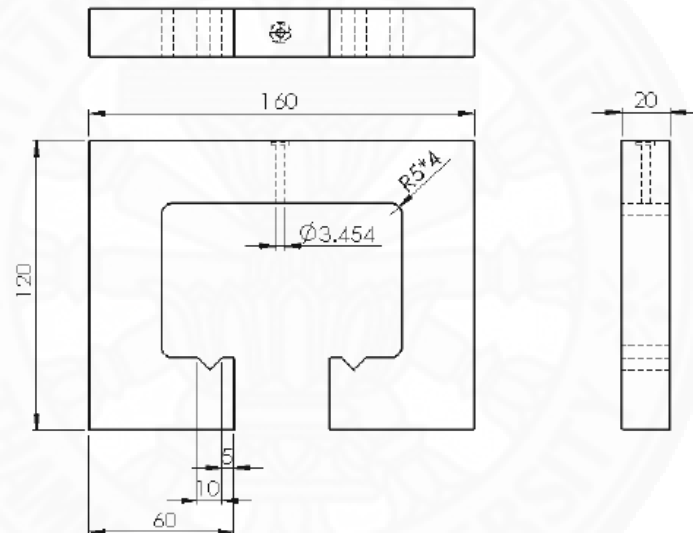


Fig. A.1 Schematic illustration of 4-point bending jig (dimension in mm).

FEA was applied for the stress analysis of 4PB jig. The maximum applied force of approximately 2.5 kN was estimated from the fracture toughness of NAB (i.e., 25 MPa.m^{1/2}) and the shortest crack length for SENB specimen (i.e., $a/W = 0.45$). Therefore, the applied force of 3 kN was used for the calculation of von Mises stress. The numerical result is shown in Fig. A-2. The maximum von Mises stress (i.e. approximately 20 MPa) is significantly lower than the yield stress of stainless steel grade 304 (i.e., 215 MPa); thus, the marginal elastic deformation of 4PB jig is expected during SCC experiment.

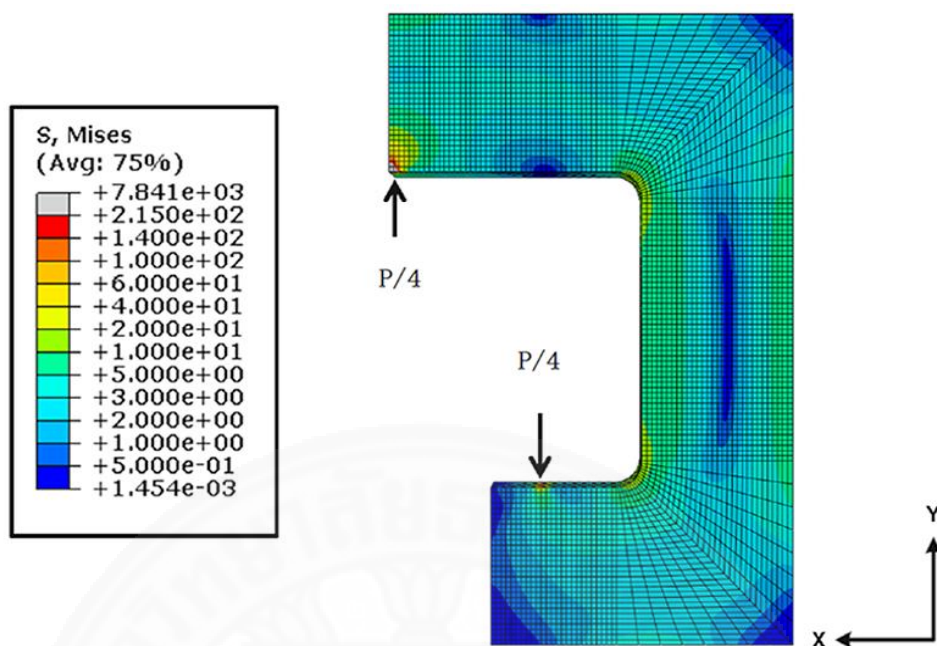


Fig. A-2 Von Mises stress of 4-point bending jig under an applied load of 3 kN.

