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Effect of Prestrain on Delayed Fracture of Stainless Steel 304

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Abstract

The purpose of this study is to investigate the relationship between delayed fracture and environment-assisted cracking of prestrained SS304 in terms of displacement versus time to failure in the environment containing 10% H_2SO_4 solution. Specimens were initially pre-strained in axial direction with the percentage of 5% and 10%. Corrosion testing was conducted according to the standard ASTM E-1681. The curve of deflection vs. time to fail and SEM of micrographs of specimens after testing, are studied to reveal the mechanism of crack formed. Data deflection and time until failure is only obtained in static bending stress levels about 20% of the yield stress of material. The incubation period for the material without prestrain is 29.9 h, with 5%prestrain 4.17 h, whereas the prestrain of 10% is 32.5 h. The longer the incubation periods was undegone by specimens with the greater percentage of prestrain. It is attributed to strain hardening effect. For the specimen with low percentage of prestrain, cracks are found tend to be more dominant transgranular. In opposite to the greater percentage of prestrain, cracks tend to grow in inter-transgranular.

Keywords: *prestrain, delayed fracture, inter- trans granular, H_2SO_4 solution*

Stainless steel with grade AISI 304 is widely used as an engineering material for structural component of piping systems in petrochemical industry and power plants because this material has welding ability and corrosion resistance in environments containing chlorine [1]. During service, the components of the structure often subjects to loading in corrosive environment. One corrosive environment is air pollution as result of residual burning such as diesel oil and coal-fired power generation. The atmosphere in the polluted area always contains SO_2 and SO_4 because of sulfur impurities in the fuel [2]. A lot of acid deposition as result of residual burning will chemically react with air and water in atmosphere as acid rain, containing of H_2SO_4 [2]. Combination of stress and H_2SO_4 environment can cause corrosion cracking and destroy subsequently the structural engineering components.

Pre-strain is essentially plastic deformation in cold working process [3] experienced by its material, for example, results of manufacturing process i.e. rolling, drawing, pressing and natural phenomenon i.e. earthquake [4,5]. Over pre-strain will affect the ductility of material in fracture behavior. The pre-strain process elongates the grains into long fibrous structures, the long axes of the fibers running in the direction of the pre strain. Therefore, this has significant effect on the mechanical properties and corrosion resistance of the material [6].

Plastic deformation induced by shot penning is susceptible to the delayed fracture of high strength steels performed using a static cantilever loading in the solution of 0.1N HCl, [7]. The significant effect of deformation in enhancing SCC is important not only because of intentional cold work but also because of fabrication-induced surface deformation and residual deformation. Cold working could severely decrease the resistance of carbon steel API X52 in liquid bicarbonate environment until abrupt failure under the 20% of total strain [8]. At 90% of high stress level of elastic limit, branched inter granular crack was found in the carbon steel immersed into the solution of 30% sulfuric acid [9].

The main purpose of this study was to investigate the effects of pre-strain on delayed fracture behavior of austenitic stainless steel 304 in the solution of 10% concentration sulfuric acid. An attempt is made to determine the relationship of delayed fracture and environment-assisted cracking of prestrained SS304 in terms of the displacement versus time to failure. Additionally, fracture surface using SEM provides some qualitative information that would help in obtaining a better understanding in this research.

REVIEW OF THE THEORY

Environments and Stress Corrosion Cracking (SCC)

The specificity of environments that will promote stress corrosion cracking is significant. It is important to realize that not all corrosive environments promote the formation of stress corrosion cracks. Those that do will usually be those that do not promote widespread corrosion in the sense of the attack being spread fairly uniformly over all exposed surfaces, since, if for no other reason, this is not likely to lead to the geometry of a crack, which requires that the crack sides remain relatively inactive whilst the tip remains active to maintain propagation into the metal.

Consequently those environments, such as sea water, that normally promote general corrosion of mild steel, are not likely to promote stress corrosion [10], whilst those chemicals sometimes used to control corrosion by addition to an otherwise corrosive environment may result in a borderline condition, between general corrosion and no corrosion, wherein the attack can be localized [11]. Thus, the addition of caustic soda to boiler feed waters to reduce the corrosiveness of the latter towards mild steel can result in the form of stress corrosion frequently referred to as “caustic cracking” [12]. The important general point is that those environments that cause stress corrosion are frequently highly specific to the particular alloy involved. A list of some environments that have been shown to promote stress corrosion in various materials is given in the following Table 1.

Although this list of environments that have been shown to promote stress corrosion cracking may appear extensive it is by no means exhaustive. For a given alloy however there are many more environments that do not cause stress corrosion than those that so act. It is possible, by appropriate electrochemical measurements or by laboratory stress corrosion tests properly conducted, to identify potent environments for a given material, although failures continue to occur in circumstances that may not reasonably have been expected.

