

UNIVERSITY OF JORDAN
FACULTY OF GRADUATE STUDIES

TO SHOT PEENER ... THANK
YOU FOR YOUR KIND
HELP

Mayyada Rimawi
Y.P. 28.10.95

EFFECT OF INTRODUCING SURFACE COMPRESSIVE STRESSES
ON STRESS CORROSION CRACKING (SCC) RESISTANCE OF HIGH
STRENGTH ALUMINUM ALLOYS IN CHLORIDE ENVIRONMENT

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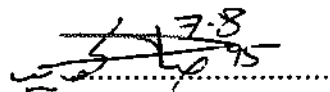
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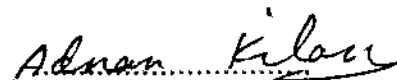
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Dedication

*To My Parents
To My Family*

*For the Donation they Give
For the Life We Live*

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LIST OF SYMBOLS

SCC	Stress Corrosion Cracking
IG	Intergranular
GB	Grain boundary
HE	Hydrogen Embrittlement
PFZ	Precipitate Free Zone
L	Longitudinal direction of rolling
LT	Longitudinal Transverse direction of rolling
ST	Short Transverse direction of rolling
VSR	Vibratory Stress Relieving
HAZ	Heat Affected Zone
WSP	Wet Shot Peening
CSP	Controlled Shot Peening

ABSTRACT

EFFECT OF INTRODUCING SURFACE COMPRESSIVE STRESSES ON STRESS CORROSION CRACKING (SCC) RESISTANCE OF HIGH STRENGTH ALUMINUM ALLOYS IN CHLORIDE ENVIRONMENT

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The high strength aluminum alloy 7075-T6 is used widely in aircraft industry. Nevertheless this alloy is susceptible to SCC especially in the welded condition.

In this investigation, an experimental program to improve the stress corrosion cracking resistance of 7075-T6 alloy in chloride environment by shot peening has been carried out.

Shot peening is a multiple impact of particles onto an elastic/plastic target, which results in compressive stresses. Thus retarding the crack formation and preventing stress corrosion cracking of metal parts. It has been proved that tensile stresses are one of the most essential factors of SCC. Therefore the compressive stresses induced by shot peening retard SCC.

The experimental work was first carried out to select the best chloride environment promoting SCC.

The SCC susceptibility of the welded specimen was metallurgically studied and was attributed to the metallurgical change induced during welding.

Finally, the shot peening effect on the SCC resistance of welded 7075-T6 specimens was studied at different intensities. It was found that shot peening is superior in preventing SCC at some intensities and retarding it at others.

CHAPTER ONE
INTRODUCTION AND LITERATURE REVIEW

CHAPTER ONE

INTRODUCTION AND LITERATURE REVIEW

1.1 Introduction

Stress corrosion failures of high strength aluminum aircraft components have been numerous and costly [1]. Although stress corrosion cracking (SCC) is not limited to aluminum, it is a particularly acute problem with this metal because of its wide use in critical aircraft structures.

One of the most economic and effective preventives for SCC of metallic materials is surface working by shot peening [2].

This work contains the results of research experiments, which demonstrate the effectiveness of shot peening to improve the SCC resistance of high strength aluminum alloy 7075-T6. This alloy was chosen because it is one of the most common aluminum alloys to be used in airframe construction [3].

1.2 Stress Corrosion Cracking

Stress corrosion cracking (SCC) is a term used to describe service failures in engineering materials that occur by slow environmentally induced crack propagation [4].

The observed crack propagation is the result of the combined and synergistic interaction of mechanical tensile stress and corrosion reactions. This is a simple definition of a complex subject, and like most simplifications, it fails to identify the boundaries of the subject [4].

SCC is very common throughout industry, and despite decades of intensive research, after which workers are only now beginning to understand the processes involved, measures adopted to control it are still frequently unsuccessful [5].

One feature of SCC which occurs repeatedly is the unexpectedness of such cases. Often, a material, chosen for its corrosion resistance in a given

environment, is found to fail at a stress level well below its normal fracture stress. Rarely is there any obvious evidence of an impending failure, and because it can occur in components which are apparently unstressed, it is even more a surprise when components are found defective [5].

SCC was found to occur in all engineering solids, in a wide range of environments, and in a wide range of compositions and solid-state structures within the material classes [6].

1.2.1 Crack Morphology

Stress corrosion cracks give the appearance of a brittle mechanical fracture [7].

Both intergranular and transgranular SCC are observed. Intergranular cracking proceeds along grain boundaries, while transgranular cracking advances without apparent preference for boundaries. Cracking proceeds generally perpendicular to the applied stress [7].

1.2.2 SCC Mechanisms: Initiation and Propagation

Since 1950, there was a major effort to find the mechanism of SCC and despite the researchers huge work there is no definite single mechanism of SCC till now [5,6].

1.2.2.1 Mechanism of SCC Initiation

SCC initiation is a poorly understood and loosely defined phenomenon necessitating further work for the development of a clear mechanistic interpretation [6].

Some investigators [4,5] suggested that tensile stress is usually the main factor responsible for SCC initiation; stress initiates SCC as follows:

The application of tensile stress to a metal component with smooth surface results in movement of dislocations in the metal crystals. This movement will be

halted when they reach either the surface of the metal or a grain boundary. The “pile-up” of dislocations at the surface forms slip step and it is at this site that the material is most vulnerable to initial corrosive attack [5].

Alloys which rely on thin films of oxide or other material for their corrosion protection are especially vulnerable because the slip step uncovers a microscopic quantity of bare metal which is highly anodic compared to the surrounding surfaces. Such process is shown in Figure 1.1.

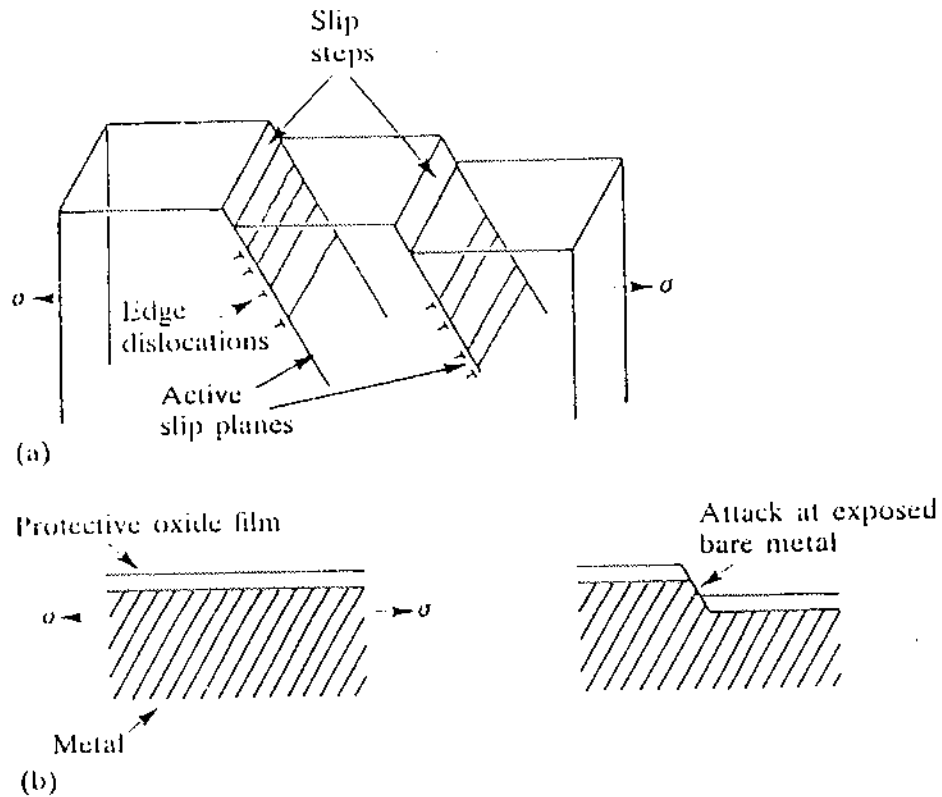


Figure 1.1: The role of slip steps in SCC [5];

- (a) The formation of slip steps on the surface of a metal by movement of dislocations along active slip planes under the action of tensile stress.**
- (b) A slip step on the surface of a passivated metal creates an active site for the initiation of a stress-concentrating pit [5].**

If the metal is able to repassivate quickly, then little danger ensues, but if the passivation time is long enough to allow corrosion of the exposed area to occur and a pit to form, then the criteria for commencement of SCC have been fulfilled.

Even in metals which are not passivated, the formation of slip steps on the surface represents a corrosion problem for the discontinuity of crystal structure causes local anodes. Investigators place great importance on pit formation in which a porous cap of corrosion product creates a tiny concentration cell. The aggressive species are concentrated within the confines of the cell to form an even more aggressive local environment. The diffusion process operates up a concentration gradient because the energy change which results from the dissolution process at the exposed bare metal is so favourable [5].

Considerable evidence has been accumulated that the local environment inside pits and cracks becomes very acidic, sometimes as low as pH 2, a factor which not only accelerates the dissolution processes, but adds considerably to the weight of evidence for the involvement of hydrogen in most, if not all, SCC mechanisms. As pH falls, the thermodynamic tendency for hydrogen evolution becomes greater at constant potential. The potential is not constant, however. The creation of an oxygen concentration differential causes an anodic polarization, which further increases the hydrogen evolution tendency [5].

The formation of a pit, then, is the usual precursor to SCC, and represents the initiation phase. It is most unpredictable aspect of this form of corrosion: It may take anything from days to many years to occur. Even then, a pit may not be of the size and shape necessary to promote a crack in the pit bottom. However, once a pit has been produced with the right conditions, crack growth begins and the propagation phase takes over [5].

There are many instances of SCC not being initiated at pits, for example, mild steels in nitrates hydroxides and bicarbonates. The SCC initiators in these cases are localized cracks at the grain boundaries. In all of these environments, the grain boundaries are attacked because of chemical heterogeneity [4,5].

Many workers place little importance upon initiation process, and take the view that in normal engineering materials there are almost always enough material defects and surface irregularities present as a result of the fabrication

process to provide ample initiation sites [5]. So some of them suggest “initiation” is solely that time necessary to achieve a localized environment in any pre-existing defect on the material surface in which the defect may result from machining, scratches, pits, intergranular corrosion notches etc. [8], the others deal only with propagation and treat initiation time as effectively zero [5].

1.2.2.2 Mechanisms of SCC Propagation

Many mechanisms have been proposed for the cracks propagation in SCC [5,6,8], but three are generally applicable and will be described here. These are:

1. Pre-existing active paths;
2. Slip-dissolution;
3. Adsorption-related mechanisms

1. Pre-existing Active Paths Mechanisms

In this mechanism, propagation is believed to occur preferentially along active grain boundary regions. Grain boundaries may be anodically polarized for a variety of metallurgical reasons, such as the segregation or denudation of alloying elements. It is quite possible that dislocation pile-up due to the tensile stress can produce the same effect [5].

The Mechanism can be viewed predominating in cases where the SCC is governed by electrochemical or metallurgical considerations, rather than the influence of stress.

Evidence in favor of such a mechanism is extensive. Most alloy systems in which grain boundary precipitates are present fail by intergranular cracking [5].

2. Slip-Dissolution Mechanism

In this mechanism, slip is hypothesized to break the protective film with the result that a dissolution transient occurs of sufficient magnitude and with

sufficient frequency that a stress corrosion crack propagates. Here, the major advance of the SCC occurs via the dissolution process [6].

The unit process of slip dissolution is conceived of as a break of the protective film followed by a transient dissolution process that consumes sufficient material to advance the SCC. This is illustrated in figure 1.2 [6].

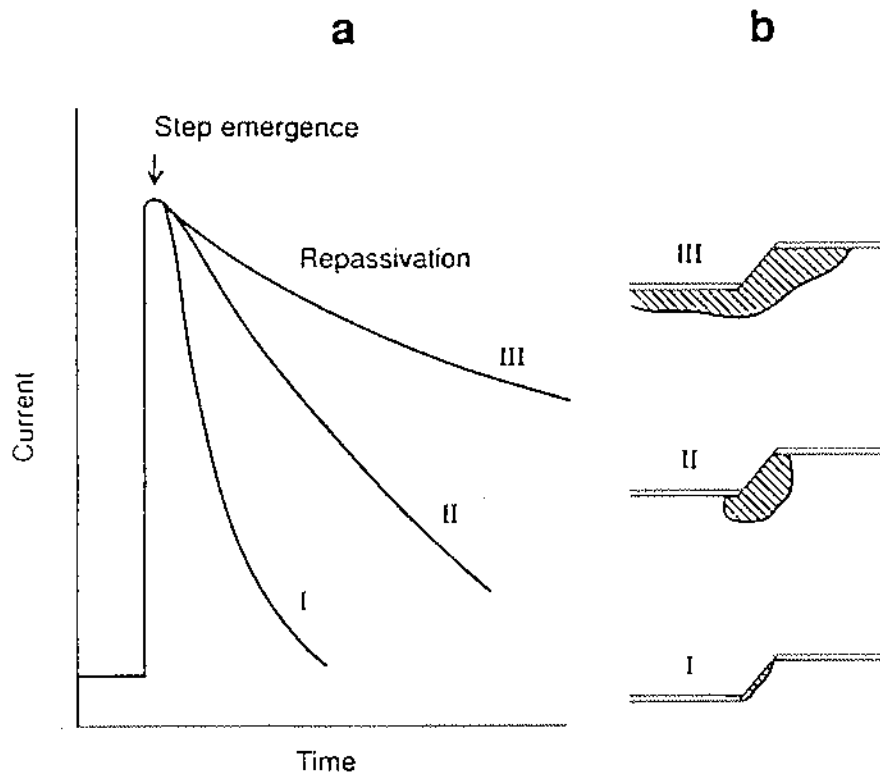


Figure 1.2: Schematic view of the processes associated with the initial stages of slip dissolution. The general shape of the individual current transient is shown (a) as associated with the morphologies of corrosion associated with slip events (b) [6].

The possible rate-controlling phenomena that can govern crack propagation by the slip-mechanism are as follows: liquid diffusion of either solvating water molecules or solvated cations to and from the crack-tip region, the overall oxidation (dissolution and oxide growth) rate, and the oxide or film-rupture rate at the crack tip itself. These fundamentally important parameters can be directly related to the stress, environment, and microstructure known from empirical observations to be the conjoint requirements for SCC (figure 1.3). In

turn, these rate-controlling phenomena are functions of myriad of system parameters such as dislocation morphology, solution viscosity, etc. [8].

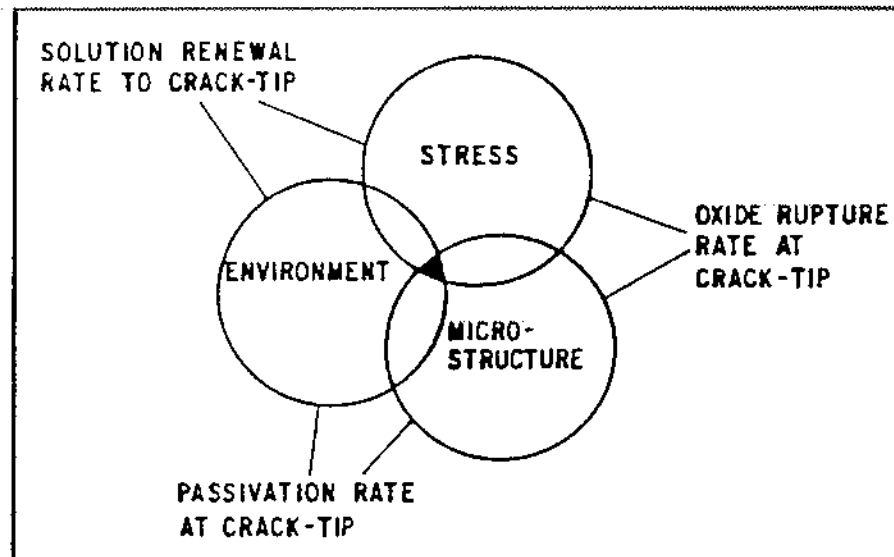


Fig. 1.3: Interrelationship between the fundamental controlling parameters (mass transport rate, passivation rate, and oxide rupture rate) and the phenomenological parameters (stress, environment, and microstructure) known to affect SCC [8]

3. Adsorption-Related Mechanisms

In the past it was thought that hydrogen embrittlement could be distinguished from “pure” SCC because the hydrogen effect could only occur if the specimen was cathodically polarised. Recently, evidence has been found that in the confines of a crack, the solution composition may bear little resemblance to that existing in the bulk. Thus even though a bulk material may seem to be outside the potential range for the evolution of hydrogen, the combination of pH and potential existing at the crack tip may allow such a cathode reaction. Thus the role attributed to hydrogen in the mechanism of SCC has increased greatly in latter years and care should be exercised in the interpretation of results [5].

Adsorption-related mechanisms propose that active species in the electrolyte degrade the mechanical integrity of the crack tip region, thus facilitating fracture of bonds at energies much lower than would be expected.

In one mechanism, the aggressive ion which is specific for the case in question is thought to reduce the bond strength between metal atoms at the crack tip region by an adsorption process which results in the formation of metal-species bonds. The energy used in binding the aggressor to the metal atoms reduces the metal-metal bond energy allowing mechanical separation to occur more easily. It is possible that the specific ion (which is normally unreactive towards the metal) is more reactive because of the increase of thermodynamic energy which occurs in the metal-metal bond as a result of the tensile stress. Figure 1.4a illustrates, in schematic form, a possible mechanism [5].

A second adsorption-related mechanism is based upon the formation of hydrogen atoms by the reduction of hydrogen ions within the crack. The hydrogen atoms are adsorbed by the metal and are thought to cause weakening, or embrittlement, of the metal-metal bonds just beneath the surface at the crack tip. A number of possibilities exist by which this can occur. These are shown in figure 1.4b. One presupposes the formation of metal hydrides, discrete chemical species which are well known for their brittle nature. In the past, hydride formation has been probably the most favored mechanism for the general phenomenon of hydrogen embrittlement, though other mechanisms have been proposed. For example, it is also possible that decrease of the bond strength occurs by an adsorption process similar to that described for the specific ion [5].

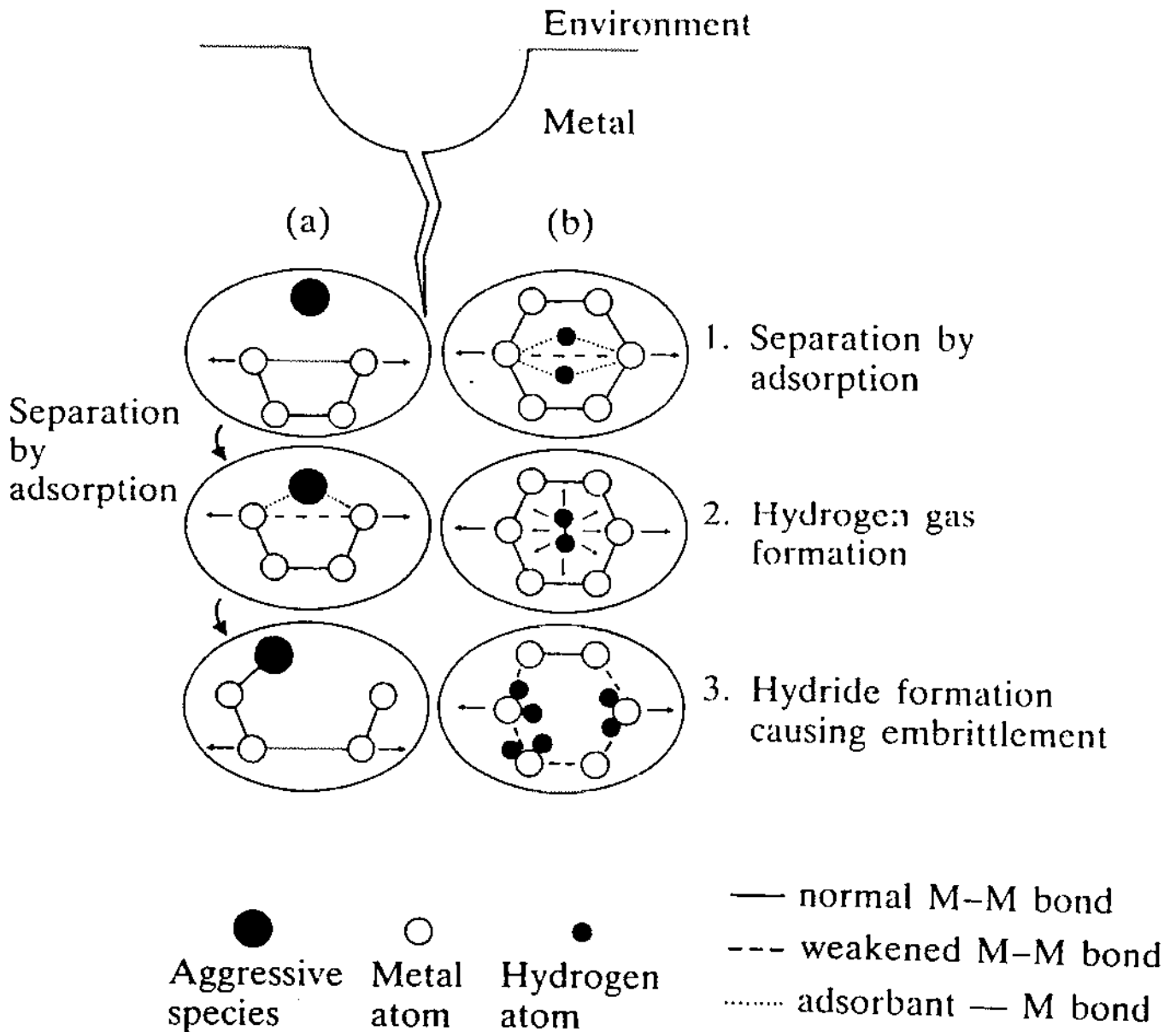


Fig. 1.4: Mechanisms for the propagation of cracks [5]:

- (a) The aggressive species is adsorbed at the crack tip and causes a reduction in the metal-metal bond strength.
- (b) (1) Separation by adsorption of hydrogen is locally dilated areas immediately ahead of the crack tip
- (2) Hydrogen gas formed in locally dilated areas or along slip planes. Pressure of gas assists in rupture of metal-metal bonds.
- (3) Formation of metal hydrides causes reduction in metal-metal bond strength and embrittlement of region ahead at crack tip [5].