Relationship between the measured compressive force (i.e., from a load cell placed on the specimen) and the measured strain (i.e., from strain gages attached on 4PB jig) was used as a calibration curve of 4PB jig. The 1-kN load cell is Kyowa compression load cell (LCN-A-1kN), while the strain gages are full-bridge TML strain gages (FLA-03-11). Setup for the calibration of 4PB jig is shown in Fig. A-3. The average results from three repeated experiments are compared with the numerical results, as shown in Fig. A-4. Relationships between the compressive force and the strain experiments and FEA show linear behaviour, and they are in good agreement. Therefore, the present 4PB jig and its calibration can be applied for SCC experiment.

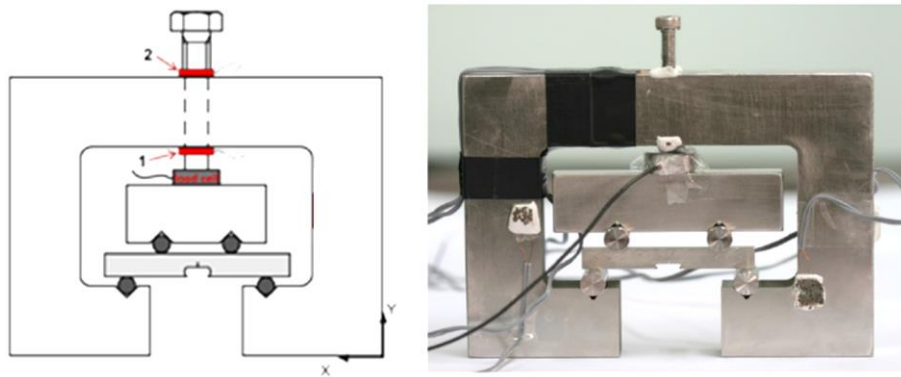


Fig. A-3 Setup for the calibration of 4PB jig.

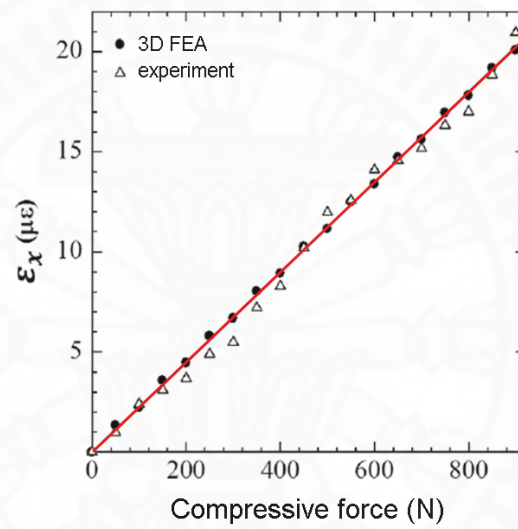


Fig. A-4 Relationships between compressive force and strain of 4PB jig.

Appendix B

Mesh sensitivity: FE model of single-edge notch bending specimen

This appendix presents the influence of mesh density on FE model of SENB specimen. For a thin SENB specimen, the deformation around the crack front becomes significantly large. Thus, the mesh density should be high enough to characterize the complex stress and strain gradient around the crack tip. To determine the proper mesh density of FE model, the relationships between number of elements and J -integral on side-surface plane and mid-thickness plane of SENB specimen were calculated, and the number of elements that provides the convergence of J -integral was evaluated. Based on the fracture toughness of NAB (i.e., $25 \text{ MPa}\cdot\text{m}^{1/2}$) and crack length used in the present work ($a/W = 0.5$), the applied force for the analysis of mesh sensitivity is 2.2 kN. Relationships between number of elements and J -integral on side-surface plane and mid-thickness plane of SENB specimens are shown in Fig. B-1. It is found that at least 44,000 elements should be used for minimizing the effect of mesh density on the calculation of J -integral.