Two types of cracks occur for SCC, either intergranular or transgranular. Usually the density of cracks increases with depth as the stress increases in the material as the crack grows. One possible method of telling SCC

Material	Environments
Al alloys	Chlorides, moist air
Mg alloys	Chloride-chromate mixtures, moist air
	Nitric acid, fluorides
	Sodium hydroxide
Cu alloys	Ammonia, moist air, moist sulfur dioxide
C steels	Nitrates, hydroxides, carbonates
	Anhydrous ammonia
Cu alloys	Ammonia, moist air, moist sulfur dioxide
Austenitic steels	Chlorides, sulfur acid
High strength steels	Moist air, water, chlorides, sulfates, sulfides
High strength steels	Moist air, water, chlorides, sulfates, sulfides
Ni alloys	Hydroxides
Ti alloys	Halides, methanol

Table 1. Combinations of some alloys and environments showing to promote stress corrosion cracking [12]

from intergranular corrosion is the density of cracks as a function of depth. For intergranular corrosion the density decreases but for SCC the density increases. Specific combinations of environment and materials are required for SCC. Brasses do not SCC in chloride environments while stainless steels do. Stainless steels do not crack in ammonia environments but brasses do [13].

Stress Corrosion Cracking Mechanisms

Regions where stress corrosion cracks are viable can be related to the potentiodynamic scans for passive materials. Two regions are possible, one at the start potential for passive films or the pit nucleation potential and a second at the active to passive transition at much lower potentials. It should be noted that at both these potentials the passive film is somewhat unstable. In one case it is just on the verge of forming and in the second case it is just on the verge of breakdown. Therefore SCC could be viewed as amplification of instability in passive films. Importantly, SCC can be viewed as an anodic process as anodic current is required for SCC to occur. It is this fact that separates it from hydrogen embrittlement which has many of the same features but is cathodic in control.

Stress corrosion cracking can be separated into two distinct regions, crack initiation and crack propagation [14].

Crack Initiation

Cracks can be initiated by several mechanisms:

Mechanical features

Cracks will often initiate at features such as scratches, nicks or dents on the surface of the metal. In this case the local environment or local stress conditions favor enhanced dissolution, poor formation of a passive film or in-situ damage to a protective film.

Local galvanic cells initiating dissolution

Local corrosive effects dominate the process where the local galvanic cell locally dissolves one phase of the material. This will also localize the stress on the material. The crack in this case may initiate in a transgranular mode or an intergranular mode. One example of the latter would be intergranular cracks during SCC of sensitized stainless steels [14,15].

Pitting type crack initiation

The pitting potential is related to formation of its and there is some correlation between the pitting potential and the potential for stress corrosion cracking. A 10:1 ratio of pit depth to width was suggested to be needed for a pit to initiate a crack [16]. The local environment in a pit may also be important in the crack growth process. For example, the environment in a pit mat favor crack growth by intergranular crack growth. Several studies were made employing a pit as an effective crack in the surface to be used in linear elastic fracture mechanics approach. These have met with some success. However electrochemical effects can nullify this approach.

Initiation at stress induced phenomena

Slip lines intersecting the surface can have a double effect. One is to provide local anodes as the site is very active. The second is to rupture passive films on the surface and locally form dissolving regions.

Once a crack has initiated, then it will grow. As pointed out above the growth mechanism may not be the same as the initiation process.

Crack Growth Mechanisms

One important feature for SCC cracks is that some show clear evidence of stopping and starting. The cracks do not continually grow to failure.

Film rupture mechanism

The tensile stress ruptures films at the crack tip and the crack grows rapidly from the bare metal exposed until the crack tip can repassivate in some cases or grow slowly to failure in other cases (Fig.1). Unfortunately, SCC cracks often have significant features on