The exact mechanism of SCC is still not well understood or known. Parkins [9] suggested that a “stress corrosion spectrum” probably existed that logically graded the SCC systems between those that were mechanically dominated (e.g., hydrogen embrittlement of high-strength steels) to those that were environmentally dominated (e.g., pre - existing active path attack in the carbon steel/nitrate system). Indeed, it was suggested that two mechanisms may operate in one alloy/environment system with a dominant mode being determined by relatively small changes in the material, environment, or stressing conditions [9].

1.2.3 Crack/Crack-Tip Processes

The processes that are involved in the enhancement of cracking in metals and alloys by aqueous environments (electrolytes) are as follows [10]:

1. Transport of deleterious species to the crack tip.
2. Reactions of the electrolyte with newly produced surfaces (e.g., slip steps and cracks) to affect dissolution, or to produce hydrogen.
3. Transport of metal cations away from the crack tip to enable continued dissolution.
4. Hydrogen entry (or adsorption).
5. Diffusion and partitioning of hydrogen to the various fracture (or embrittlement) sites.
6. Embrittlement reaction (breaking of M-H-M bonds).

These processes operate in sequence. The overall rate is governed by the slowest of these processes, operating in conjunction with the mechanical driving force for cracking. The mechanical driving force is characterized by the crack-tip stress or stress-intensity factor and crack-tip strain rate, or indirectly through the applied stress or specimen extension rate [10].

The first step, or transport processes, really involves not only the transport of deleterious species to the crack tip, but also of the other species to and away

from the crack tip. The transport processes in aqueous media include electromigration, diffusion, and convection. Coupled with reactions at the crack tip and along the crack flank, they define the local environment within the crack and at the crack tip. The near-tip region of interest may extend from 1nm to 0.1mm from the crack tip, and the overall crack depth may range from about 0.1 μ m to tens of millimeters, to span crack initiation to crack growth [10].

The anodic processes (dissolution reactions) in the second step are directly responsible for crack initiation and crack growth enhancement, according to various dissolution mechanisms for SCC. To sustain dissolution-controlled crack advance, transport processes must be sufficient to maintain local crack-tip chemistries that promote dissolution. For the hydrogen embrittlement mechanisms, on the other hand, dissolution reactions function primarily as sources of electrons for the reduction of hydrogen.

The remaining processes are important only for the hydrogen embrittlement, and governs the rate of supply and distribution of hydrogen to the potential embrittlement sites and the rate of embrittlement [10].

1.2.4 SCC Testing

SCC experiments can be classified into three different categories:

1. Tests on statically loaded smooth specimens [4].
2. Tests on statically loaded precracked specimens (Fracture mechanics testing) [4,6].
3. Tests using slowly straining samples [4,6].

1. Tests on statically loaded smooth samples

These tests are usually conducted at various fixed stress levels, and the time to failure of the sample in the environment is measured. Figure 1.5 illustrates the typical results obtained for this type of test. In figure 1.5, the logarithm of the

measured to failure, t_f , is plotted against the stress, σ_{applied} , and the time to failure can be seen to increase rapidly with decreasing stress; a threshold stress, σ_{th} is determined where the time to failure approaches infinity [4]. The total time to failure at a given stress consists of the time required for the formation of a crack (the incubation or initiation time), t_{in} , and the time of crack propagation, t_{cp} . These experiments can be used to determine the maximum stress that can be applied to avoid SCC failure, to determine an inspection interval to confirm the absence of SCC crack propagation, or to evaluate the influence of metallurgical and environmental changes on SCC. However, the actual time for the formation or initiation of cracks is strongly dependent on a wide variety of parameters, such as surface finish and prior history. If a crack like flaw or a crevice is present in the material, then the time to initiate a crack may be dramatically reduced [4].

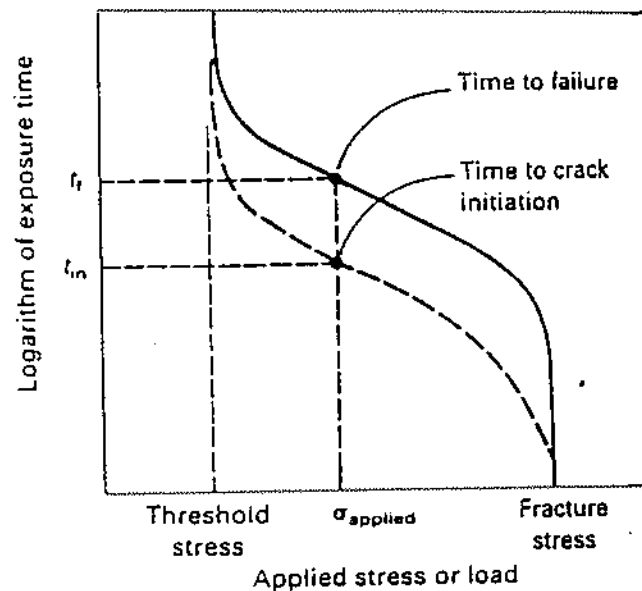


Fig. 1.5: Schematic of a typical time to failure as a function of initially applied stress for smooth sample SCC tests [4]

2. Fracture Mechanics Testing

Fracture mechanics testing provides direct information on the velocity of SCC under known conditions of stress and defect depth. Essential features of fracture-mechanics-type testing have by now, been well defined, and such testing is in widespread use [6].

A typical fracture mechanics specimen is illustrated in figure 1.6. The depth of the crack is determined as a function of time and the stress intensity is known from the crack depth and the load. The data are then plotted as crack velocity (m/s) versus stress intensity ($\text{MPa}\sqrt{\text{m}}$). The resulting data usually take the form shown in figure 1.7 where there are often well-defined stages 1,2 and 3 [6].

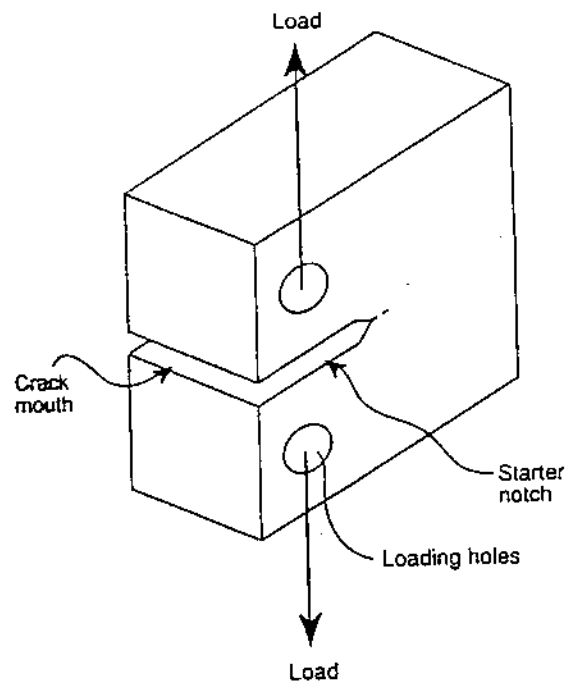


Fig. 1.6: Typical configuration of specimens used for fracture-mechanics type testing where crack velocity versus stress intensity is obtained

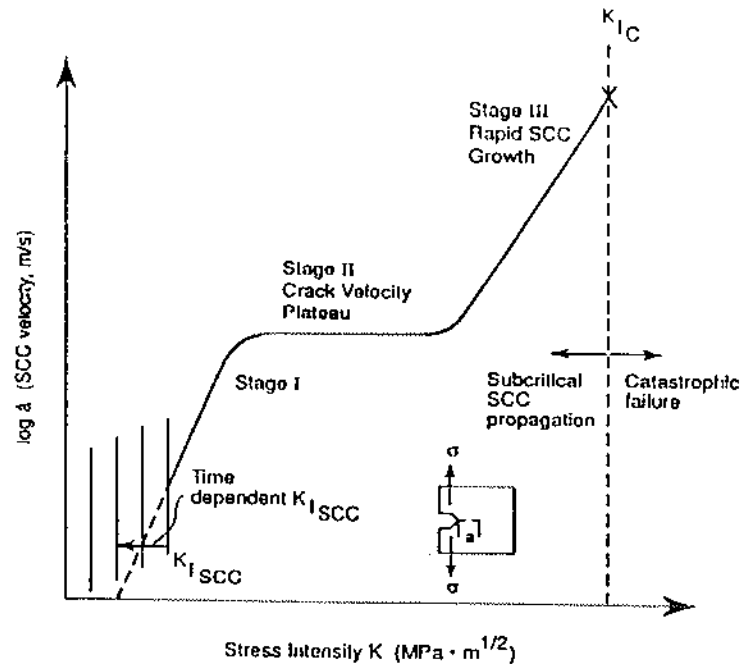


Fig. 1.7: Schematic plot of data from fracture-mechanics type testing where the log of crack velocity is plotted versus log of stress intensity. Above the value of K_{IC} fast or catastrophic fracture occurs. The minimum stress intensity below which SCC does not occur is shown by $K_{I,SCC}$. Here, $K_{I,SCC}$ is shown to depend on time [6]

In general, most testing is performed in the opening mode (tension mode) which is designated as Mode I, although some work has been done using Mode II (shearing mode) and Mode III (tearing mode) [6,11].

Of great interest to design with SCC from the fracture mechanics point of view is the stress intensity below which SCC will not initiate. This is usually designated as $K_{I,SCC}$ meaning that it is the stress intensity in the opening mode below which SCC will not initiate. It is presumed that once SCC starts, it will propagate at increasing rates following first the Stage 1 curve and then the Stage 2 curve until stress intensity is sufficiently great that K_{IC} is reached and catastrophic failure is reached. This is illustrated in figure 1.7 [6].

There are several problems in using the fracture mechanics approach. One is that the minimum stress intensity below which SCC does not occur ($K_{I,SCC}$) is often time dependent, so that over the lifespan of a component, there may never be a stress low enough to avoid SCC in certain environments (figure 1.8) [6].

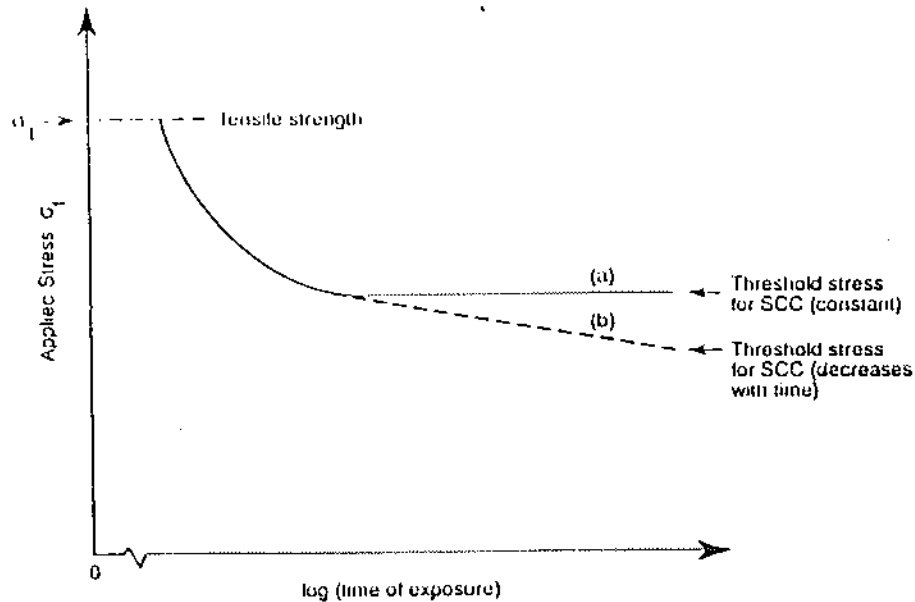


Fig. 1.8: Schematic plot of stress to fail versus time for static-loaded SCC experiments showing both a constant stress below which SCC does not occur and a lower limit that decreases with time. Curve (a) shows no time dependence of the lower limit; curve (b) does [6]

3. Slow-Strain-Rate Testing

The slow-strain-rate testing provides a method for avoiding the uncertainties of initiating SCC. It has the advantage of determining whether SCC will occur under any circumstance in a given environment. It is a characteristic of these experiments that strain rates that are too rapid fail, as in tensile tests by mechanical overload, the strain rates must be sufficiently slow, usually less than $10^{-6}/s$, so that the SCC processes can operate. Figure 1.9 shows some typical results in a plot of ductility ratio versus log strain rate [6].

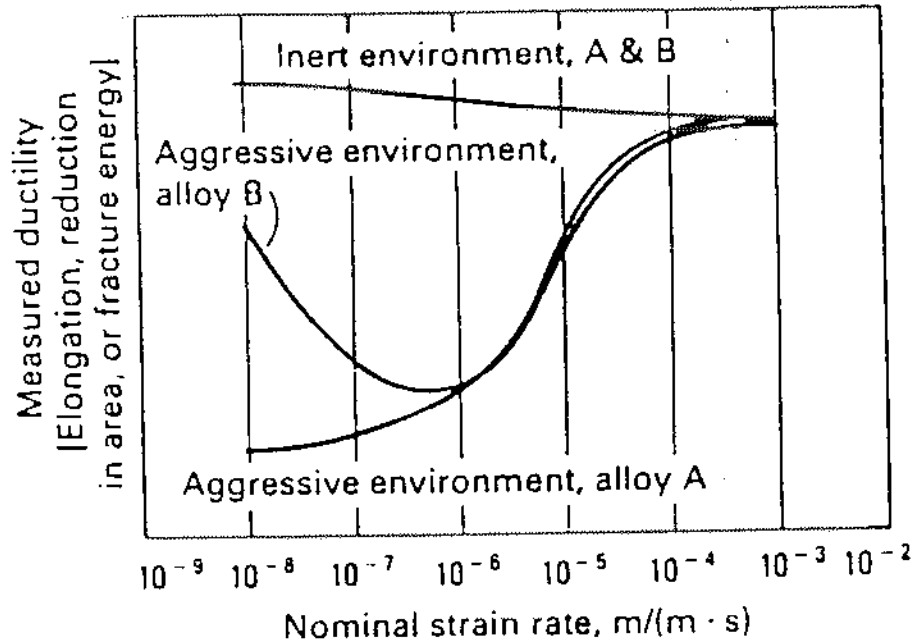


Fig. 1.9: Strain to failure plots resulting from slow-strain rate testing [4]

The output from these slow-strain-rate experiments, i.e., the dependent variable, may be an area or percent of fracture surface with SCC, time-to-fail, load to failure, or SCC velocity (length of SCC propagation divided by time-to-failure). These results are generally equivalent. Where SCC velocity is determined, it is usually inferred that this is the "plateau velocity" [6].

Slow-strain-rate testing provides mainly a qualitative indication of SCC intensity but does not give directly useful information on design stresses, initiation, and the part of the SCC velocity profile that is actually operating [6]. Nevertheless it is an excellent test for comparing the relative susceptibility of alloys to cracking in an environment or for studying the influence of metallurgical variables on the susceptibility of an alloy [4].

1.2.5 SCC Controlling Parameters

The fundamental controlling parameters of SCC are [4,8]:

1. The material.
2. The stress.
3. The environment.

1.2.5.1 Material

It is now clear that all materials sustain SCC regardless of heat treatment. There is no such condition as a heat treatment that renders an alloy immune from SCC. Some heat treatments reduce or eliminate the SCC susceptibility in some environmental conditions but do not prevent the alloy from sustaining SCC in all environments. Such different environments do not need to be as drastically changed as, for example, from aqueous to liquid metal; rather, slight changes in pH or in electrochemical potential may be sufficient to increase substantially the SCC susceptibility for a heat treatment that had previously been considered immune [6].

Increasing the alloy strength was found to increase the SCC susceptibility and decrease K_{IC} . This effect of increasing strength increasing SCC susceptibility seems to persist in many alloys despite the basic composition or how the high strength is achieved; e.g., whether the strength is achieved by transformations or by precipitation. This general pattern of increasing strength increasing sensitivity to SCC and decreasing K_{IC} follows generally in titanium and aluminum base alloys [6].

It was found that even pure metals are susceptible to SCC. These metals usually sustain SCC transgranularly [6].

In general there is no metal immune to SCC [6].

1.2.5.2 Stress

The requirement for tensile stress is beyond doubt. SCC is the synergism of stress and corrosion: the absence of either eliminates the problem [5].

It is well known that SCC susceptibility increases generally with stress. If the stress is zero, SCC does not occur.

The tensile stress types are: applied, residual and stresses due to corrosion products [5,6]. It was found that approximately 80% of all SCC failures are related to residual stresses. It was also reported that the stresses produced by corrosion products can be substantial and sufficient to cause surrounding materials to exceed their yield strengths, and often produce continuous, inexorable straining of the kind analogous to the slow-straining SCC test [6].

Compressive stresses do not cause SCC [4,5]. In fact, shot peening is used in practice to reduce the possibility of SCC [1,2,5].

1.2.5.3 Environment

It is now clear that SCC is a more general phenomenon with respect to environments in which SCC occurs and that the SCC susceptibility in a single alloy varies regularly with changes in composition of the environments [6].

Generally, designers define the environment to which their materials of construction are exposed as the nominal ones or ones that are predisposed to make their components and materials perform ideally. Whereas, the non-nominal environments involve the same nominal environment chemistry but as modified in crevices, deposits, heat-transfer surfaces (with and without crevices), zero flow, eddies, and wetting and drying etc. Such conditions usually produce environments on the exposed component surface that are vastly different from the nominal conditions under which their component were designed. These aggressive conditions can alter pH or/and potential for the material/environment system which greatly influence the SCC susceptibility as shown in figure 1.10. The pattern of figure 1.10 can apply to any metal-environment system [6].