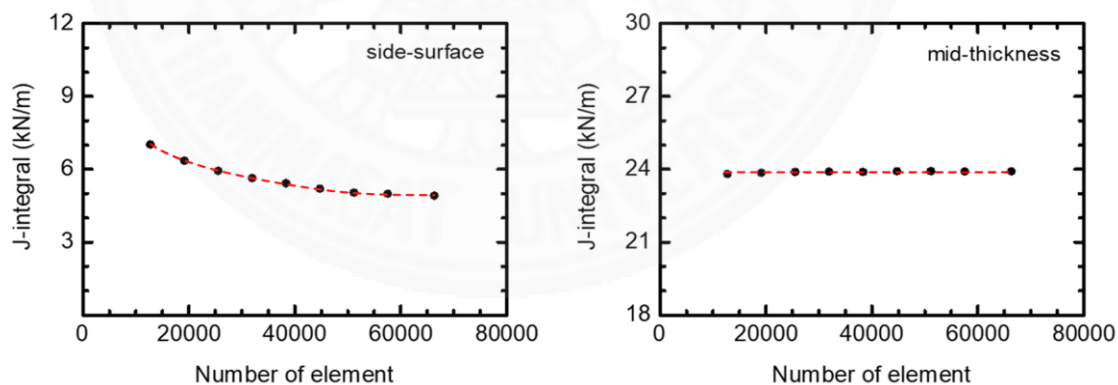


Fig. B-1 Relationships between number of elements and J -integral on side-surface plane and mid-thickness plane of SENB specimens ($a/W = 0.5$) under an applied force of 2.2 kN.

Appendix C

Mesh sensitivity: FE model of T-type wedge opening loaded specimen

For T-type wedge opening loaded specimen (i.e., T-WOL), the CTOD 45° parameter was used to characterize the severity at the crack tip. The stress-strain field around the crack tip become complication, especially for a blunt crack. Because the stress-strain field around the crack tip depends on the mesh size; thus, the mesh size should be properly selected. To determine the proper mesh size at crack tip, CTOD 45° were calculated using various element's sizes (r), and the element's size that provides the convergence of CTOD 45° was evaluated. Based on the fracture toughness of NAB (i.e., $25 \text{ MPa}\cdot\text{m}^{1/2}$) and crack length used in the present work ($a/W = 0.46$), the applied force for the analysis of mesh sensitivity is 13 kN. Relationship between size of elements and CTOD 45° on mid-thickness plane of T-WOL specimen are shown in Fig. C-1. It is found that the element's size of 0.2 mm or smaller should be used for minimizing the effect of element's size on the calculation of CTOD 45° .

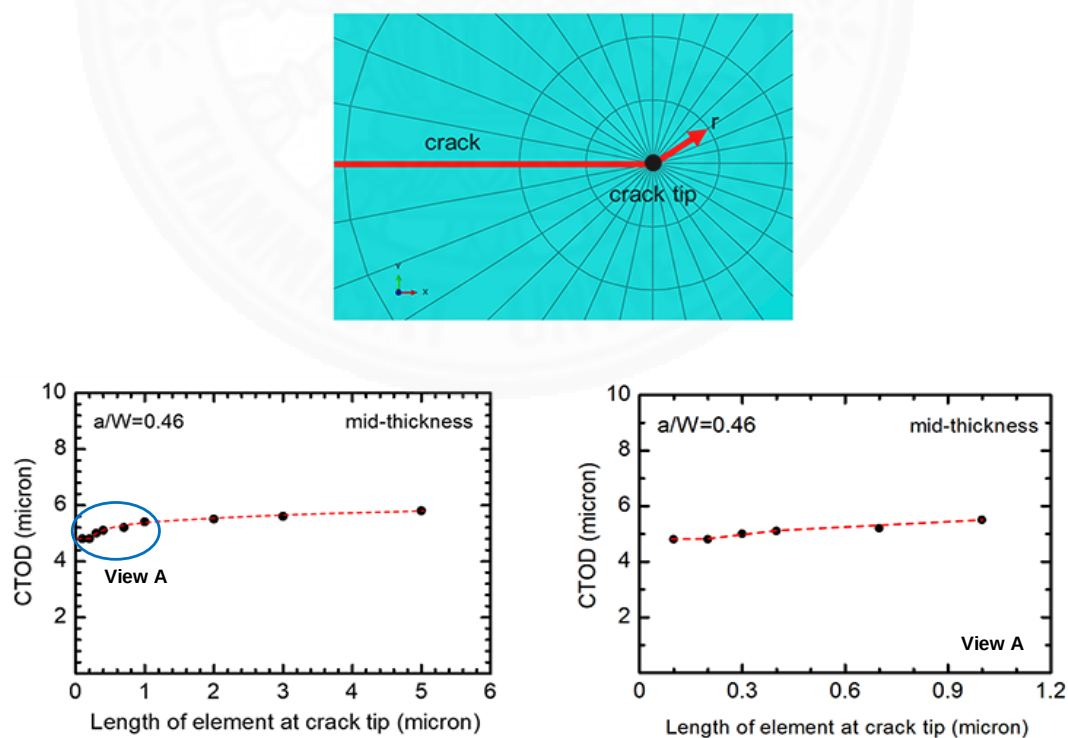


Fig. C-1 Relationship between size of elements and CTOD 45° on mid-thickness plane of T-WOL specimen.