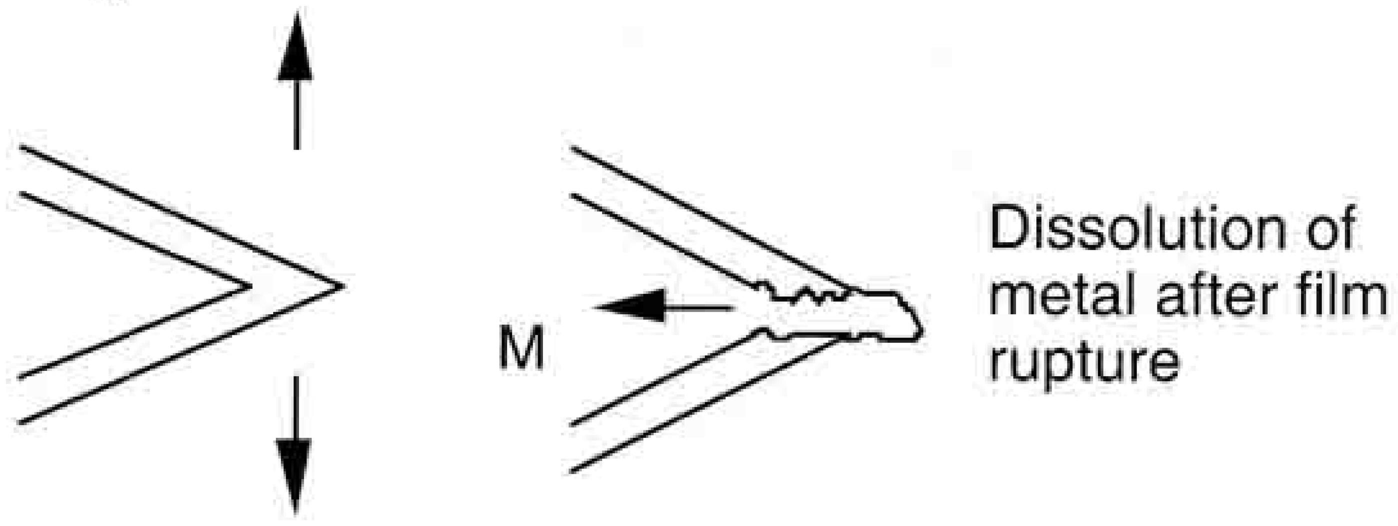


Figure 1. Propagation of crack corrosion after passive film breakdown [12]

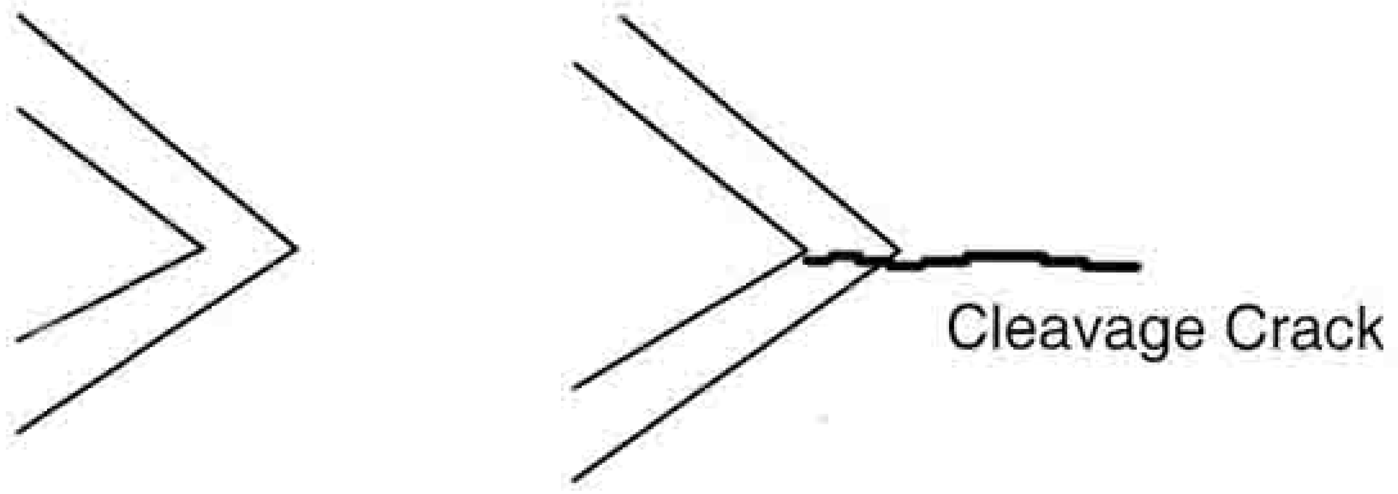


Figure 2. Crack corrosion caused by film cleavage [12]

their faces which should be removed by local dissolution effects. In addition the crack plane in transgranular failure is often not the active slip system in the metal. Others have suggested a similar model in which the film is formed by a tarnish process. Intergranular corrosion is proposed to occur by preferential oxidation of the grain boundaries.

Film cleavage mechanism

In this mechanism, the surface film grows and may increase in internal stress with thickness. This combined with the applied tensile stress induces brittle failure in the film which propagates across into the metal and provides a period of crack growth. The loss of the film stress and plasticity in the metal than blunts the crack and stops it growing to give periods of crack growth followed by rest while the film grows back to the conditions for cleavage.

Adsorption induced cleavage

During the electrochemical process atoms are absorbed on the surface that weakens the bonds. The stress to initiate a crack then decreases and a crack grows until it is blunted by plastic deformation or grows out of the adsorbed region.