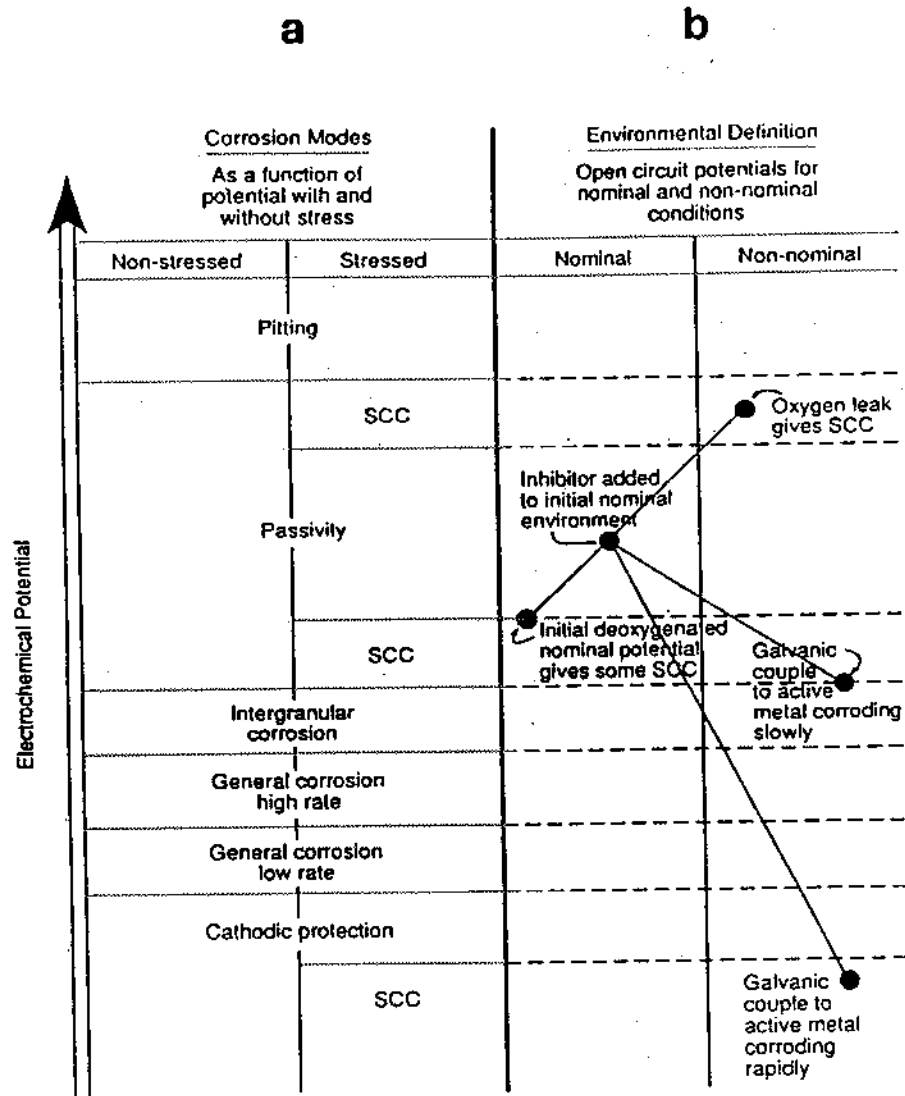


Figure 1.10: Schematic view of relationship between modes of corrosion and environmental definition as a function of increasing oxidizing electrochemical potential. (a) Corrosion modes as a function of potential with and without stress. (b) Environmental definition as possible open-circuit potentials that might occur in nominal and non-nominal environments. All conditions here are hypothetical [6]

It is now clear that pure aqueous environments without any ionic contamination or intentional ionic content produce SCC whether the potentials are above or below the hydrogen equilibrium. For cases below the hydrogen equilibrium, it might be expected that hydrogen-related SCC would occur in pure water owing simply to the availability of hydrogen from the electrochemical reduction of water. However, SCC occurs in pure water above the hydrogen equilibrium potential in sensitized stainless steels [6].

Virtually all environments to which metals are exposed will produce SCC of some intensity under some circumstances, with changes in such variables as pH and potential for aqueous environments and vapor pressure for gaseous environments [6].

1.2.6 SCC of High-Strength Aluminum Alloys

SCC of high-strength aluminum alloys is almost exclusively intergranular (IG) [1,3,4,12] unless the mechanical loading conditions are severe and/or the local grain boundaries (GBs) are unfavorably oriented [12].

The wrought products of commercial alloys for which the grains are very much elongated in the direction of rolling exhibit SCC resistance strongly related to the grain shape and orientation with respect to the applied stress, if the stress is parallel to the longitudinal direction, the path of the crack in this direction is most difficult and thus resistance to cracking is high. Whereas, if the stress is parallel to the short transverse direction, corrosion easily follows the path of the long, thin grains and cracking is rapid. Resistance to cracking in long transverse direction is somewhat intermediate (figure 1.11) [1,3,4,13].

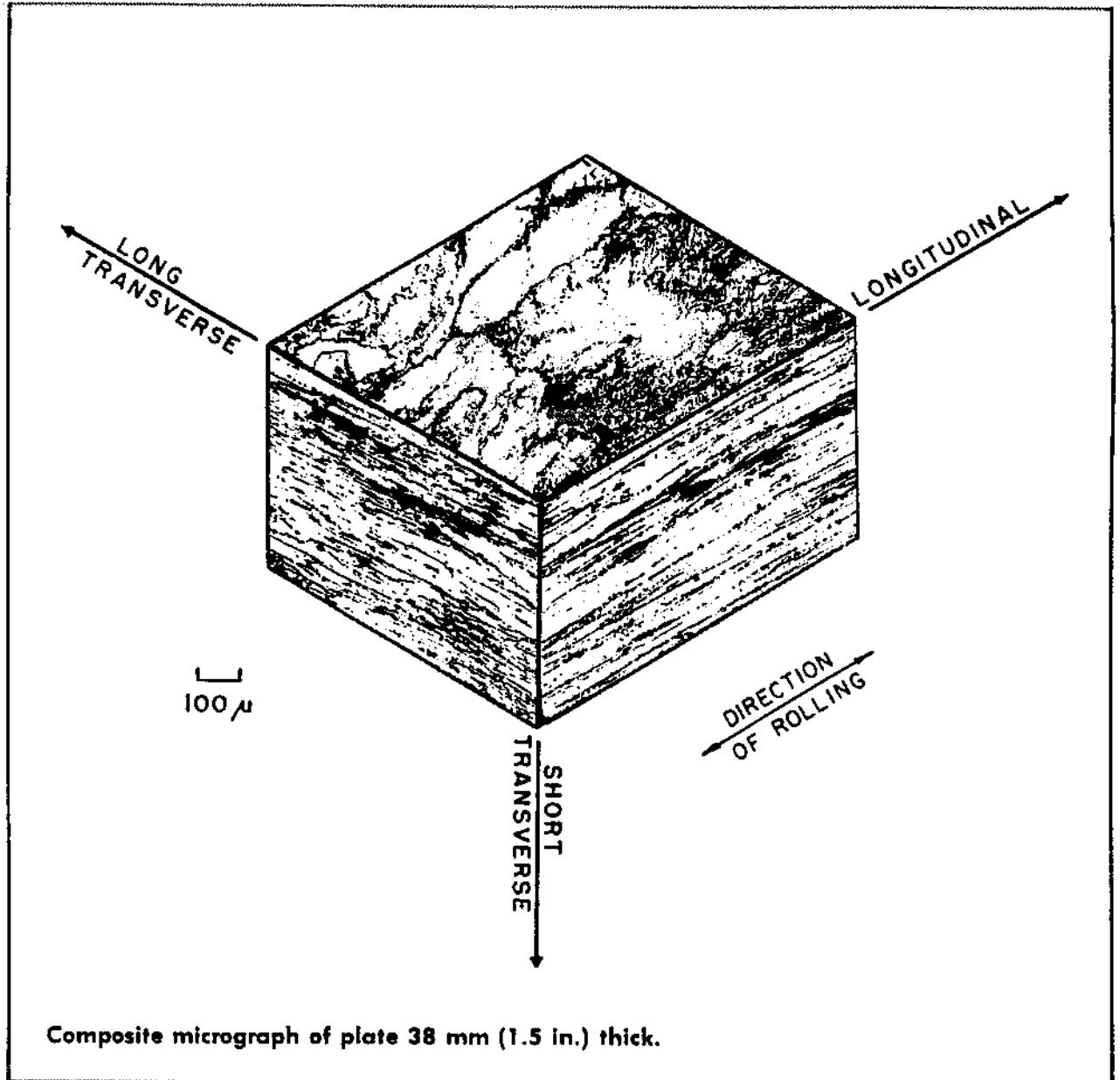


Fig. 1.11: Three-dimensional micrograph showing grain structure of alloy 7075-T6 plate [3]

The effect of grain orientation on the SCC of 7075-T6 plate is shown in figure 1.12. It is obvious that 7075-T6 SCC resistance is highest in the longitudinal direction and lowest in the short transverse direction [1,3,4,13].

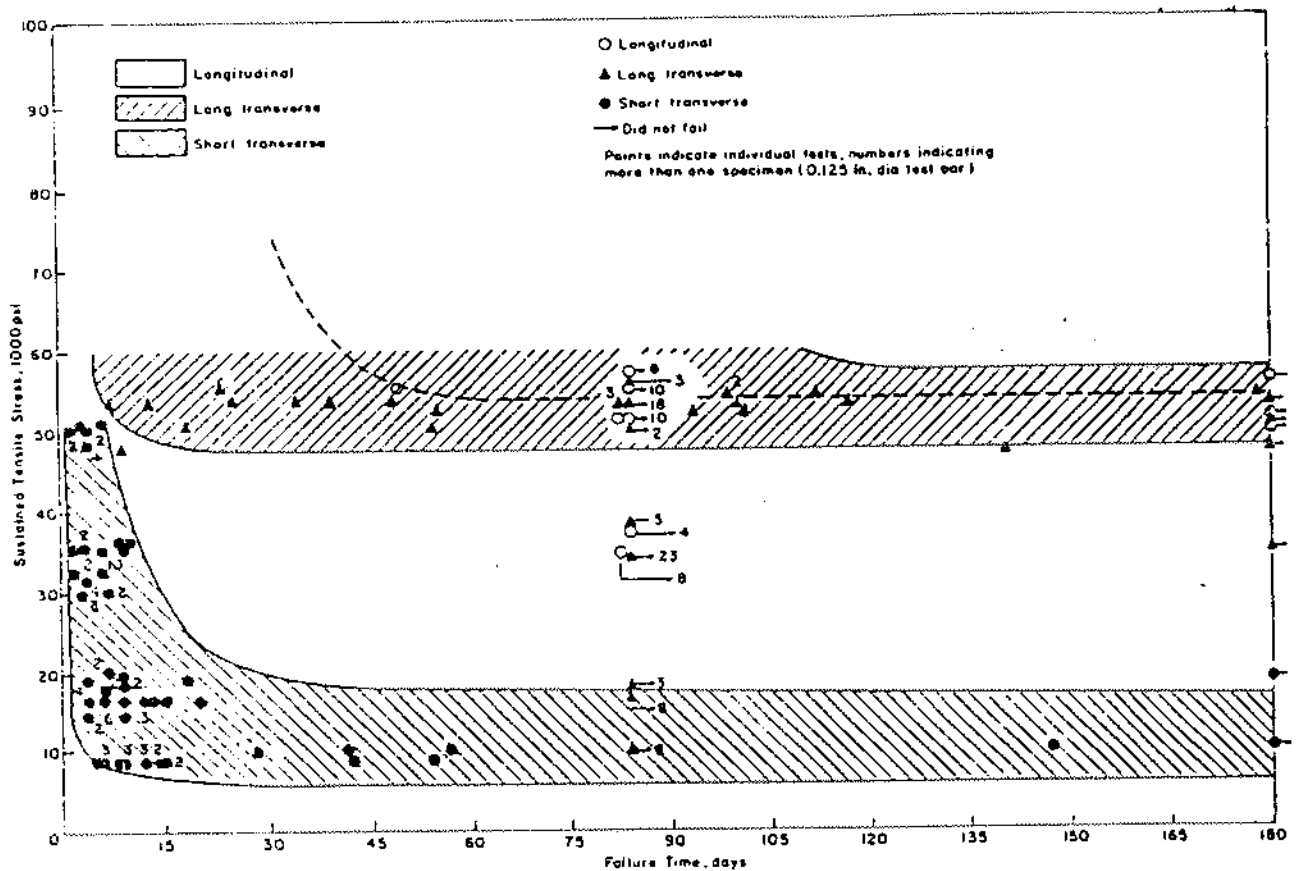


Fig. 1.12: Resistance to SCC of 7075-T6 plate in 3.5% NaCl solution as influenced by direction of loading [1]

Although numerous detailed SCC mechanisms abound, two basic theories have evolved. One envisages cracking as highly localized anodic dissolution of GB regions under the combined influence of stress and environment; the other suggests embrittlement and loss of ductility is promoted after the ingress of an aggressive species (usually atomic hydrogen) onto or into the alloy. Some investigators concluded that the SCC of 2XXX (Al-Cu) and 5XXX (Al-Mg) series alloys occurs via an anodic dissolution mechanism, whereas mechanistic attributions for 7XXX [Al-Zn-Mg(-Cu)] series remain controversial [12].

Many investigators favored anodic-dissolution-based SCC mechanisms for all aluminum alloy systems until the work of Gest and Troiano triggered

numerous studies showing that hydrogen embrittlement (HE) generally has a role in the SCC of 7XXX series aluminum alloys and may be involved for 2XXX and 5XXX series alloys [12].

SCC of aluminum alloys is a complex process involving a multiplicity of time-dependent interactions that remain difficult to evaluate independently. SCC to date eludes a complete mechanistic interpretation. Nevertheless, considerable progress has been made over the last two decades, with most resulting from the availability of sophisticated experimental techniques involving high-resolution electron microscopy, electrochemistry, and the implementation of fracture mechanics [12].

1.2.6.1 Microstructural Aspects

The SCC susceptibility of 2XXX and 5XXX series alloys is directly related to the development of local anodes and cathodes within GB regions [12].

The general dependence of SCC susceptibility upon iso-thermal aging is well established for 2XXX, 7XXX series alloys and the Al-Li-Cu-Mg alloy 8090 as shown in figure 1.13 and table 1.1.

Table 1.1: Dependence of SCC resistance upon thermal aging condition for precipitate-hardening aluminum alloys

Microstructure	Susceptibility
Solution Treated (No GB precipitation)	Immune
Underaged	Maximum
Peak Aged	Susceptible
Overaged	Decreases with aging

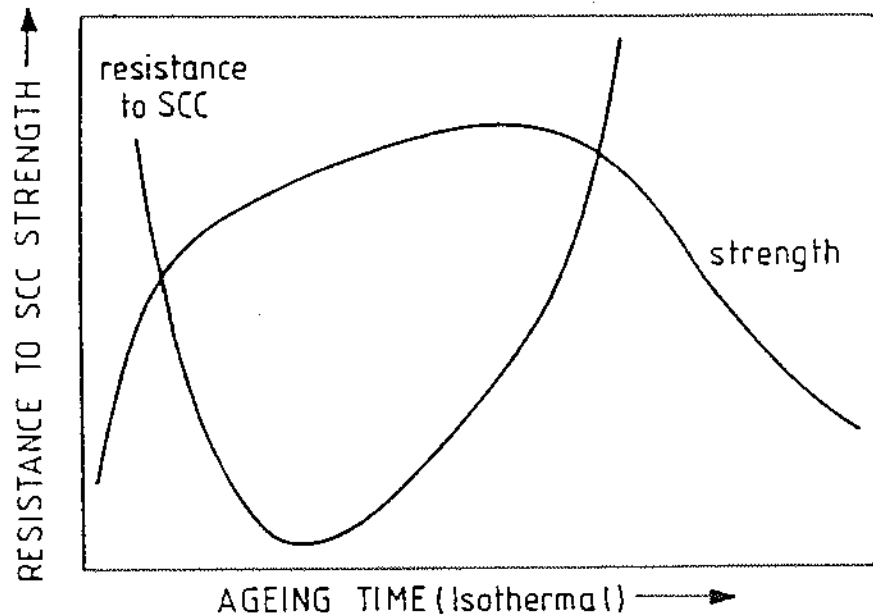


Fig. 1.13: Typical dependence of SCC resistance upon thermal aging condition for precipitate-hardenable aluminum alloys [12]

For 7XXX series alloys, numerous attempts to correlate susceptibility with microstructural features known to be influenced during thermal treatment include the following:

1. Grain-boundary precipitate size and spacing;
2. Precipitate free zone (PFZ) width or preferential slip in the PFZ;
3. Solute profiles in the PFZ; and
4. Grain-boundary segregation [12].

No single microstructural parameter has yet been identified that uniquely controls the SCC of 7XXX series alloys [12].

Middleton and Parkins suggested SCC propagation in Al-Zn-Mg alloys occurs via $MgZn_2$ GB precipitates acting as an active path for corrosion with creep preventing the formation of an effective passivating film over the crack tip and allowing dissolution of the material between the GB precipitates [12].

Concurrent with attempts in the mid-1970s to characterize SCC in terms of GB precipitation, other workers focused attention upon the solute content of the GB region away from precipitates. Lormier and Ruder suggested that the solute profiles within the PFZ may provide an electrochemical sensitive path [12]. Dix suggested that SCC occurs by ductile tearing of the soft PFZ, but Pugh and others noted that such ductility should be visible on fracture surfaces; in fact this is not obvious [14].

During the late 1970s and early 1980s, considerable effort was made to determine whether GB segregation of magnesium influenced the SCC of Al-Zn-Mg(-Cu) alloys. It was found that magnesium oxide developed preferentially at GB surface inter-sections during solution treatment and was thought to be responsible for accelerated hydrogen entry and subsequent embrittlement under tensile stress (pre-exposure embrittlement). This result led some researchers to propose that magnesium was also enriched in ambient oxide films adjoining GBs and that magnesium was segregated at GBs within Al-Zn-Mg(Cu) alloys [12].

1.2.6.2 Environmental Aspects

Almost all published SCC data for aluminum alloys (2XXX, 5XXX, 7XXX, and Al-Li-based alloys) involves cracking in aqueous saline environments. Some data is available for 7XXX series alloys in the following environments:

- A. Various Gases.
- B. Aqueous environments containing halide anions other than chloride or nonhalide anions.
- C. Nonaqueous organic environments [12].

A. Gaseous Environments

The influence of gaseous environments upon the SCC of Al-Zn-Mg(-Cu) alloys is as follows:

1. SCC neither initiates nor propagates in dry gases.
2. SCC initiates almost immediately in wet gases when precracked specimens are subjected to stress-intensity factors approaching K_{IC} .
3. SCC fracture morphology is IG with crack growth kinetics controlled by the humidity level and independent of gas composition [12].

On the basis of the above it was suggested that a hydrogen embrittlement (HE) mechanism must control the SCC of 7XXX alloys in moist, gaseous atmospheres [12].

B. Aqueous Environments

1. Halide Solutions:

Published information on the influence of environmental parameters, such as solution composition, pH, temperature, viscosity, etc., and potential on SCC in halide solutions is limited, almost exclusively, to that available for 7XXX series alloys [12].

**Solution Composition:* It was proposed that the presence of cations other than Hg^{+2} and H^+ , e.g., Li^+ , Na^+ , K^+ , Rb^+ , Al^{+3} and NH_4^+ , have little or no specific effect upon the SCC growth kinetics for 7XXX series alloys in neutral aqueous chloride solutions [12].

Some investigators reported that SCC crack growth rates increase with increasing halide ion concentration above around $10^{-2} M$ and are not significantly different in Cl^- , Br^- , and I^- solutions of given anion concentration [12].

Detailed study of the influence of halide ion concentration upon SCC initiation has not been reported [12].

**Solution pH:* There is general agreement that the occurrence of SCC in high strength aluminum is reduced as saline solutions become more alkaline [12].

**Solution Temperature:* A change in SCC performance of 7XXX series alloys at temperatures around $40^\circ C$ is a common experience, and is found to be associated

with surface film characteristics, which also change significantly at temperatures around 40°C. An example of such behavior is shown in figure 1.14 [12].