Appendix D

Mesh sensitivity: FE model of instrumented reaction pin

3D elastic-plastic FEA was carried out to investigate the normal strain in vertical direction of the strain gage adhesive. Because the strain of strain gage adhesive depends on the mesh size; thus, the mesh size should be properly selected. Since the strain gage adhesive is significantly small than the instrumented reaction pin; the mesh sensitivity of the strain gage adhesive in an instrumented reaction pin was investigated using a sub-modeling technique. A half of an instrumented reaction pin model was used as a global model, while a half of cylindrical volume for strain gage adhesive was used as a local model (Fig. D-1).

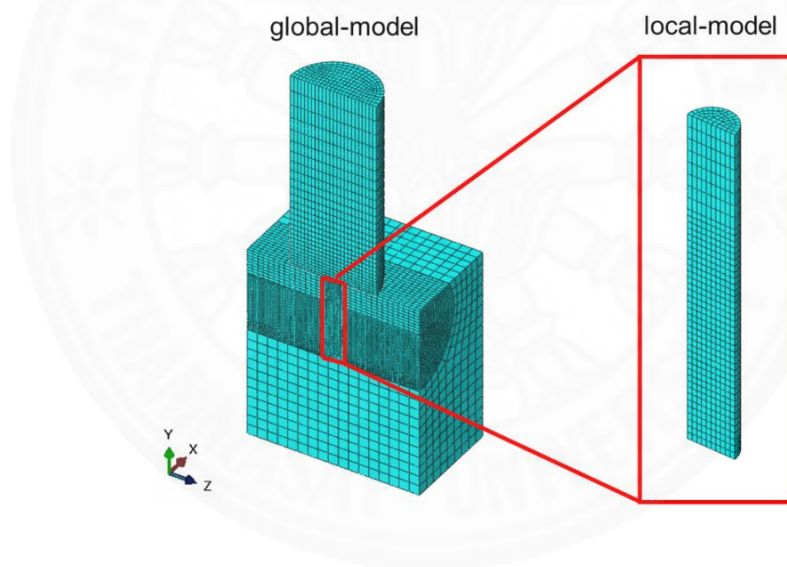


Fig. D-1 FE model of instrumented reaction pin with strain gage adhesive.

Base on the fracture toughness of NAB (i.e., $25 \text{ MPa}\cdot\text{m}^{1/2}$) and the expected shortest crack length (i.e., $a/W = 0.4$), the applied force is approximately 14 kN. The normal strains in vertical direction of the strain gage adhesive calculated using various numbers of elements are shown in Fig. D-2. The numerical results from various numbers of elements are similar, thus, FE model with 323 elements was selected for the calculation of normal strains in vertical direction of the strain gage adhesive.

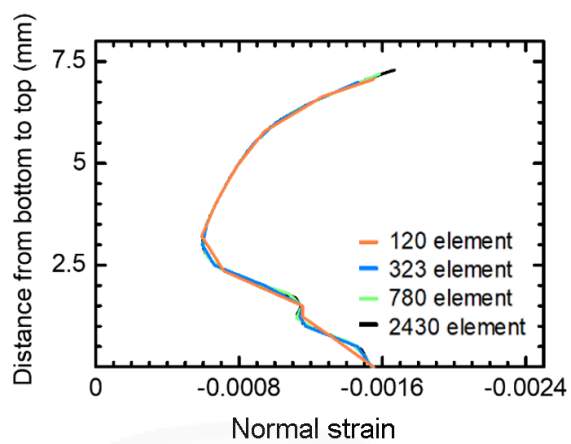


Fig. D-2 Normal strains in vertical direction of the strain gage adhesive calculated using various numbers of elements.

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Scholarship	2016: Thammasat University Doctoral Student Scholarship 2019: Thammasat School of Engineering Research Fund

Journals

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