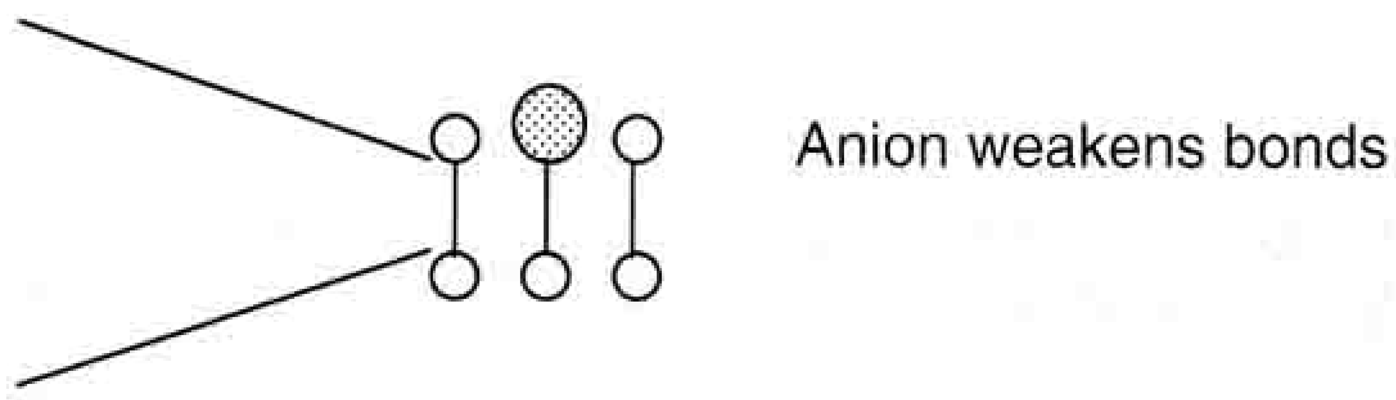


Figure 3. Anions adsorption induced crack cleavage [12]

EXPERIMENTAL PROCEDURES

The material used in this research is austenitic stainless steel of AISI 304 type with the thickness of 5 mm, and then cut in the direction of rolling with the dimension of 200 mm in long and 40 mm in wide. Later, specimens were axially strained up until reaching the uniform strain of 5% and 10% using servo pulsar UTM 9506 under displacement control of 0.3 mm/sec.

The pre-cracked beam specimen dimension is of 200 x 20 mm^2 and the testing method for the delayed fracture tests according to ASTM E-1681 is shown in Fig. 4 [17]. The bending moment was applied by giving a static load at the extended end of the test piece in a cantilever type load (Fig. 4). Solution of 10% concentration H_2SO_4 was dropped on the notched part of the specimen with feeding rate