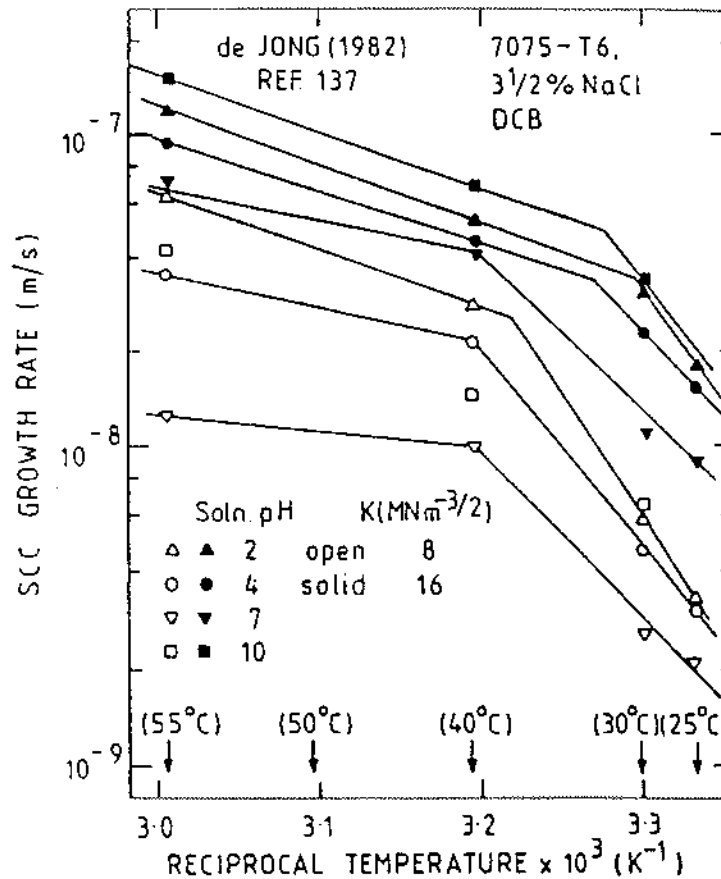


Fig. 1.14: Arrhenius plots using SCC crack growth rates for 7075-T6 in 3% NaCl at various temperatures and solution pHs [12]

* *Electrochemical Potential:* Under appropriate anodic polarization conditions, all medium and high strength aluminum alloys can suffer SCC. Even commercial 6XXX series alloys (Al-Mg-Si) are susceptible to SCC [12].

Some researchers investigated the effect of electrochemical potential upon SCC crack growth rates. Typical crack velocity-stress-intensity factor (cv-k) curves for 7XXX series alloys in halide solution is given in figure 1.15. These data imply for a given k, a crack velocity in Stage 1 decreases as anodic

polarization decreases, while a Stage 2 crack velocity remains constant at high levels of anodic polarization [12].

The influence of crack-tip potential on SCC is complex and is not well-established [12].

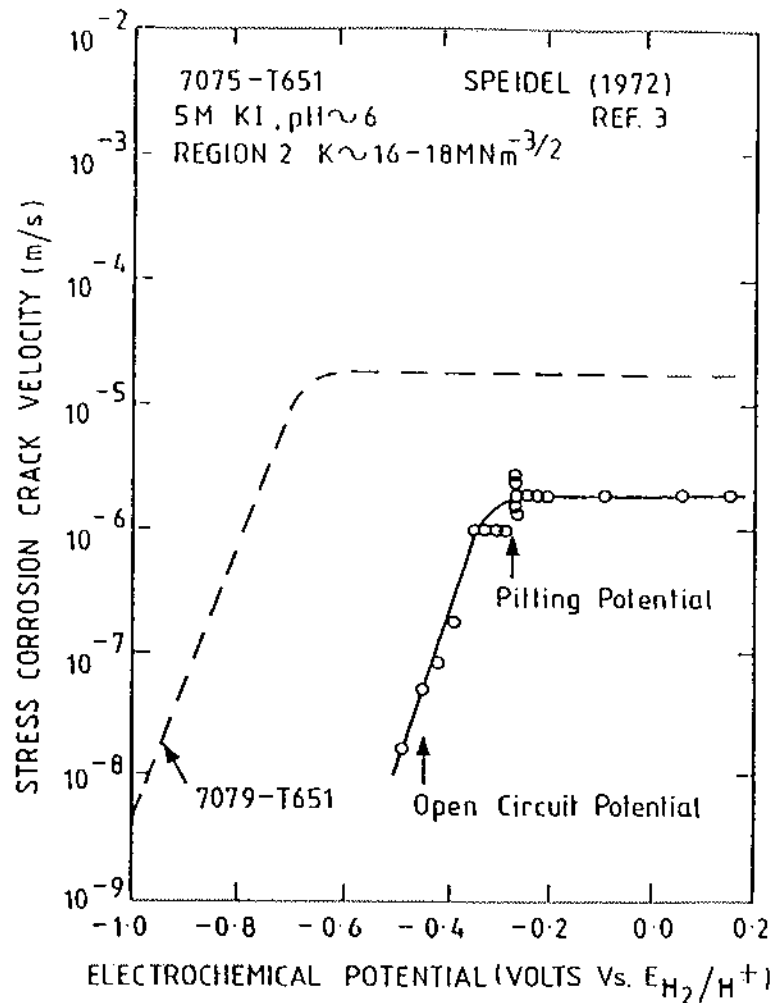


Fig. 1.15: The effect of imposed electrochemical potential on SCC crack growth rates for 7079-T651 and 7075-T651 in an aqueous 5M potassium iodide solution [12]

2. Nonhalide Solutions

It was reported that 7075-T6 alloys suffer SCC in aqueous sulfate, nitrate, perchlorate and chromate solutions, with significant SCC being promoted in both sulfate and nitrate solutions [12].

3. Environmental Conditions Within Cracks

It is well known that the environmental conditions developed within cracks generally differ significantly from bulk conditions and that a detailed knowledge of these solution chemistries should help elucidation of the mechanisms of SCC in aluminum alloys [12].

**Solution pH:* Direct in situ measurement of local environmental conditions within cracks for 7075-T6 exposed to a bulk 4.5% potassium chloride solution revealed that solution pHs within cracks moved to values around 3.5 to 4.0 independent of bulk pHs in the range of 2 to 10 [12].

It is also well established that the pH inside the cracks in aluminum alloys falls to near 3 in chloride solutions [12,15].

**Crack/Crack-Tip Chemistry:* For 7075-T6 alloy exposed to 3.5% NaCl, the solution found within the cracks was a gelatinous solution containing aluminum hydroxychloride complexes such as $\text{Al}(\text{OH})_2\text{Cl}^-$ and gel products $\text{Al}(\text{OH})\text{Cl}_2$ and $\text{Al}(\text{OH})_2\text{Cl}$ [12].

1.2.6.3 Mechanical Aspects

The issue of whether the SCC of aluminum alloys occurs via a continuous process, driven by anodic dissolution of aluminum, or a discontinuous process involving mechanical and electrochemical components has been debated in the literature for at least the last three decades [12].

** Crack Initiation:* Some investigators suggested that SCC initiation in aluminum alloys involves a predominantly corrosion-driven process, whereas crack propagation requires a significant mechanical component [12].

Other investigators suggested that the critical process controlling SCC initiation probably involves the establishment of a sufficient stress raiser, located at an appropriate microstructural site within a susceptible microstructure, with the

site of attack being dictated by the size and distribution of suitable anodic and cathodic reaction sites within an alloy's local microstructure [12].

* *Crack Propagation:* It is suggested that SCC propagation in aluminum alloys is a discontinuous process, probably involving a rapid micromechanical crack jump followed by a slower step involving corrosion prior to the next mechanical jump [12].

Recent fractographic studies of 7XXX series alloys stress corrosion cracks revealed that SCC crack growth initiation from the notches was internal and associated with a region ahead of the notch. It was suggested that the only plausible explanation is that during SCC test, hydrogen was generated via a cathodic electrochemical process in aqueous electrolyte, with some diffusing into the alloy via GB diffusion to accumulate at sites of high stress ahead of the notch and lead to internal cracking [12].

1.2.6.4 SCC of Welds

For weldable aluminum alloy systems other than Al-Zn-Mg alloys, service failure resulting from SCC is rare.

SCC of welded Al-Zn-Mg joints has been observed in service and laboratory experiments. Two types of SCC failures were reported in literature for welded Al-Zn-Mg joints these are:

1. Weld-toe cracking, which initiates at weld toes and propagates into the interfacial region between the weld bead and heat-affected zone (HAZ) (figure 1.16), commonly known as the "white zone" because of its etching behavior in nitric acid.

Weld-toe cracking is a complex phenomenon that involves at least two processes; the first being separation of the mechanically weak/chemically reactive weld overlap region from the plate (the weld toe region) and the second crack initiation and propagation in the white zone driven by HE [12].

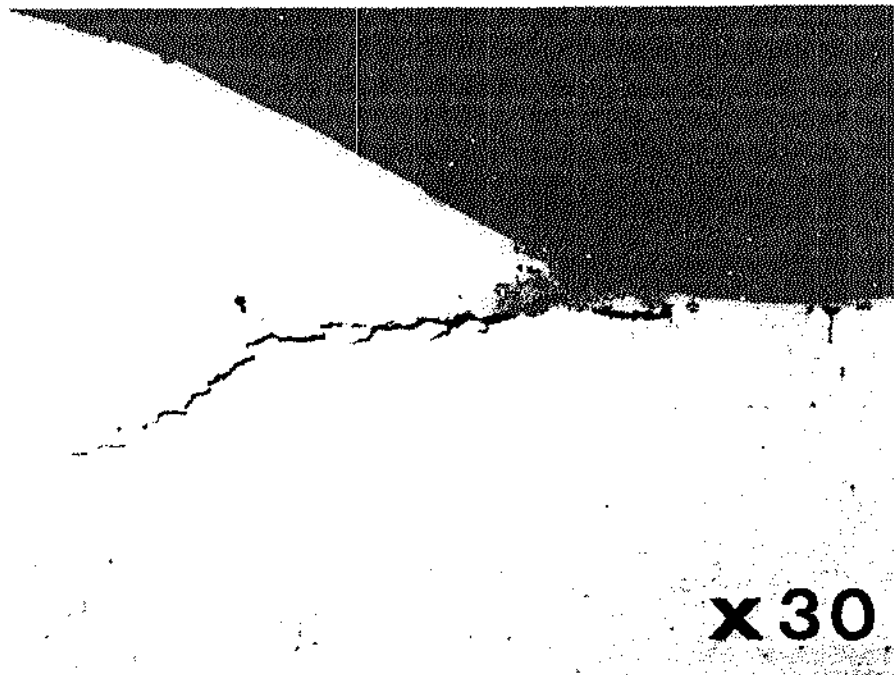


Fig. 1.16: A weld-toe crack propagating in the “white-zone” region [12]

2. Low-temperature HAZ cracking, which occurs as SCC within the HAZ a few millimeters from the weld bead as shown in figure 1.17 [12,16].

This is also a complex phenomenon and is attributed to the difference in potentials between the SCC susceptible region and the other regions [16].

Considerable efforts has been expended to prevent these types of cracking. Some workers suggested protecting the welded Al-Zn-Mg alloys by coatings, others preferred post-weld heat treatments and very recent approaches is using controlled shot peening to reduce stress levels in welded joints [12,15].

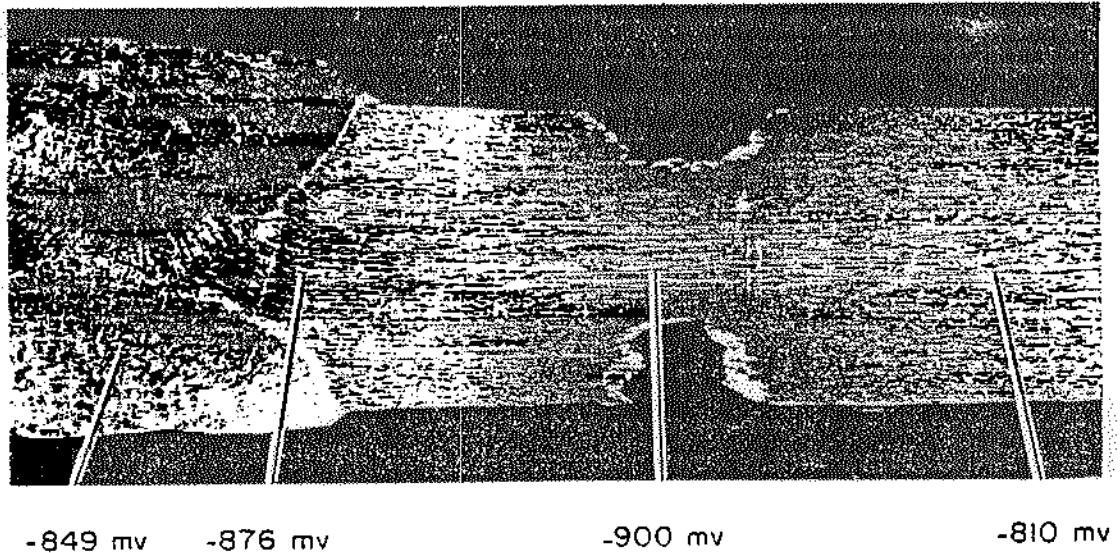


Fig. 1.17: Selective attack and SCC in HAZ in a welded 7075-T6 alloy (Potential measurements are shown in relationship to site of SCC developed by (7075-T6 welded) pannels exposed to 3.5% NaCl solution) [16].

1.3 Shot Peening

Shot peening is a cold working process in which the surface of a part is bombarded with small spherical media called shots. Each shot striking the material acts as a tiny peening hammer, imparting to the surface a small indentation or dimple. In order for the dimple to be created, the surface fibers of the material must stretch beyond its yield point. Below the surface, the fibers try to restore the surface to its original shape, thereby producing below the dimple, a hemisphere of cold-worked material highly stressed in compression.

Overlapping dimples develop an even layer of metal in residual compressive stress. It is well known that cracks will not initiate or propagate in compressively stressed zone. Since nearly all fatigue and stress corrosion failures originate at the surface of a part, compressive stresses induced by shot peening provide considerable increases in part life. The maximum compressive residual stress produced at or under the surface of a part by shot peening is at least as

great as half the yield strength of the material being peened [17]. The depth of compressive stress varies according to the part's material composition and hardness, the shot media used (steel, glass, ceramic), the size and velocity of the shot etc.

These factors affect the size of the dimple which gives an indication of the depth of compression (Fig. 1.18) [18].

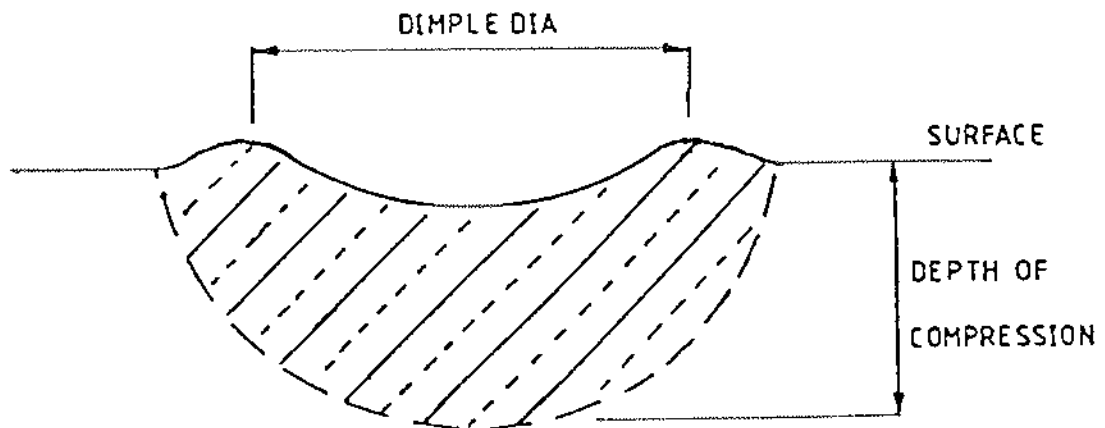


Fig. 1.18: Proportion of dimple diameter and compressive depth [18]

There is some evidence to suggest that, for some materials at least, the depth of compression is approximately equal to the dimple diameter [18], and was found experimentally to vary from 0.1 to 1 mm [19]. Fig. 1.19 shows a typical profile of residual compressive stress in a plate which has been shot peened on the upper surface, it is assumed that there were no residual or applied stresses in the plate prior to shot peening. In this figure there are four important characteristics.

- 1) SS-Surface Stress - The stress measured at the surface.
- 2) CS-max - Maximum Compressive Stress - The maximum value of the compressive stress induced, which normally is highest just below the surface.
- 3) d-Depth - The depth of the compressive stress is the point at which the compressive stress crosses over the neutral axis and becomes tensile.

4) TS-Maximum Tensile Stress - The maximum value of the tensile stress induced. The offsetting tensile stress in the core of the material balances the surface layer of compressive stress, so that the part remains in equilibrium [17].

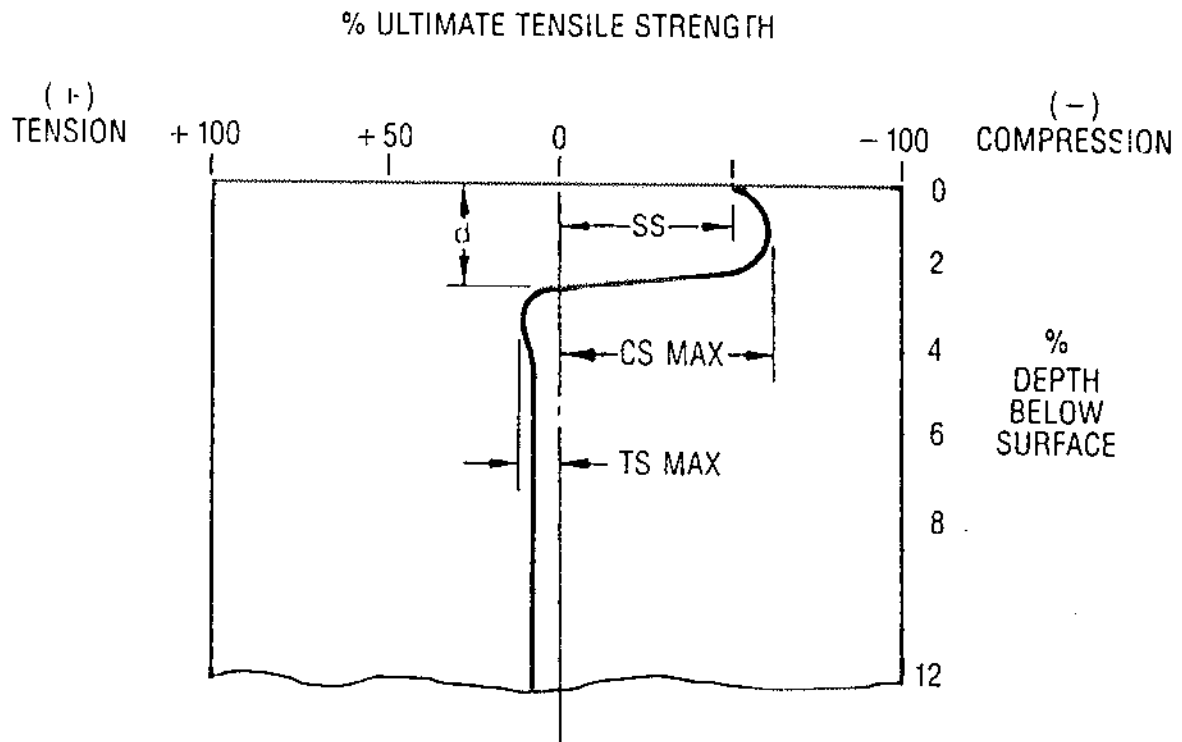


Fig. 119: Example of residual stress profile created by shot peening [17]

1.3.1 Shot Peening Variables

Shot peening is influenced by a complex interaction of a variety of variables summarized in Table 1.2. These are divided into two groups which are characterized by either the shot peening or the workpiece conditions. The two variable groups, however are largely interactive [20].

Table 1.2: Shot Peening Parameters [19]

Shot Peening Parameters	
<u>Shot Peening conditions</u> <ul style="list-style-type: none"> • type and density • shape, diameter and size distribution. • hardness. • velocity or pressure. • exposure time or coverage. • impact angle. • mass flow. • nozzle diameter. • distance from nozzle to workpiece. 	<u>Workpiece Conditions</u> <ul style="list-style-type: none"> • hardness or yield point. • work hardening rate. • residual stresses. • surface topography. • phase composition. • phase stability. • defects in the surface layer.

1.3.2 Methods for the control of Shot Peening Variables

As shown in table 1.2; there is a large number of variables in the peening process. In order to have a consistent peening result, the dominant process variables must be identified and properly controlled, this is done by standard measures and methods related to these variables (table 1.3) [21].

The most prominent measures however are the intensity and coverage which represent the dominant variables as shown in fig. 1.20 [21].

Table 1.3: Methods and properties to characterize the shot peened surface state [19]

Methods and properties to characterize the shot peened surface state
<ul style="list-style-type: none"> • microhardness HV • residual stress σ^r • Almen intensity $i \sim v.d.t$ • surface roughness R_t • coverage

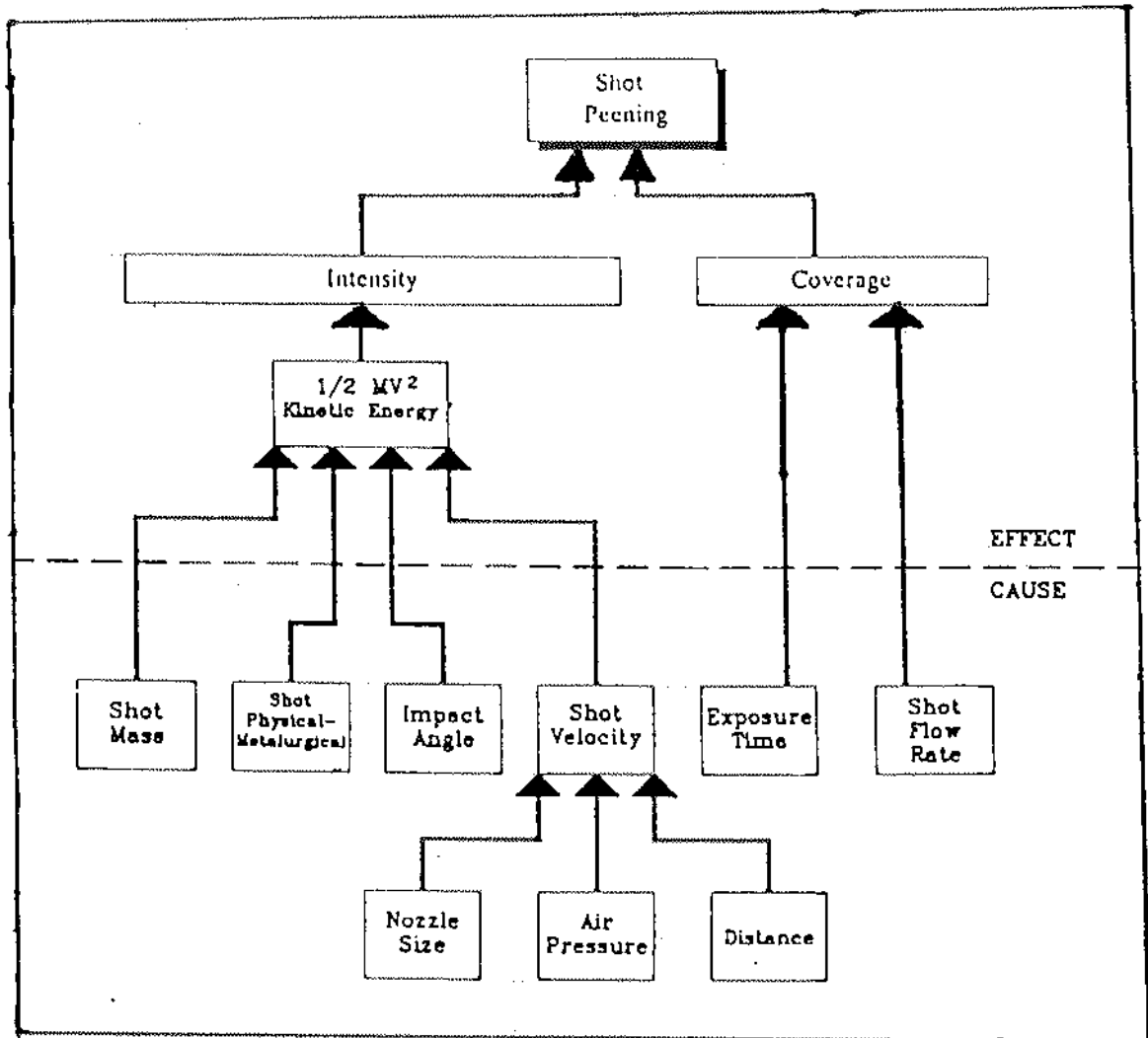


Fig. 1.20: Controlled Shot Peening Process [20]

1.3.2.1 Peening Intensity

Calibration of the impact energy or peening intensity of the shot stream is essential to controlled shot peening. The kinetic energy of the shot stream is a function of the media mass, material, hardness, velocity and impact angle (Fig. 1.20). In order to specify, measure and calibrate peening impact energy, J.O.

Almen of General Motors Research laboratories developed a method utilizing SAE1070 spring steel specimens which he called Almen strips [17].

The Almen strip is the only available non-destructive measurement instrument utilized to represent the amount of aggregate kinetic energy transfer to a workpiece resulting from the shot peening process [22].

In this method, an unpeened Almen strip is fastened to a steel block and exposed to a stream of peening shot for a given period of time. Upon removal from the block, the residual compressive stress and surface plastic deformation produced by the peening impacts will have caused the Almen strip to curve, convex on the peened surface. The height of this curvature when measured in a standard Almen gauge is called arc height. Fig. 1.21 illustrates the concept of the Almen system. An Almen strip must not be reused after peening [17].

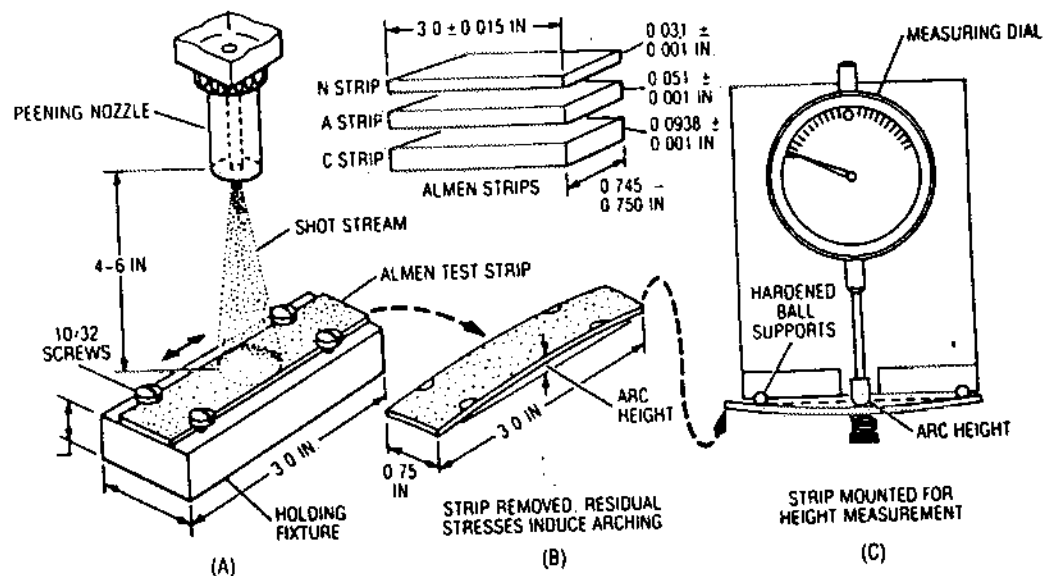


Fig. 1.21: The Almen System concept [17]

The Almen strip test uses three standard size strips to measure ranges of peening intensity. All strips are 3.0 inch long and 0.75 inch wide. The thickness ranges is [23]:

“N” - 0.0310 inch - for low intensity.

“A” - 0.0510 inch - for average intensity.

“C” - 0.0938 inch - for high intensity.

The useable range of curvature on the Almen strips is 0.004 to 0.024 inch. Intensity designations should include both the arc height and the type of Almen strip used:

e.g., 10A intensity 0.01inch (0.25mm) arc height on the A strip.

The depth of the compressive layer is proportional to the Almen intensity (Fig. 1.22). Intensity control must be regarded as one of the essential means of ensuring process repeatability [17].

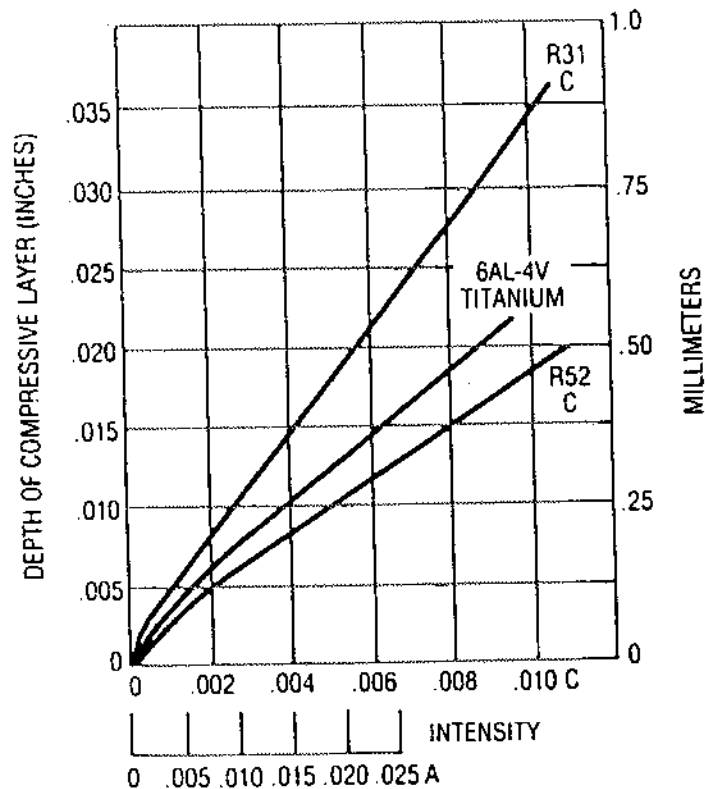


Fig. 1.22: Depth of compression versus Almen arc height [17]

1.3.2.2 Saturation

An Almen arc height is not properly termed intensity unless saturation is achieved. In order to measure Almen strip saturation, an intensity curve must be developed. When a series of Almen strips are peened with a fixed machine setting for different exposure times, a saturation curve similar to the curve shown in Fig. 1.23 can be developed. Saturation is defined as the earliest point on the curve where doubling the exposure time produces no more than a ten percent (10%) increase in arc height [17].

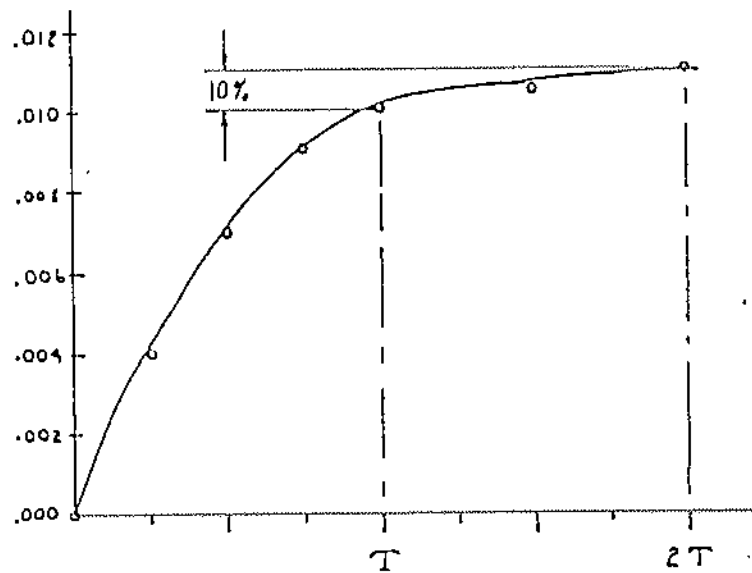


Fig. 1.23: Almen Strip Saturation Curve [17]

1.3.2.3 Coverage

Coverage is defined as the extent (in percent) of uniform and complete dimpling or obliteration of the original surface of the part or workpiece. Inspection of percent coverage can be accomplished using a ten power (10X) magnifying glass [17,21].

100% coverage is reached when the original surface of the material is obliterated entirely by overlapping peening dimples. Coverage above 100% are simply multiples of the exposure time necessary to achieve 100% coverage.

200% coverage will require exposure time to be twice as long as 100% coverage exposure time. This is often requested as a “safety factor” to insure that 100% coverage was actually achieved [17,21].

1.3.2.4 Overpeening Effect

Overpeening is peening for too long time or for too high intensity, which can result in the damage of grain boundaries and the formation of grain boundary microcracks of the peened metal.

It was found that there is no real danger from overpeening (by time) within the range up to 400% coverage [24,25].

1.3.3 Shot Peening Applications

Typical shot peening applications are listed in Fig. 1.24: improvement in fatigue properties in virtually all metal alloys, retardation of certain forms of corrosion. (such as stress corrosion cracking, intergranular corrosion and hydrogen assisted cracking), forming of metal parts such as wingskins, cold work of metal surfaces to improve wear characteristics and miscellaneous applications such as texturing, testing for adherence of coating, elimination of porosity, etc.. [26].

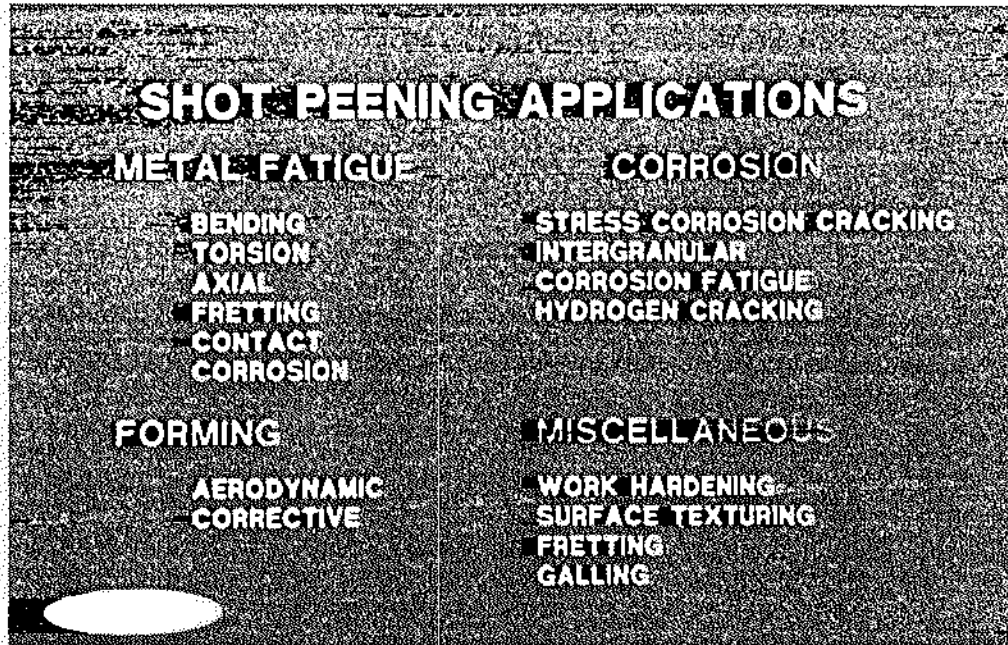


Fig. 1.24: Shot peening applications [26]

1.3.4 Effect of Shot Peening on SCC

Sprowls and Brown [27] studied the effect of shot peening on SCC of aluminum alloy 7075-T6. It was found SCC resistance of 7075-T6 was highly improved by shot peening as shown in Fig. 1.25.

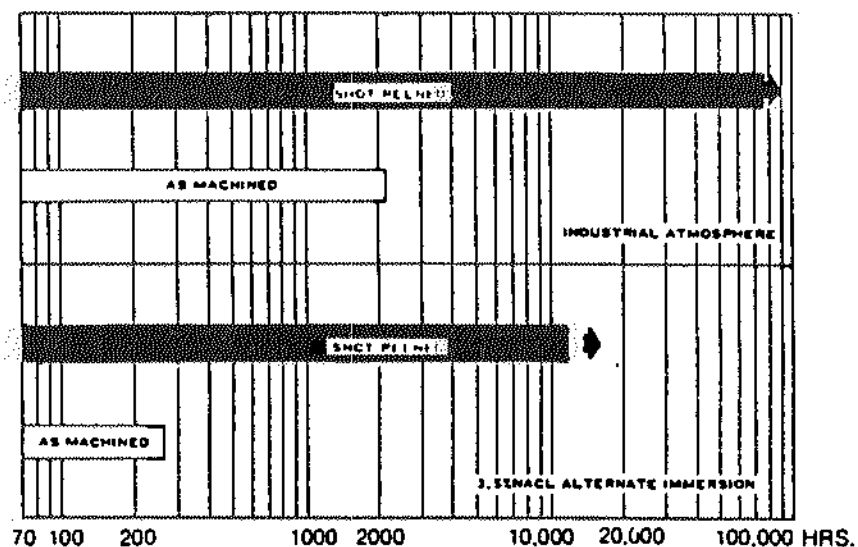


Fig. 1.25: Effect of peening on resistance to SCC of 7075-T6 [27]

Zoeller and Cohen [28] discussed the application of shot peening to rectify SCC of the aircraft components.

Several service failures were mentioned, following are three of them,

1. Landing gear cylinder: An unusual stress corrosion problem occurred on a heavy landing gear, 7079-T6 aluminum forging, where failure occurred in three different areas over a period of eight years. The treatment was to shot peen both interior and exterior surfaces completely, no further failures were reported.
2. Dagger fittings: These fittings were made of 7079-T6 aluminum, and were used to attach the wing to the fuselage. A series of SCC failures occurred soon after fabrication of wing. These fittings were shot peened and no further failures were reported after.
3. Landing gear truck: This truck was constructed of 4340 steel, heat treated to 260-280 ksi ultimate tensile strength, and was shot peened during manufacture it failed due to SCC, but extensive corrosion was evident prior to failure indicating that failure could have occurred much sooner if shot peening had not been employed.

Lifka and Sprowls [2] studied the effectiveness of shot peening as a protection against SCC of high strength aluminum alloys. Tests were performed on 11mm diameter tensile specimens machined from a forged 7075-T6 alloy. The specimens were oriented in the short-transverse direction with relation to the grain structure. Then they were given different protective surface treatments described in table 1.4, stressed to an average cross-section stress of 75% of the yield strength in "constant strain" loading frames and exposed to an industrial atmosphere.

Table 1.4: Description of the Various Protective Treatments Studied [2]

-
- A. Tension Specimens of 7075-T6 Alloy (Surface working and anodic coatings applied to specimens before stressing; paint coatings applied after stressing.)
1. Shot peen, S230 steel shot; two 30-second passes with an air pressure of 80 psi and the nozzle at a distance of 6 in from the specimen.
 2. Grit blast, No. 25 steel grit.
 3. Chromic acid anodic coating plus one coat of zinc chromate primer plus one or two topcoats of oleoresinous aluminum paint.
 4. Grit blast plus paint (System 2 + System 3).
- B. Ring Specimens of 2014-T651 and 7079-T651 (Shot peening applied to rings before stressing; coatings applied after stressing.)
1. Shot peen, S230 steel shot, Abmen intensity 0.011A to 0.012A. Ring placed on a mandrel rotating at 10 rpm and with an air pressure of 70 psi and a nozzle distance of 12 in, the flat exterior surface was given an exposure of 3 min and each edge an exposure of 5 min. Iron contamination was removed from peened surface by immersion in a nitric-sulfuric acid solution.
 2. Alodine 1200 plus one coat of strontium chromate epoxy primer (1 mil) plus one topcoat of epoxy-polyamide paint (2 mils).
 3. Shot peen plus paint (System 1 + System 2).
-

Individual specimen lives for the various test specimens are given in Fig. 1.26 As shown in this figure, shot peened specimens were still intact after a 17-year exposure, whereas as-machined specimens exposed with no protection failed in less than six months.

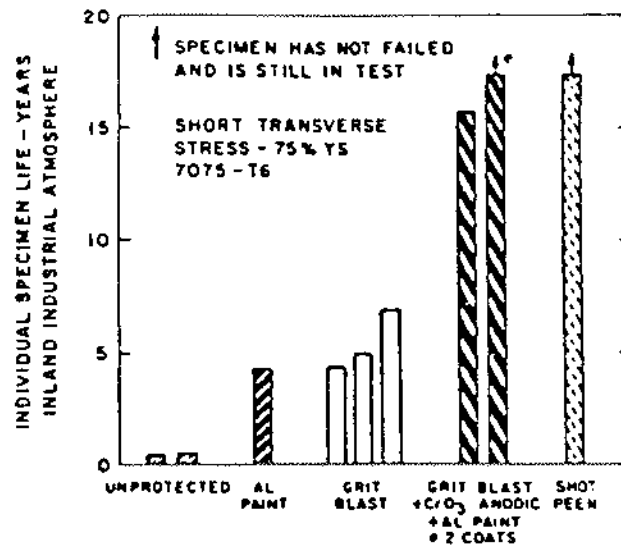


Fig. 1.26: Prevention of SCC of 7075-T6 with surface working treatments and an organic coating [2]

Tests were also conducted on 2014-T6 and 7079-T6 wrought aluminum alloys. The specimens were interference-stressed rings, the rings were given different protective surface treatments described in table 1.4, stressed to develop on the external surface a hoop tension stress equal to 75% of the yield strength and exposed to three types of environments, 3.5% sodium chloride solution, seacoast atmosphere and industrial atmosphere. Test results are shown in figures 1.27 and 1.28.