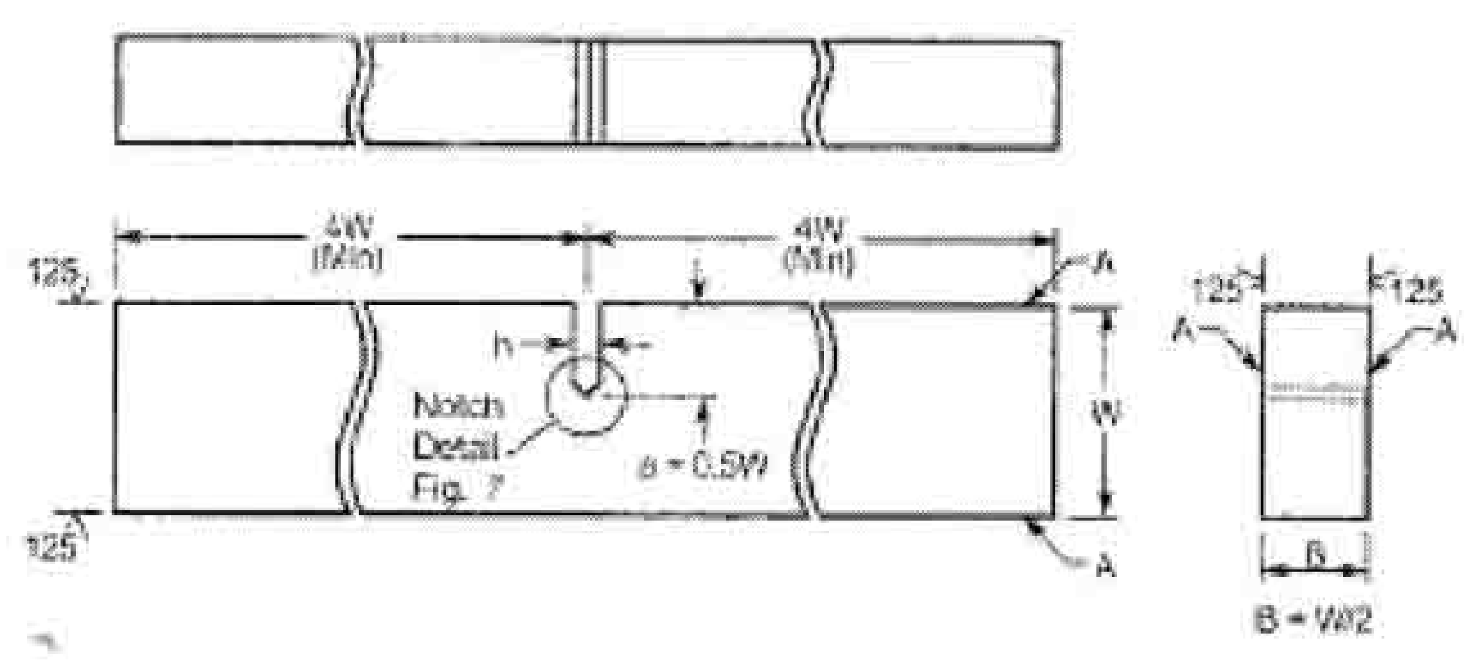
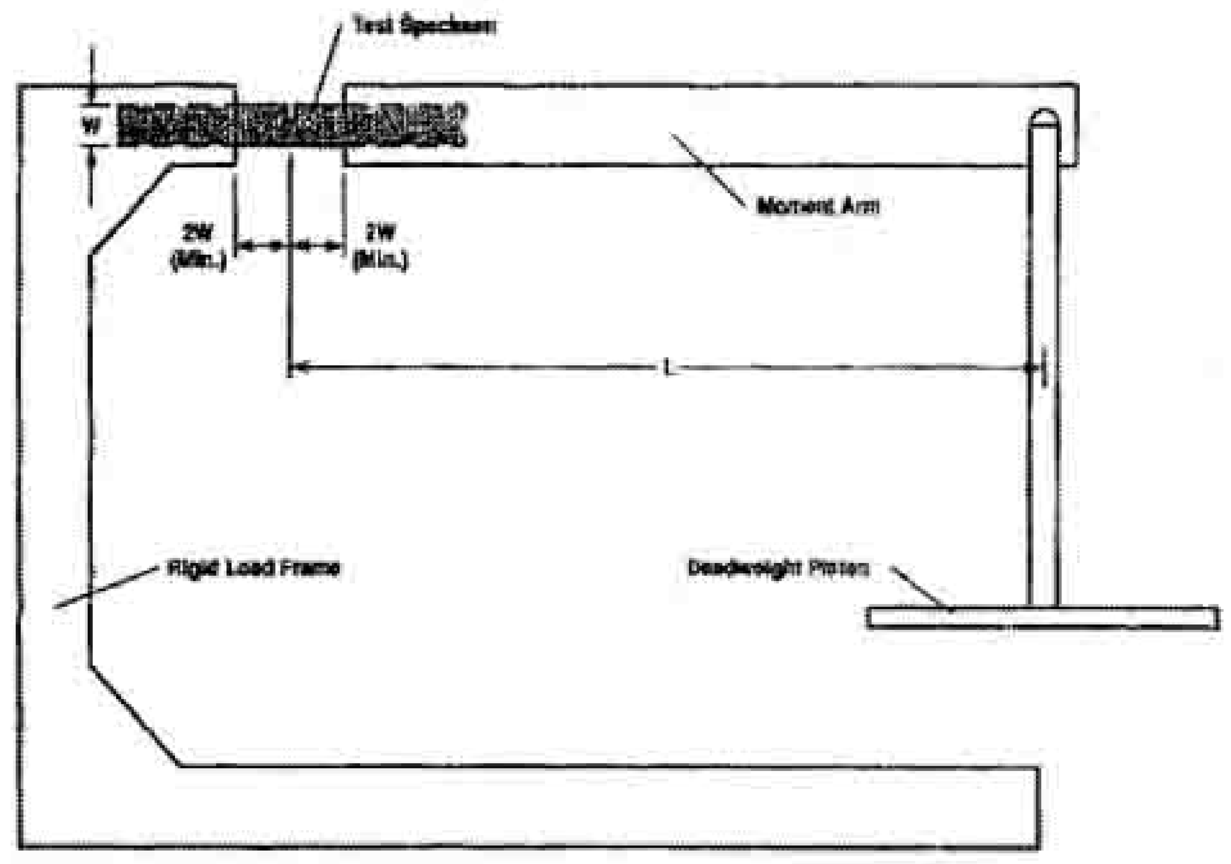


Figure 4. Specimens geometry and testing device [17]



of 0.2 mL/sec. The deflection data and time were obtained by measuring the deflection using a digital dial gauge, placed at the distance of L mm on the moment arm and digital timer, respectively. The cantilever load was statically given by a various bending stress of 20–200% of the yield strength of material. These data were then plotted in both the displacement versus the time to failure.

RESULTS AND DISCUSSIONS

Generally, the delayed fracture process is composed of three regions, incubation period, the crack propagation region, and the final fracture stage [15]. Incubation period is equivalent to the range of slow increasing deflection until to initiate crack. Three regions (primary, secondary and tertiary) in displacement vs. time to failure curve correspond to nucleation and propagation periods for SCC process is shown in Fig. 5.

The steady state deflection rate becomes a parameter for prediction of time to failure such as that in sulfuric acid. The primary region is nucleation period, while the secondary and tertiary regions correspond to the propagation period. In addition, the transition time distinguishing between the steady and terminal propagation periods is a critical time of whether or not the reduction in cross sectional area affect deflection rate. In this study, the delayed fracture test applied can not observe crack propagation of specimen at crack tip directly. The data can just be read by the change of displacement in vertical direction (deflection) to clarify the effect of pre- strain on the delayed fracture. Delayed