As shown in Fig. 1.27, shot peening was found of no appreciable benefit to stressed rings of 2014-T6 alloy in the 3.5% NaCl solution where pitting rapidly penetrated the cold worked layer. However, peening considerably extended the time to failure of stressed specimens of 7079-T6 alloys [2].

A high degree of protection of both alloys in all environments was obtained when the cold worked surface layer was protected from pitting corrosion by good paint systems as shown in figures 1.27 and 1.28.

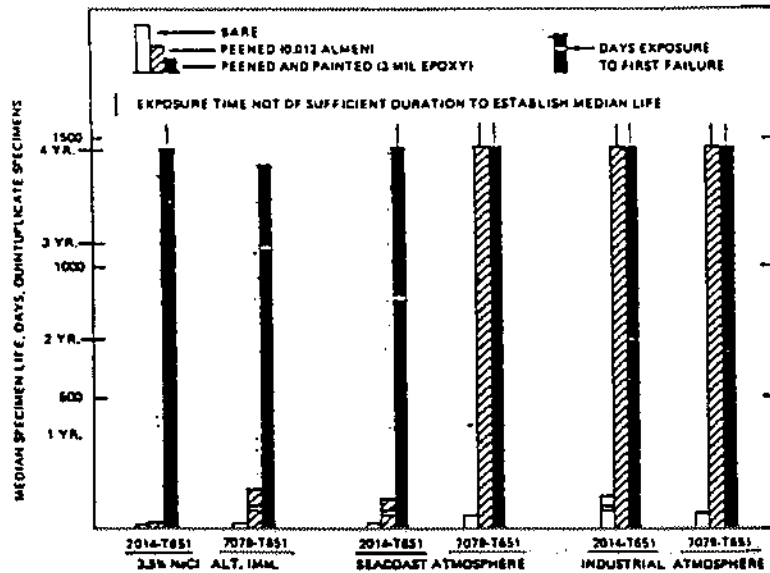


Fig. 1.27 Protection against SCC afforded by peening and peening plus painting on specimens of two high-strength aluminum alloys (2014 and 7079) [2]

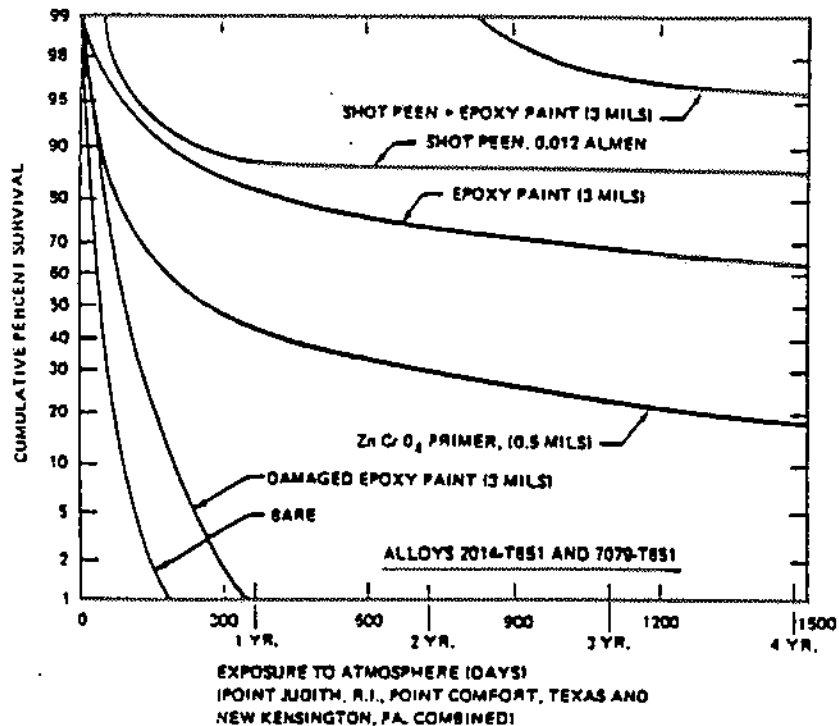


Fig. 1.28: Relative protection against SCC of various protective systems [2]

Microscopic examination of peened specimens that failed revealed that the type of attack in the cold worked surface layer was pitting only and that intergranular attack and SCC did not occur until the compressive layer had been penetrated by pitting corrosion.

It was concluded that the improvement of SCC resistance of aluminum alloys by shot peening resulted from obliteration of grain boundaries in the highly distorted cold worked layer and development of high compressive stresses [2].

Using shot peening to control the corrosion of austenitic stainless steel was the topic of Friske's study [29]. It was suggested that shot peening could influence the SCC of these steels by replacing tensile stresses with compressive stresses.

Tests in boiling 42% $MgCl_2$ showed that "U" bend specimens did not suffer SCC in more than 1000 hour. Unpeened samples on the other hand failed in approximately 1 hour. Friske showed a number of other samples where shot peening prevented the SCC of austenitic stainless steels. Several of them illustrated the effectiveness of the treatment by showing that stress corrosion cracks were present only on the unpeened side of a sample. Thus, full coverage is necessary if the shot peening is to prevent corrosion.

Loescher [29] related several case histories where his company had been able to use shot peening to solve corrosion problems. One of the case studies cited was a carbon steel vessel in service that could lead to caustic cracking. Even though stress relief had been specified after fabrication, it was discovered that the fabricator had rewelded some baffles after heat treatment. Rather than dismantle the equipment, it was chosen to shot peen the reworked areas. They were successful in that the equipment had operated without problems for better than 9 years.

In another case, stress relief was required to avoid SCC of a 316L vessel, but thermal treatments caused warping that had to be straightened. The interior of the vessel was shot peened and has been in service since 1968 without cracking.

Wigmore and Miles [30] examined the effect of shot peening in austenitic 8Mn8Ni4Cr generator end ring steel in chloride environment and the control of the process which was required to give optimum resistance to SCC initiation.

Three point bend specimens which were shot peened at different intensities were tested in boiling 1% NaCl solution.

It was found as intensity was increased corrosion cracking went through a maximum, peaking sharply at a value around 0.012 A Almen peening intensity then decreasing sharply.

It was suggested that this sharp decrease was due to phase transformation to martensite which was found highly susceptible to SCC due to local stress as a result of volume change associated with the transformation [42].

It was concluded shot peening of austenitic stainless steels was very beneficial in delaying the initiation of SCC in such materials provided that the process control was such that martensite formation did not occur during the peening.

Friske and Page [31] studied the feasibility of preventing SCC of austenitic stainless steel in boiling 42% magnesium chloride solution by shot peening. Their study was conducted on austenitic stainless steel types 304, 316, 321 and 347.

It was found that their resistance to SCC was improved over a wide range of shot peening conditions, shot sizes and materials and was effective on sensitized as well as unsensitized material.

It was suggested that effectiveness of shot peening for preventing this type of corrosion was dependent on achieving complete surface coverage rather than on any particular processing parameter.

It was concluded that shot peening prevented SCC by imposing compressive stresses on the workpiece surface and recommended 200% surface coverage to achieve adequate compressive stress levels.

Takemoto et al. [32] studied mainly the protective effects of shot peening against chloride-SCC of welded austenitic stainless steel type AISI304. For this purpose the SCC susceptibility of butt-welded specimens shot peened under various peening conditions in boiling 42% MgCl₂ solution was investigated.

The threshold stresses for SCC and the residual compressive stresses were measured for shot peened specimens. Then the threshold stresses were drawn as a function of the compressive stresses as shown in Fig. 1.29, but there was no distinct correlation between these two factors and when these specimens were exposed to the solution, the specimen with lower SCC threshold and higher residual compressive stress (denoted by B specimen) failed before those with higher SCC threshold and lower residual compressive stresses (e.g. specimens A and D) (Fig. 1.30).

This result was not discussed extensively and there was no clear explanation for such behavior.

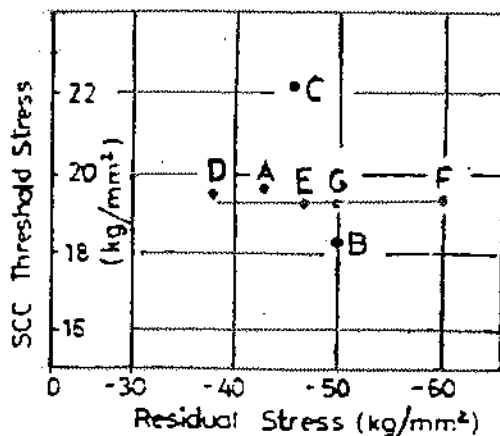


Fig. 1.29: Relationship between SCC threshold stresses and residual compressive stresses [32]

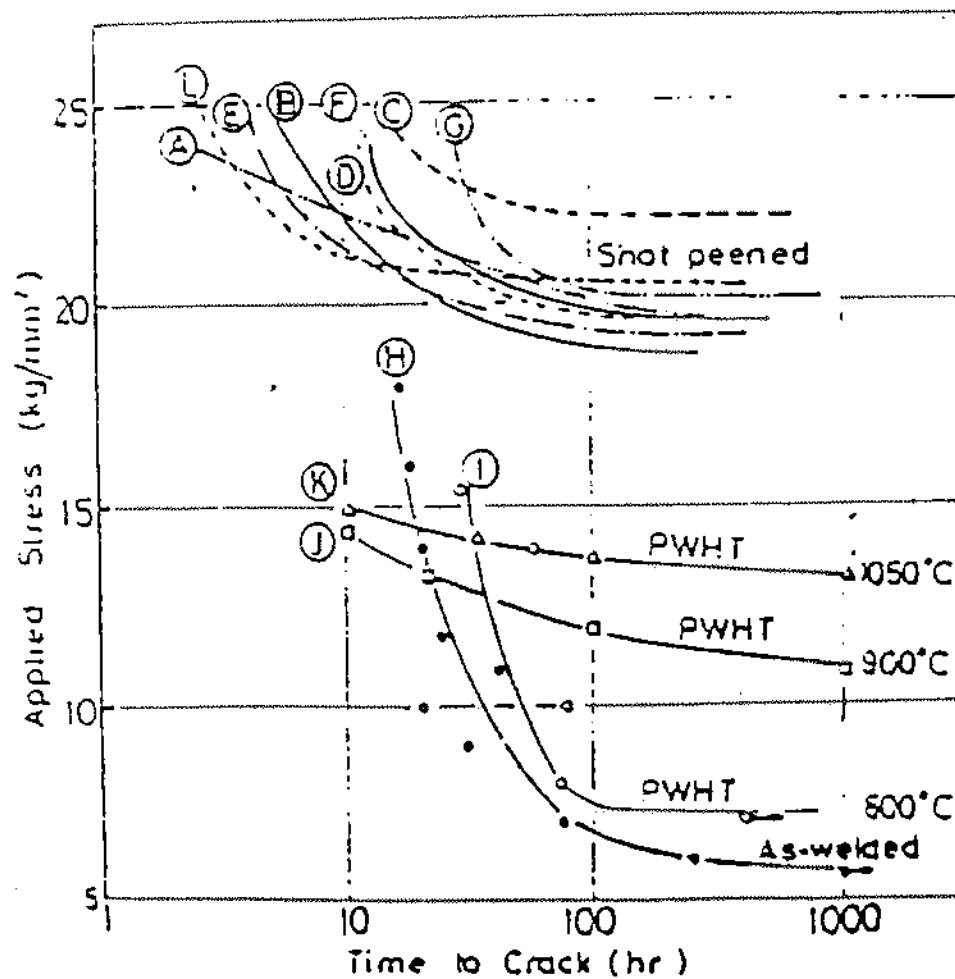


Fig. 1.30: Time to crack curves [32]

It was found that shot peened specimens had higher threshold stresses than as welded specimens and superior resistance to SCC at all the peening intensities.

It was noticed that shot peening was an effective measure to protect the weldment against SCC in spite of shot peening and specimen's conditions provided that all the specimen surface was shot peened so that coverage is more than 100%.

It was suggested that residual compressive stresses imposed by shot peening was the main factor of protecting AISI304 against SCC in $MgCl_2$.

The environmental effect on the SCC susceptibility of shot peened specimens was elucidated using 2 kinds of corrodenet which caused different types of SCC; i.e., boiling 20% sodium chloride ($NaCl$) plus 1% sodium dichromate ($Na_2Cr_2O_7$) solution which usually causes pitting and then SCC from the pits, and 4N sulfuric acid (H_2SO_4) plus 0.4N. $NaCl$ solution which usually causes general corrosion then SCC [32].

It was found that there was a minor improvement of SCC resistance of the specimens tested in 20% boiling NaCl with an increase in the pitting corrosion.

It was suggested that pitting corrosion increased due to the imbedded pieces of broken cast iron shots used in shot peening in the base metal.

It was found that shot peening accelerated the SCC of specimens tested in 4N-H₂SO₄ and 0.4N-NaCl solution and this acceleration was related to the formation of deformation induced martensite which was preferentially dissolved in this acidified solution.

Birely [33] studied the depths of compressive layers produced by mechanical surface treatments such as shot peening, needle peening, hammer peening, roller burnishing, grit blasting and roto peening. The efficiency of these treatments were compared to thermal relieving and vibratory stress relieving (VSR) as stress corrosion preventives in Al-Zn-Mg plate edges, i.e. short transverse directions, which is highly susceptible to SCC.

For the mechanical surface treatments the following remarks were drawn:

1. For the majority of treatments the maximum compressive stress appeared at a short distance below, rather than immediately at the deformed surface which is a common feature of stress distribution induced by peening.
2. Each mechanical surface treatment introduced a considerable magnitude of compressive stress.
3. The depth effect increased with the diameter of the indent, and decreased with increasing deviation from the normal of the angle of application.

It was anticipated that shot, needle, roto and hammer peening, roller burnishing and grit blasting could be employed as preventives for SCC in edges of Al-Zn-Mg alloys in appropriate circumstances if applied in a controlled manner.

The effect of VSR was one of stress re-distribution and was unlikely that this could be employed as a stress corrosion preventive in Al-Zn-Mg structures.

Little or no benefit was derived from the thermal stress relieving treatments applied.

Speidel [34] discussed the shot peening benefits and limitations as a SCC preventive measure. He reviewed some of previous test results of shot peening beneficial effects on SCC resistance for stainless steels, a low alloy high strength steel (Fe-0.37C, 4.88Cr, 1.34 Mo) and a high strength aluminum alloy (Al-6.0Zn-2.4Mg-0.74Cu) in chloride solution.

It was demonstrated that shot peening improved both, the time to failure and the threshold of the applied stress below which no failures were observed in the indicated testing times, these beneficial effects were due to the build-up of residual compressive stresses.

On the other hand it was advised not to draw overoptimistic interpretations of the beneficial effects shot peening had on SCC, since the laboratory testing times were always only several hundred or thousand hours, while components of machines and structures in actual service are most often required to last many times longer. If during that time corrosion penetrates the residual compressive stress layer on the surface of the alloy, then SCC may be observed again.

Further it was suggested where localized corrosion might occur, a combination of shot peening plus a surface protection system against corrosion would yield far better results than shot peening alone.

For maximum protection however, an inherently SCC-resistant alloy was preferred over a shot peened SCC-susceptible and protected alloy, since SCC might result in the susceptible alloy where the surface protection might break down locally, subsequently the surface layer which contained compressive stresses from shot peening would be penetrated by pitting corrosion, as shown in Fig. 1.31.

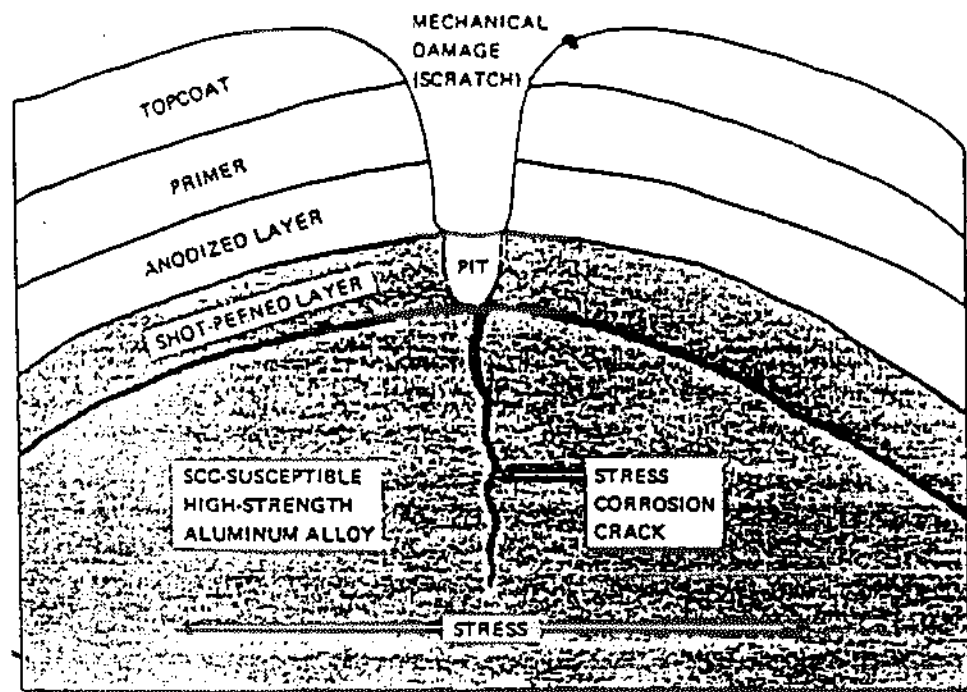


Fig. 1.31: Schematic representation of a stress corrosion failure resulting from the local breakdown of several protective systems. Obviously, the best defense against SCC is an entirely SCC-resistant alloy, if available [34].

Takemoto [35] developed a Wet Shot Peening (WSP) method in order to prevent SCC of austenitic stainless steel butt-welded pipes.

In the WSP, glass beads were injected to member surface with water jet pressurized by high speed centrifugal slurry pump.

In this work, the effect of WSP on residual stresses, surface roughness, metallurgical changes and removability of scale and deposits was investigated. Upon this investigation it was concluded that WSP prevented SCC due to the followings:

1. It introduced compressive stresses at the most critical weld's areas, i.e. under cut; overlap and poor penetration areas.
2. It removed weld slag and scale.
3. WSP reduced significantly both sensitization and martensitic transformation by controlling the water temperature.

The usefulness of WSP method was compared with that of air-blast shot peening and it was found that WSP was superior in controlling the residual stresses and metallurgical changes as well as removing the weld slag and scale.

Two examples of application of this method to chemical process equipment were discussed. It was concluded that WSP was found to be a very flexible surface treatment of welded and/or cold worked members, for large towers and vessels in site.

Feld and Gillespie [36] related several case histories in industry where Controlled Shot Peening (CSP) was a cost effective technique to reduce SCC failures which costed industry millions of dollars following are two of them:

One of the cases sited was the failure of a Hastelloy blower wheel used in hot nitrogen (300°F) in less than six months of operation. The treatment was to shot peen the component with 170 shot to an intensity of 10-14A followed by glass bead peening for decontamination with minimum coverage of 200%, no failure was reported after three years of operation.

Another case was the failure of Hastelloy B-2 welded reboiler heads used in water containing chlorides after eight months of service due to SCC in the formed dome and heat affected zones. The solution was to peen the part with 230 shot to an intensity of 12-14A followed by glass peening for decontamination with minimum coverage of 200%. No failure was reported after two years of service.

Koehler [37] studied the influence of shot peening on the stress corrosion resistance of the welded high strength aluminum alloy AlZn4.5Mg2 state T6.

It was reported that the welded tensile samples of the alloy were shot peened with glass, steel and ceramic beads as well as granulated aluminum material; then the samples were tested in 2% and 3.5% NaCl-solution for stress corrosion resistance.

It was found that shot peening significantly improved the stress corrosion behavior.

The beneficial effects of shot peening on corrosion behavior was related to the formation of strong cold-worked surface zones as well as to compressive residual stresses induced by shot peening.

Parkins [38] stated that shot peening is very effective in preventing or reducing the incidence of SCC in various systems due to the compressive layer introduced by this process. If this layer is removed, even locally, by slow general or pitting corrosion, the susceptibility to SCC will return. To avoid such situation a subsequent coating application is very beneficial.