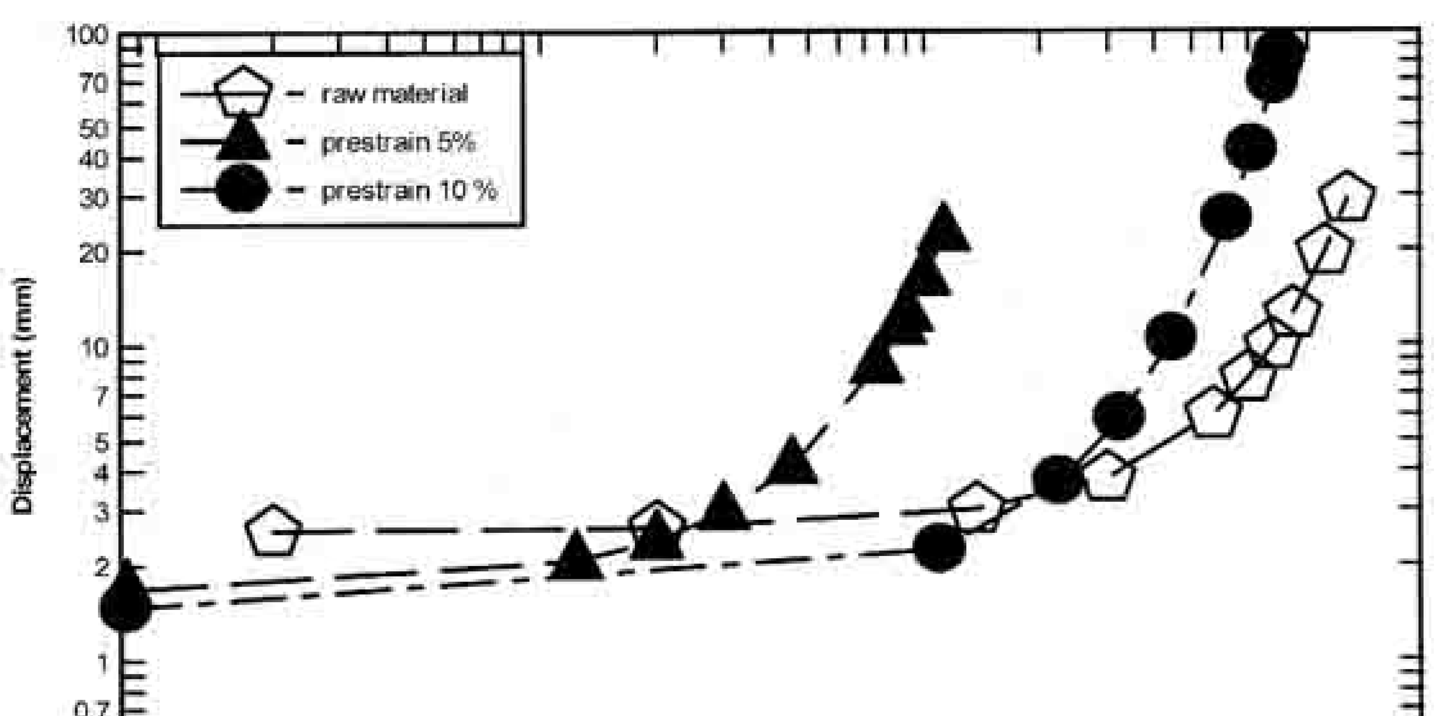


Figure 5. The relation between time to failure and deflection of moment arm at bending stress of 369,9MPa

fracture is defined as one of the hydrogen embritlement failures. The recorded data in the digital dial gauge are only for the 20% loading, whereas the loading over 20% of yield strength was not recorded because both the change of deflection and time to failure are very fast. The deflection data and time to failure just could be recorded at the initial time (set up loading) and final fracture.

The change of deflection for the raw material is greater than both the pre-strain of 5% and 10% at the first one hour. The period of incubation of raw material, the pre-strain specimen of 5% and the pre-strain specimen of 10% prior to starting crack are 29.9, 4.17 and 32.5 hours, respectively. Even though, the incubation of periods for the specimen without pre-strain was greater than those of specimens pre-strained but the time of failure is longer. This cause is attributed to the plastic deformation experienced by the raw specimen after loading, as shown in Fig. 5. The material still has a higher elastic property, while the initial loading applied, initial crack on the notch tip will be plastic, so that the incubation period will be prolong. Pre-strain or initial plastic deformation of the metal will create strain hardening due to the moving of dislocation that interact each others. The great of strain hardening effect depends on the percentage of pre-strain given. In the pre-strained specimen of 5%, the material has the little plastic property, when the initial loading applied, initial crack on the notch tip will be brittle, so that the specimen will fail abruptly under the static bending. For the pre strained specimen of 10%, the effect of strain hardening is greater than raw material and 5% pre strained specimens. Thus, this will increase the hardness of material [18]. The greater hardness of the material, the longer incubation period and time to failure than those of specimen with 5% pre-strained. As shown in Fig. 5, it can be confirmed that the deflection gradually increases with the increasing loading, and then rapidly increase up to failure.