O'Hara [39,40] discussed the beneficial effect of controlled shot peening on SCC resistance of aircraft aluminum structural components and those manufactured from titanium and high strength steels.

It was stated that shot peening retards or eliminates the SCC by eliminating one of the essential SCC factors; the tensile stress, and placing the surface in compression.

Shot peening was preferred over thermal stress relief since it does more than just eliminate the tensile stress, it leaves the surface with a very high compressive stress.

Yoshie [41] described the application of shot peening for preventing SCC of chemical processing equipment at his company.

It was stated that shot peening was applied to more than 20 cases in the past eight years and three cases of them were presented.

An absorber column and reactor experienced intergranular SCC due to sensitization which had been caused by heat effect of welding without pitting or crevice corrosion. These cracks were repaired by welding and then shot peened, no SCC failure reported ever since 8 years of service.

Further a gas separator experienced transgranular SCC which crossed the weld line due to chloride environment after six months of service. Cracks were repaired by welding and then shot peened, there was no reported SCC failure since six years of operation.

In all of the three cases a minimum coverage of 100% was required and media of shot peening was austenitic stainless steel to avoid contamination of the

surface by embedded fragments of the common used carbon steel shot which would cause galvanic and pitting corrosion.

Before performing shot peening as a preventive measure against SCC for all the cases, the effectiveness of shot peening in preventing SCC was examined performing laboratory testing of austenitic stainless steel (SUS 304) welded plates in boiling 42% magnesium chloride solution, it was found that while unpeened specimens cracked within few hours, the peened ones didn't crack in one or two weeks time [41].

It was concluded that although shot peening is a very economic preventive measure against SCC it is not yet the prevailing way for the prevention of SCC due to the following reasons:

1. Its effectiveness is not properly recognized.
2. Concrete ways of application is not widely known.

Mukhopadhyay et al. [42] studied the effect of shot peening on the SCC of 18Mn4Cr austenitic steel in oxygenated aqueous chloride medium at 70°C.

The specimens were shot peened at different shot sizes, pressures and peening times, then the peening effects on the surface presented by the compressive stress layer, martensite transformation and reduced surface roughness were studied.

Based on these results a set of optimum peening parameters which gave the maximum compressive stresses, the minimum amount of martensite and the best surface finish was found. This set of parameters was used to peen stress corrosion test specimens for further investigation.

SCC test revealed that while unpeened specimens failed within 200 hours of exposure, peened specimens withstood stress corrosion for as long as 1040 hours. Therefore using optimum parameters at least a five-fold increase in stress corrosion life had been achieved.

Verpoort and Gerdes [24] reported that the SCC resistance of a component is improved due to the homogeneous deformed surface zone produced during shot peening which will improve the resistance of passive films to breakdown.

Tsai, et al., [43] studied the effect of shot peening on SCC behaviors of sensitized alloy 600 (UNS N06600) in thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) solution at 95°C.

It was found that shot peening improved SCC resistance of this alloy.

It was concluded that the existence of compressive residual stress and the change in microstructure of surfaces; i.e. the homogeneously deformed surface layer with no sharp slip; induced by shot peening delayed crack initiation.

1.4 Objective of the Work

It appears from the literature that there is a great shortage in the investigations being carried out to study the effect of shot peening on the SCC resistance of high strength aluminum alloys. However, the effect of such economic process has been studied extensively to evaluate the resistance to several types of failure, especially fatigue failure.

In this study it is intended to evaluate the effect of shot peening using different intensities, on SCC resistance of 7075-T6 aluminum alloy. This alloy is not recommended to be used in as-welded condition unless a post-weld heat or mechanical treatment is carried out due to the metallurgical change prevailing after welding. Thus this investigation intends first to study these metallurgical structures which reduce the SCC resistance and then to examine the effectiveness of shot peening in reducing their effects and improve SCC resistance.

CHAPTER TWO
EXPERIMENTATIONS

CHAPTER TWO

EXPERIMENTATIONS

2.1 Materials

Aluminum Association (AA) 7075-T6 age-hardened Al alloy was selected for this study.

The AA7075 aluminum was obtained as commercial heat treated Alclad sheet of 2.54mm thickness and plate of 25.4 mm thickness. It was in the T6 temper condition. T6 is a solution heat treated and artificially aged condition designed to produce maximum tensile strength [3].

The chemical composition and mechanical properties are given in tables 2.1 and 2.2 respectively.

Table 2.1: Chemical Analysis of 7075-T6 Aluminum Alloy

Element	Al	Zn	Mg	Cu	Fe	Cr	Si	Mn	Ni	Ti	Pb	Sn
Weight (%)	89.744	5.553	2.380	1.578	0.326	0.184	0.123	0.069	0.013	0.013	0.009	0.008

Table 2.2: Mechanical Properties of 7075-T6 Aluminum Alloy of Thickness up to 6.3mm [3,13,44]

Grain Orientation w.r.t rolling direction	Yield strength (MPa)	Tensile strength (MPa)	Maximum Elongation (%)	Modulus of Elasticity (MPa)
Longitudinal (L)	503.3	572.3	7	$71.0 \cdot 10^3$
Longitudinal transverse (LT)	468.9	558.5	5	$71.0 \cdot 10^3$
Short Transverse (ST)	410.0	-	-	$71.0 \cdot 10^3$

2.2 Specimens Configurations

Three sets of test specimens were used. Each set had different orientation with respect to the original rolling plane and rolling direction.

The first and second sets were prepared such that the greatest dimension, i.e. the length, of the specimens was in the longitudinal (L) and the longitudinal transverse (LT) directions of rolling respectively. (Fig. 2.1).

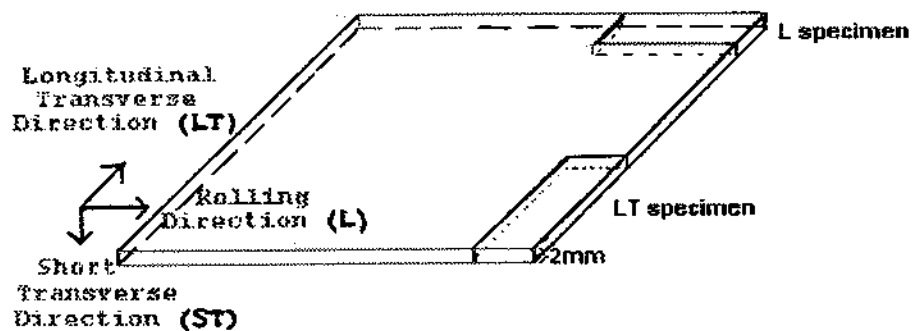


Fig. 2.1: First and Second Sets Orientations (L and LT specimens)

The specimens were flat strips cut from an Alclad sheet of 2.54mm thickness. The Alclad layer was removed by grinding to obtain specimens of 140*26*2 mm.

The third set were prepared such that the specimens length was in the short transverse direction (ST) (Fig. 2.2).

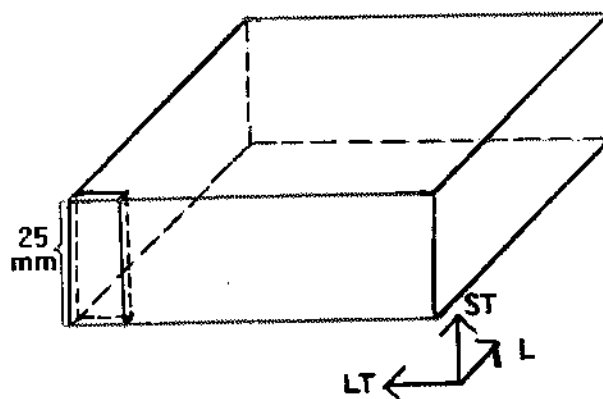


Fig. 2.2: Third Set Specimens Orientation (ST specimens)

The specimens were cut from a plate of 25.4 mm thickness. The specimens dimensions were 140*25.4*2.3 mm.

The above three orientations were used to prepare specimens in welded and unwelded condition.

2.3 Welding Conditions

Bead-on-plate weldment were fabricated by depositing a weld bead on one side of the specimen using AC gas tungsten arc (TIG) welding (Fig. 2.3). Welding parameters are shown in table 2.3.

Table 2.3: Welding Parameters

Parameters	Welding
Power	AC
Voltage (V)	68
Current (A)	75
Filler Wire	AMS 4190, size 0.062 inch
Number of passes	1
Shielding Gas	Argon

2.4 Equipments

2.4.1 Shot Peening Machine

A Pangborn ES-1580 shot peening machine was used (Fig.2.4). This is an air blast machine in which the compressed air is introduced at the rear of the nozzle (also called a gun) producing a low pressure, high velocity air flow in the gun body, the peening shots are stored in an overhead hopper and are directed to the

nozzle by means of gravity assist, where they are accelerated by the air which is traveling at a high velocity.

The shot velocity is varied by varying the air pressure fed to the nozzle.

The used shots drop into a collecting hopper, where they are picked up by the abrasive elevating system to the gun feed hopper for recirculation.

The workpiece is usually fixed by a special fixture on a rotating table which speed is controlled by a gear and a motor system.

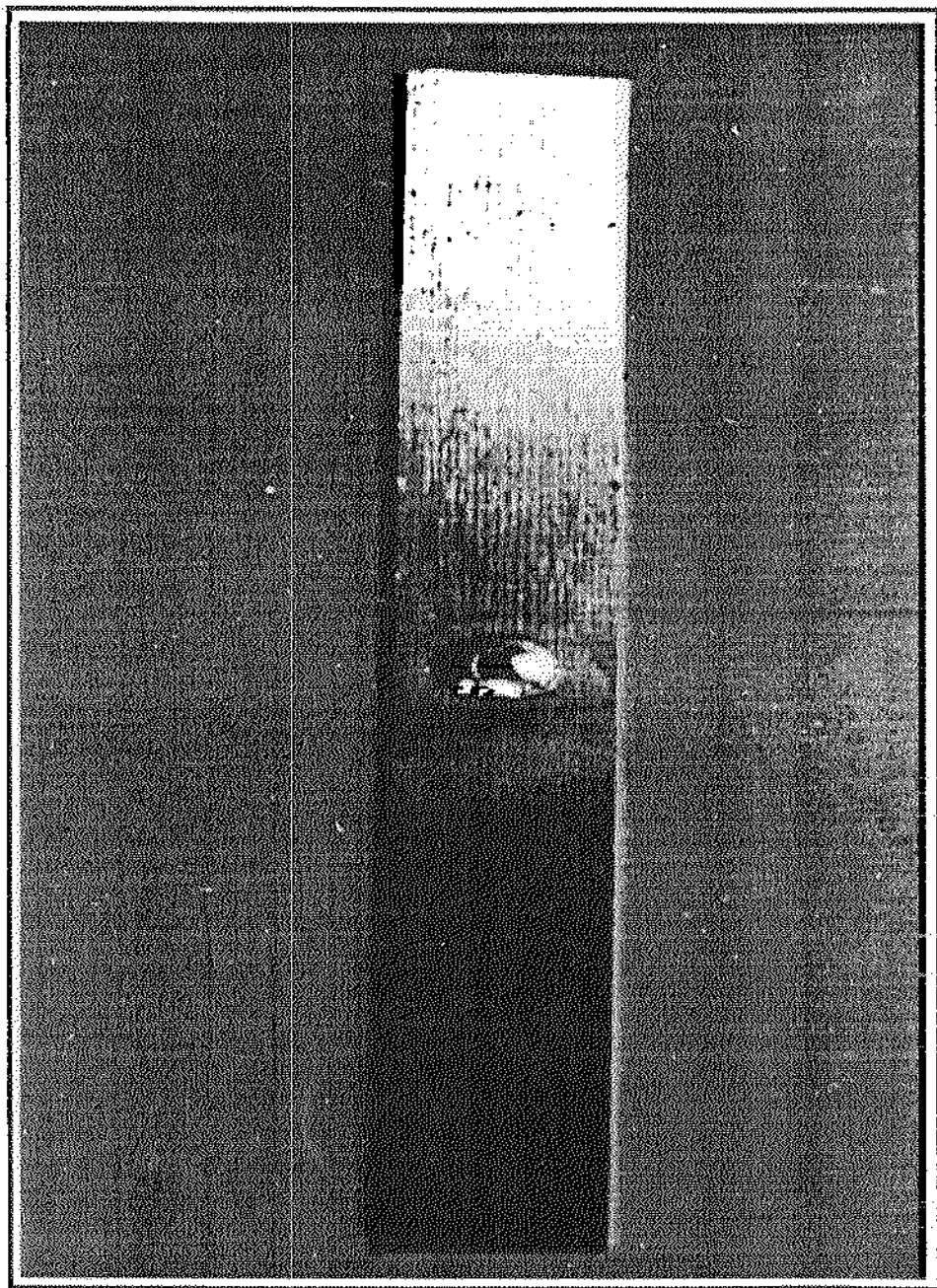


Figure 2.3: Welded specimen

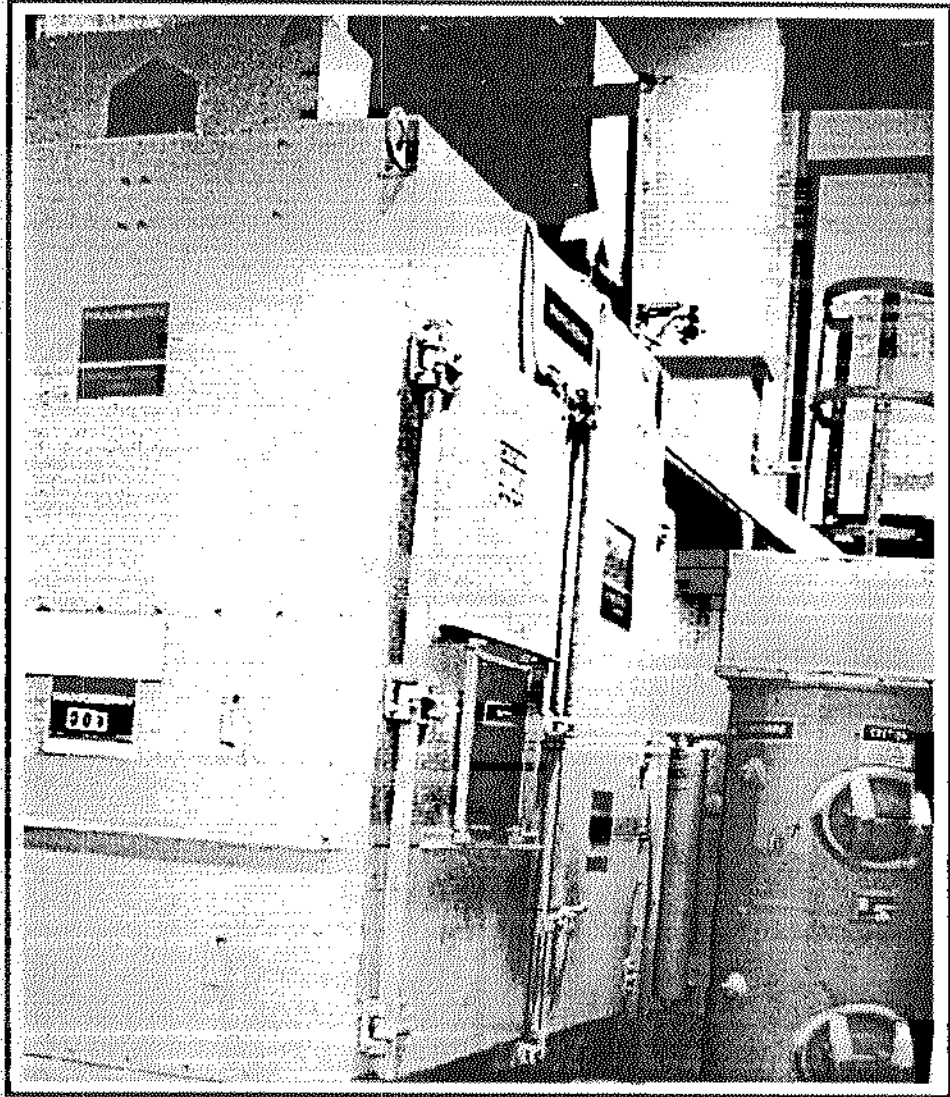


Figure 2.4: The shot peening machine

2.5 Shot Peening Procedure

Welded LT and ST specimens were shot peened on one surface (i.e. the welded side) as follows:

2.5.1. Media Specification

Shot peening was performed with the standard cast steel shot grade S230 having a mean diameter of 0.6 mm conforming to MIL-S-851 [45].

The shot size was specified per SAE-AMS 2432A [46] (table 2.4).

2.5.2. Determination of Shot Peening Intensity

Shot peening intensities were determined conforming SAE-AMS 2432A [46] (table 2.4). Thus shot peening was at 3 intensities, these were 6,8, and 10A.

The peening conditions used to obtain these intensities were:

1. Peening angle was chosen to be 90°. This value was found to be the most suitable one to produce the shot peening effects in terms of maximum compressive stresses and best surface finish [24,47].
2. Standoff distance (distance between the nozzle and target) was selected to be 150 mm because of its practical convenience [2, 28,48,49].
3. Turntable speed was 3 rpm.
4. Shot peening pressures were varied from 40 to 80 psi to obtain the required intensities.

TABLE 2.4—Shot Sizes and Intensities for Aluminum Parts,
Except as Specified in 3.4 [46]

Areas to be Peened	Intensity (1)	Shot Size (1)
Sections over 0.375 inch (9.52 mm)	10 to 14A	230, 330
Sections 0.090 to 0.375 inch (2.29 to 9.52 mm), incl, in thickness	6 to 10A	170, 230
Holes under 0.735 inch (18.67 mm) in diameter	10 to 15N	70, 130

- NOTES:
1. Any shot size and intensity specified on the engineering drawing shall take precedence.
 2. Sizes in Table 4 are for ASR (cast steel shot, regular) and ASH (cast steel shot, hard). Equivalent sizes of other media, in accordance with AMS 2431, shall be used when shown on part engineering drawing.
 3. For material thickness under 0.090 inch (2.29 mm), See 3.1.1.2.

In accordance with MIL-S-13165C [50], for each intensity a series of test strips were peened at progressive exposure times to allow the strips to be run for the saturation time established by the saturation curve, the required intensity was obtained when saturation was reached (Fig. 2.5). The test strip, holder and micrometer gage are shown in Fig. 2.3.

During shot peening process an Almen test strip was attached to each specimen to insure the intended intensity was obtained since the test strip was achieving the same intensity as the specimen (Fig. 2.7).

2.5.3 Surface Coverage

Specimens were shot peened for 200% coverage (Fig. 2.5) per other researchers who recommended 200% coverage as a "safety factor" to insure that 100% coverage was actually achieved. Thus the risk of islands with tensile residual stresses at the surface would be avoided [20,31,32,36].

Peened surface was visually inspected for complete coverage with the aid of 30X magnifier conforming MIL-S-13165C [50] (Fig. 2.8).

2.5.4. Clean after Shot Peening

The specimens peened with steel shots were subsequently peened with glass beads at 15 psi in accordance with some researchers advice, to remove the impurities left by cast steel shots from the aluminum surface. Thus preventing galvanic and pitting corrosion [34,35].