The stage of the delayed fracture process as mentioned above, describing the behavior of stress corrosion crack with respect to displacement of initial crack formation is shown in Fig. 6. Test of specimen was stopped at proper deflection to examine whether crack

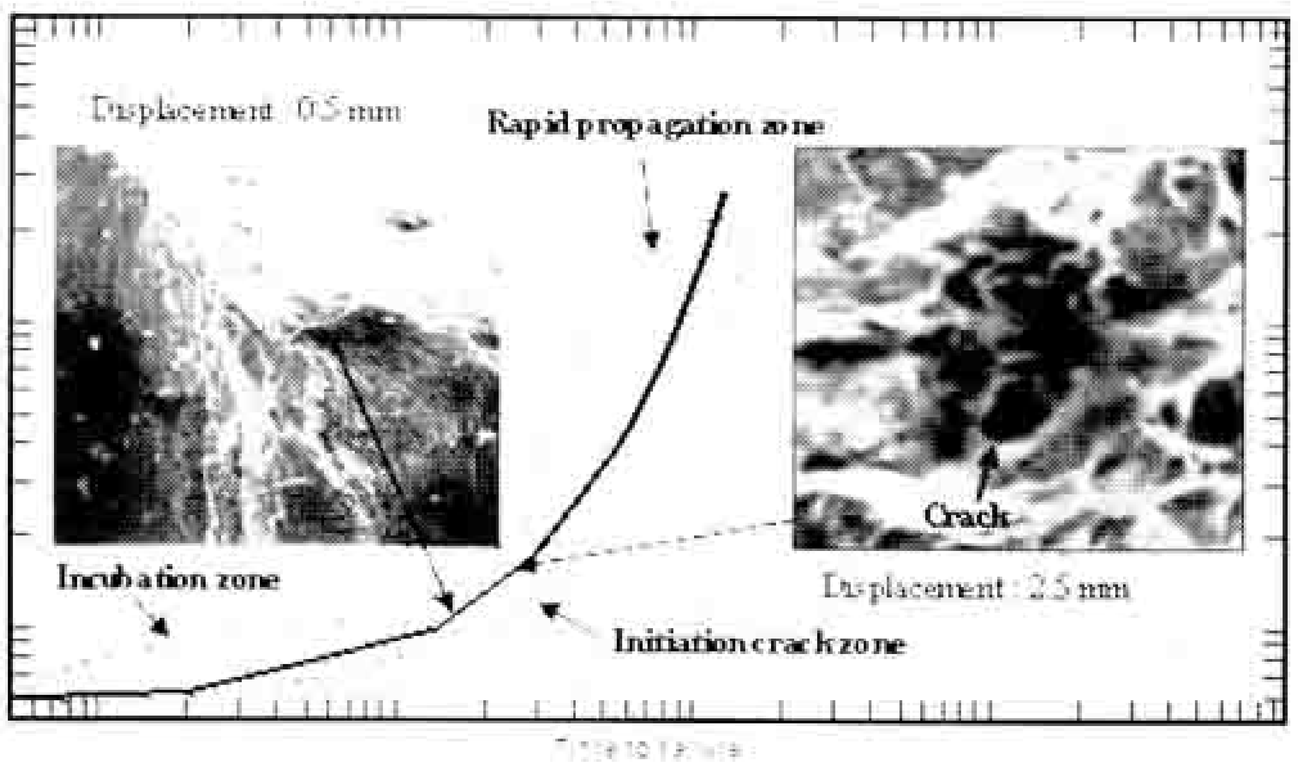


Figure 6. Photomicrograph of crack initiation for the specimen of 10% pre-strain at the bending stress of 616 MPa

is initiated or not. As shown in Fig. 6, the fracture surface after testing (inset in Fig. 6) that crack was not initiated at the range of slow increasing deflection but after deflection of 2.5 mm, the crack was formed, leading to the rapid deflection until to failure. This result leads to the conclusion that the greater percentage of pre strain, the slower crack formation. Therefore, the incubation period is prolonged. Furthermore, hydrogen from the solution of sulfate acid penetrated and diffused in the crack zone, generating an increase of the critical concentration at the crack tip. This is caused by combination of both the increase of density of dislocation and hydrogen absorption at the surface layer in the plastic deformation zone where crack is formed. The great density dislocation has ability to trap diffused hydrogen into the crack tip [8].

It is deduced that the inhibiting effect of sulfuric ions on SCC is caused by hydrogen adsorption on active surfaces after film breakdown, which would lead to the inhibition of active dissolution. Adsorption of sulfuric ions at higher sulfuric acid concentrations could accelerate active dissolution and/or inhibit film formation (repasivation). Therefore, it can be concluded that both greater percentage of pre strained specimens and the magnitude of critical hydrogen diffusion can affect formation of crack initiation and later, the incubation period is prolonged until an appropriate time [8].

- If the corrosion rate of the alloy in the environment is low, and if the experimental conditions are such that plastic deformation of the cracked surfaces is minimal, both sides of the crack should match perfectly after cracking

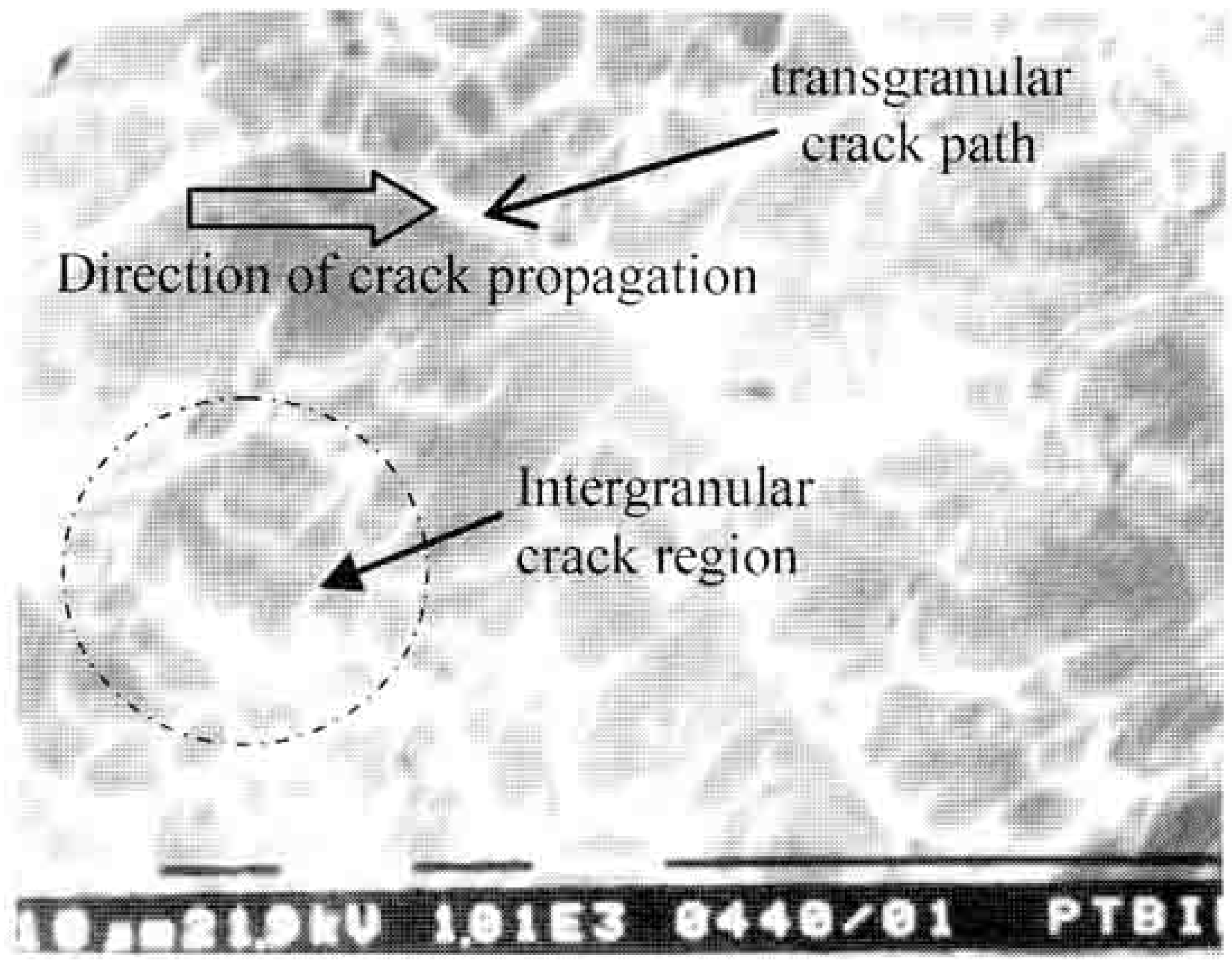


Figure 7. The representative fracture surface of prestrained specimen of 5% at the static bending of 369. MPa (X1010)

[19]. In this case, crack will propagate on the slip steps, but it will break into smaller cracks. A direction perpendicular to the tensile stress direction suggests that cracks will try to follow those crystallographic planes.

Figure 7 shows the representative fracture surface of the pre strained specimens of 5% at the static bending of 369.9 MPa with the failure time of 11.5 hours in the SCC dominated region. The fracture surface of the specimens pre strained at 5% appeared to be a mixture of an inter-granular (the cycle) and a trans-granular mode (the small arrow). The fracture surface of the pre strained specimen of 5% is predominantly inter granular. The inter granular cracks followed the elastic grain boundary, whereas trans granular crack broke the brittle grain boundary. It means that the change of fracture mode depends on the percentage value of the pre-strain.

CONCLUSIONS

1. The period of incubation of raw material is longer than both the pre-strain of 5% and 10% specimens at the first one hour because the material still has the elastic property, while the initial loading applied, initial crack on the notch tip will be plastic.
2. Pre-strained hardening of metal will affect the incubation period to form crack initiation, and hydrogen trapped in

the crack cause rapid displacement of crack.

3. The intergranular crack is predominantly found at the specimens of 5% pre strain. The increase of percentage of pre-strain tends to yield the trans-granular crack surface after testing exposed to corrosion environment containing 10% H_2SO_4.

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