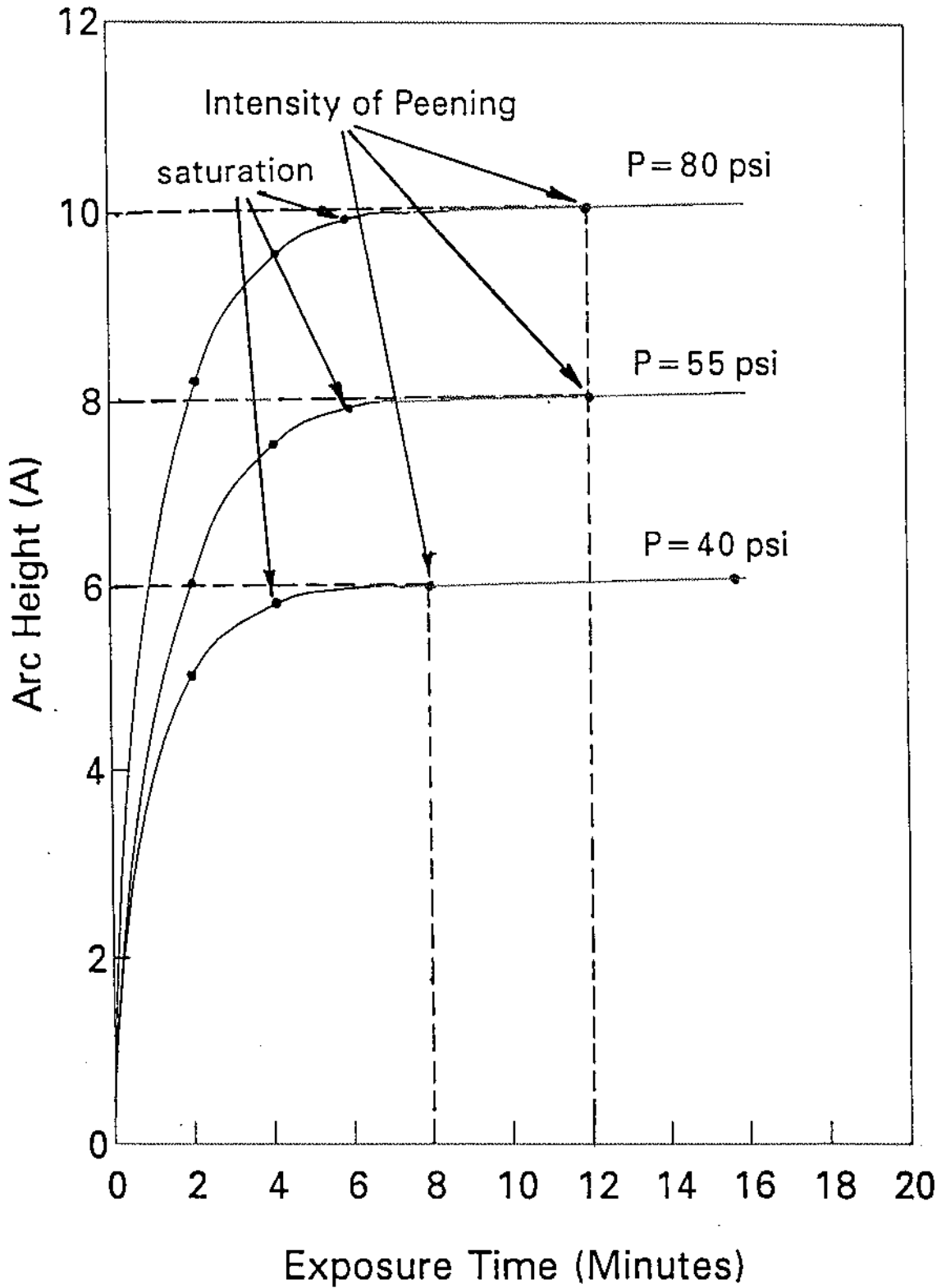


Figure 2.5 Almen strip saturation curves used to specify intensities of 6,8 and 10A.

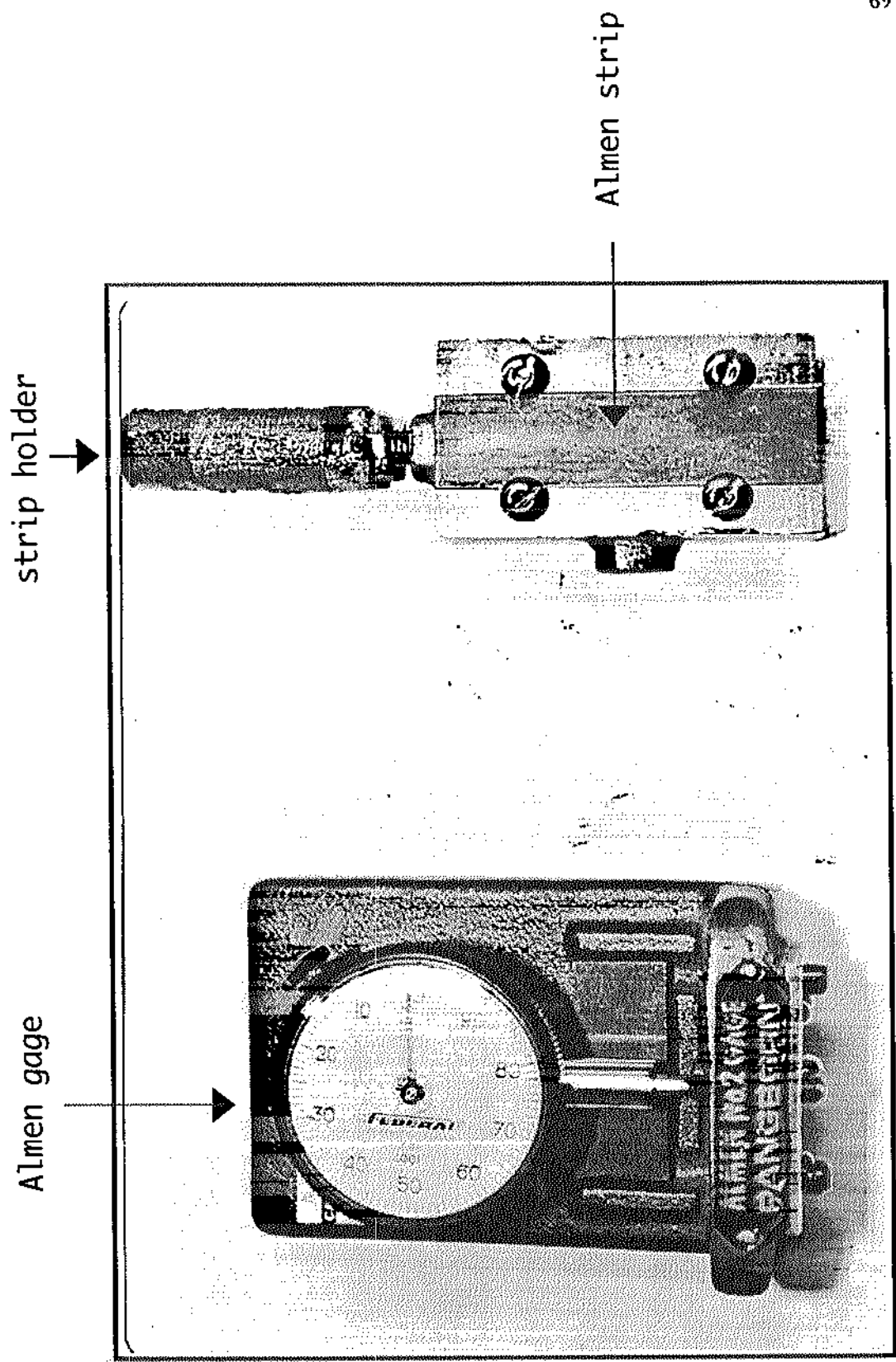


Figure 2.6: Almen gage system

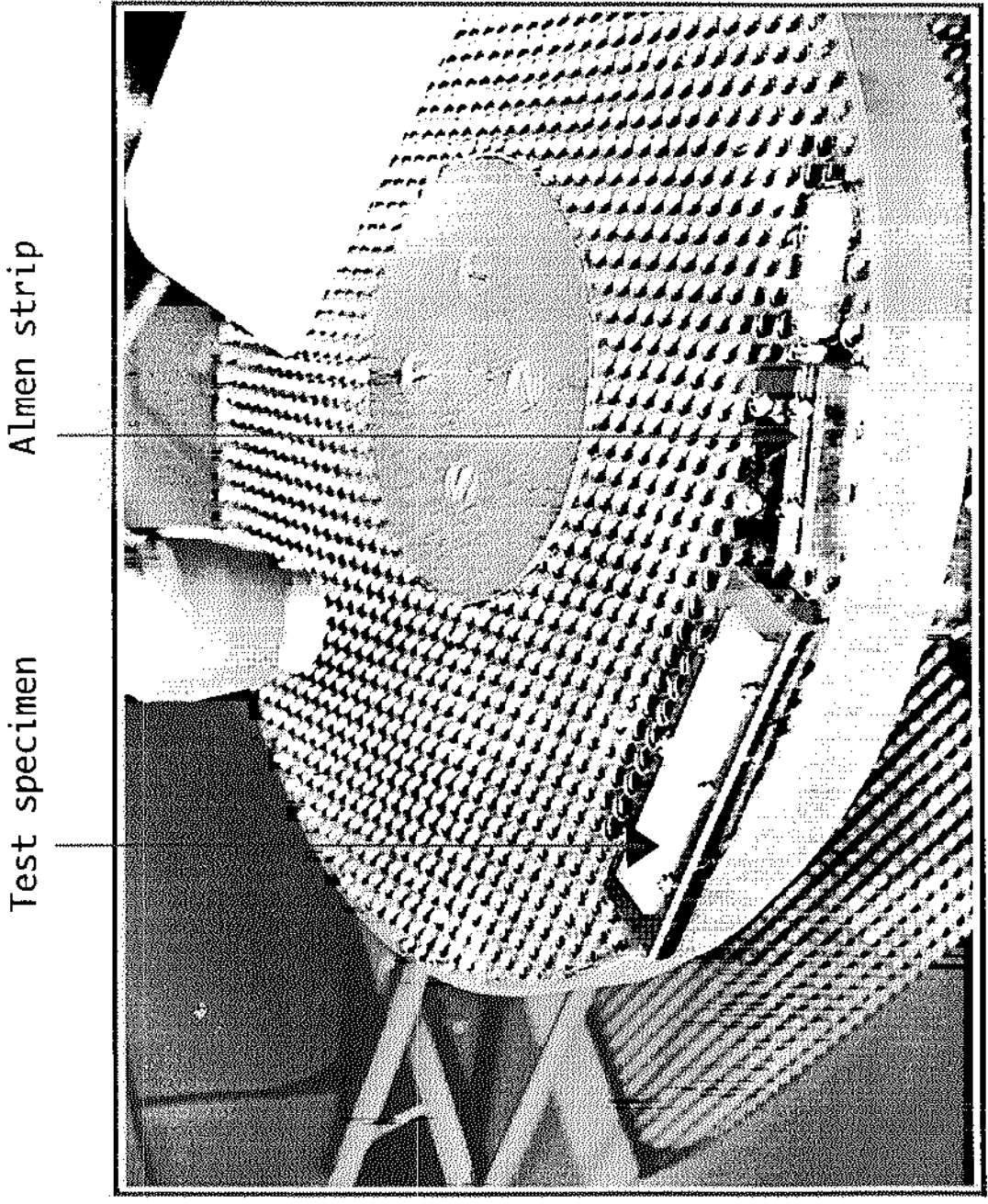


Figure 2.7: An Almen strip is attached to the specimen during shot peening



Figure 2.8: Surface appearance after shot peening, Coverage :200%, Intensity : 6A (30X)

2.6. SCC Tests

The SCC tests were carried out per the followings:

2.6.1 SCC Test Specimens

SCC test specimens were designed conforming ASTM G39-90[51] which is a standard test covers procedures for designing, preparing and using bent-beam stress-corrosion specimens. This constant strain test is applicable to specimens of any metal that are stressed to levels less than the yield strength of the material and therefore the applied stress can be accurately calculated or measured.

In this work peened and unpeened specimens were tested in three point bending system (Fig. 2.9). The specimen holder as well as the stressing jig (loading screw) were fabricated fully from 7075-T6 to avoid galvanic corrosion between the specimen and the holder.

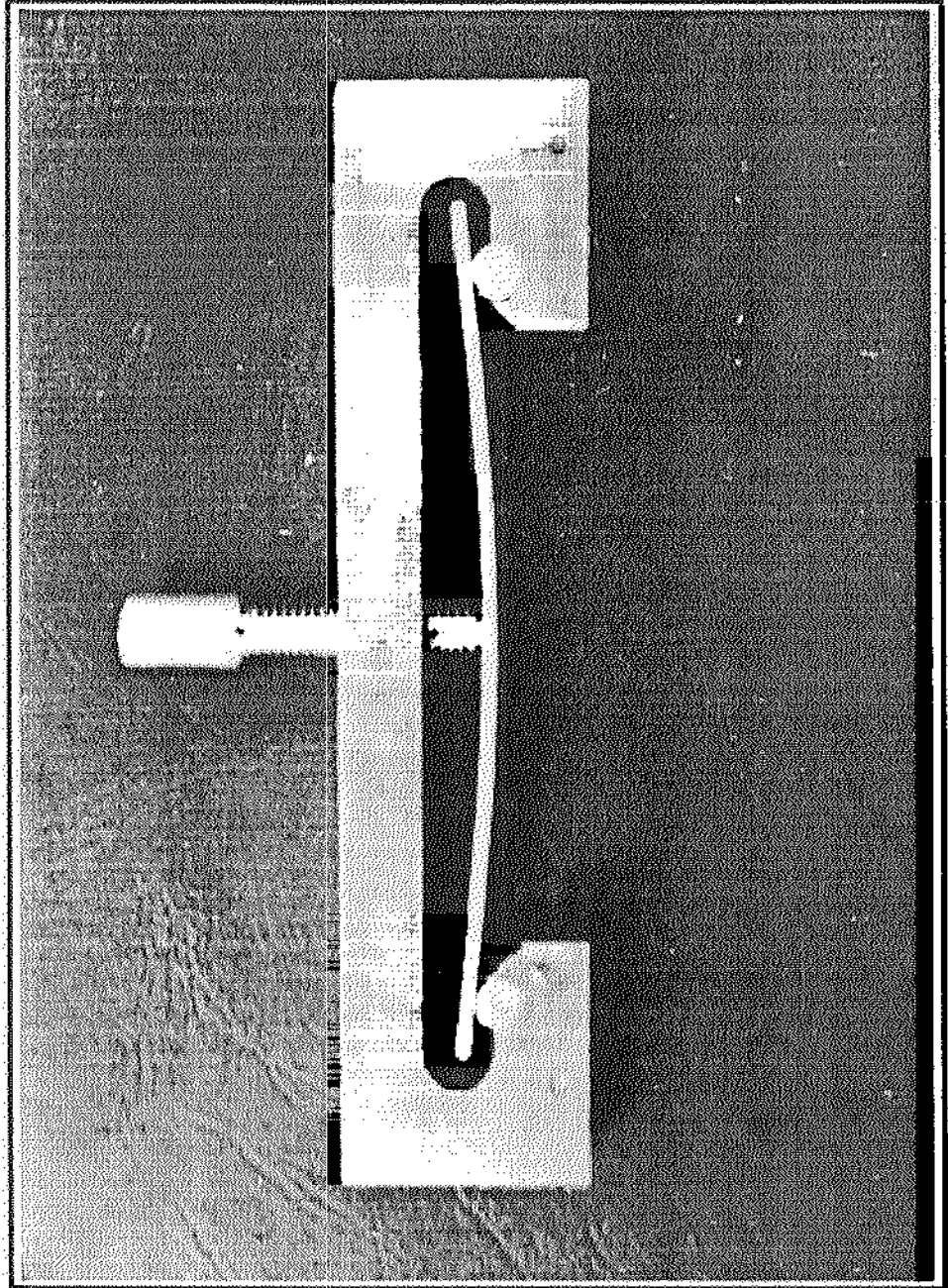


Figure 2.9: Three-point loaded specimen

The maximum bending elastic stress at midspan in the outer fibers of the three-point loaded specimen was calculated from the following relationship (Fig. 2.10):

$$\sigma = \frac{6Ety}{H^2} \quad [51,52]$$

Where: σ = maximum tensile stress,
 E = modulus of elasticity,
 t = thickness of specimen,
 y = maximum deflection and
 H = distance between outer supports.

The test specimens were tested at an outer fiber stress corresponding to 25,50,75 and 95% of the yield strength.

L, LT and ST specimens were used to represent different direction of loading (i.e. in the L, LT, and ST direction).

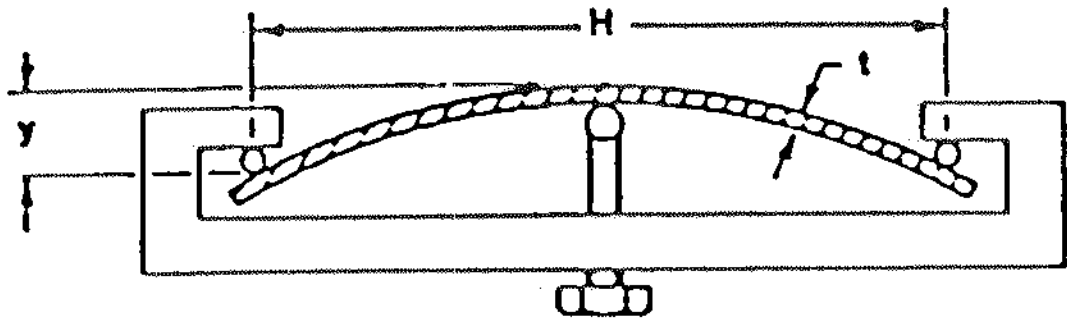


Fig. 2.10: Schematic specimen and holder configuration [51]

2.6.2 Stress Corrosion Test Environments

Preliminary experiments to evaluate SCC resistance of 7075-T6 specimens were conducted using six different SCC test solutions (having different chemical composition); in order to test the severity of these solutions as a SCC promoter, these are:

1. 3.5% sodium chloride (NaCl) solution prepared per ASTM G44-88 [53].

This is the most well known SCC test solution for high strength aluminum alloys. The solution was prepared by dissolving 35gm of NaCl in 965 ml water. Solution pH was adjusted to 6.5 ± 0.5 using hydrochloric acid (HCl) and temperature to $27^\circ\text{C} \pm 2^\circ\text{C}$.

The exposure time for specimens in this solution was 1008 hours (i.e. 42days).

2. Aqueous solution containing 3% sodium chloride (NaCl), 0.5% hydrogen peroxide. (H_2O_2) (30%), 100ml/l 1 N sodium hydroxide (NaOH), and 20ml/l acetic acid (CH_3COOH) (100%), which is an accelerated stress corrosion test solution for 7000-series aluminum alloys [54].

The solution was prepared per the above specifications, the solution pH was adjusted to be 4.0 ± 0.5 using acetic acid and was adjusted to $18^\circ\text{C} \pm 2^\circ\text{C}$.

The test duration for specimens exposed to this solution was 672 hours (i.e. 28 days).

3. Aqueous 1% sodium chloride (NaCl) plus 2% potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$).

This is a fast stress corrosion test solution for 2000-series and 7000-series aluminum alloys with 168 hours maximum exposure time [55].

In this work the solution was prepared in accordance with the above specification, the solution temperature was adjusted to $60 \pm 2^\circ\text{C}$ whereas pH was only measured and found to be 3.60.

The test duration for specimens exposed to this solution was 168 hours (7days).

4. Aqueous solution containing 2% sodium chloride plus 0.5% sodium chromate (Na_2CrO_4) which is used mainly as an inhibitor for pitting and exfoliation corrosion.

This is a test solution used for high strength aircraft alloys [56].

The solution was prepared per the above specification with the pH adjusted to 3.0 ± 0.5 by HCl and temperature to $40\pm 2^\circ\text{C}$.

The test duration for specimens exposed to this solution was 672 hours.

5. Aqueous solution containing 3.5% sodium chloride (NaCl) plus 0.5% sodium chromate (Na_2CrO_4).

This is also a test solution used for high strength aircraft alloys [37].

The solution was prepared per the above specifications with the pH adjusted daily to 3.0 ± 0.5 and temperature to $40\pm 2^\circ\text{C}$.

The test duration for specimens exposed to this solution was 336 hours (i.e. 14 days).

6. Non-Standard aqueous solution containing 5% sodium chloride (NaCl) plus 0.5% sodium chromate (Na_2CrO_4).

This solution was prepared during this work based on the above solutions and the results obtained.

The pH was adjusted daily to 3.0 ± 0.5 and temperature to $40\pm 2^\circ\text{C}$.

The test duration for specimens exposed to this solution was 168 hours (i.e. 7 days).

Each of the solutions was changed every week.

In all of the test solutions the specimens were suspended during exposure as shown in Fig. 2.11.

2.6.3 Failure Criteria

The failure criteria was based on the first detection of cracks on specimens examined under of 30X.

2.6.4 Inspection Period

Specimens were inspected every 8 hours.

2.6.5 Optical Metallography

Optical metallography is an excellent tool for examining the specimens microstructure and the determination of peening depth, because various etchants may be used to exhibit the microstructure and others can be used to reveal near-surface cold work in the cross sections of an alloy [57].

Specimens were cut for metallographic observation across the specimen's width. After mounting they were grinded and polished.

After polishing, the following etchant was used to reveal the microstructure:

10gm NaOH,

90 ml water [57].

This etchant is commonly used for aluminum alloys, the samples were immersed in this etchant; which was heated to 70°C, for 5-20 sec, then they were dipped in concentrated nitric acid HNO_3 to remove stain [57].

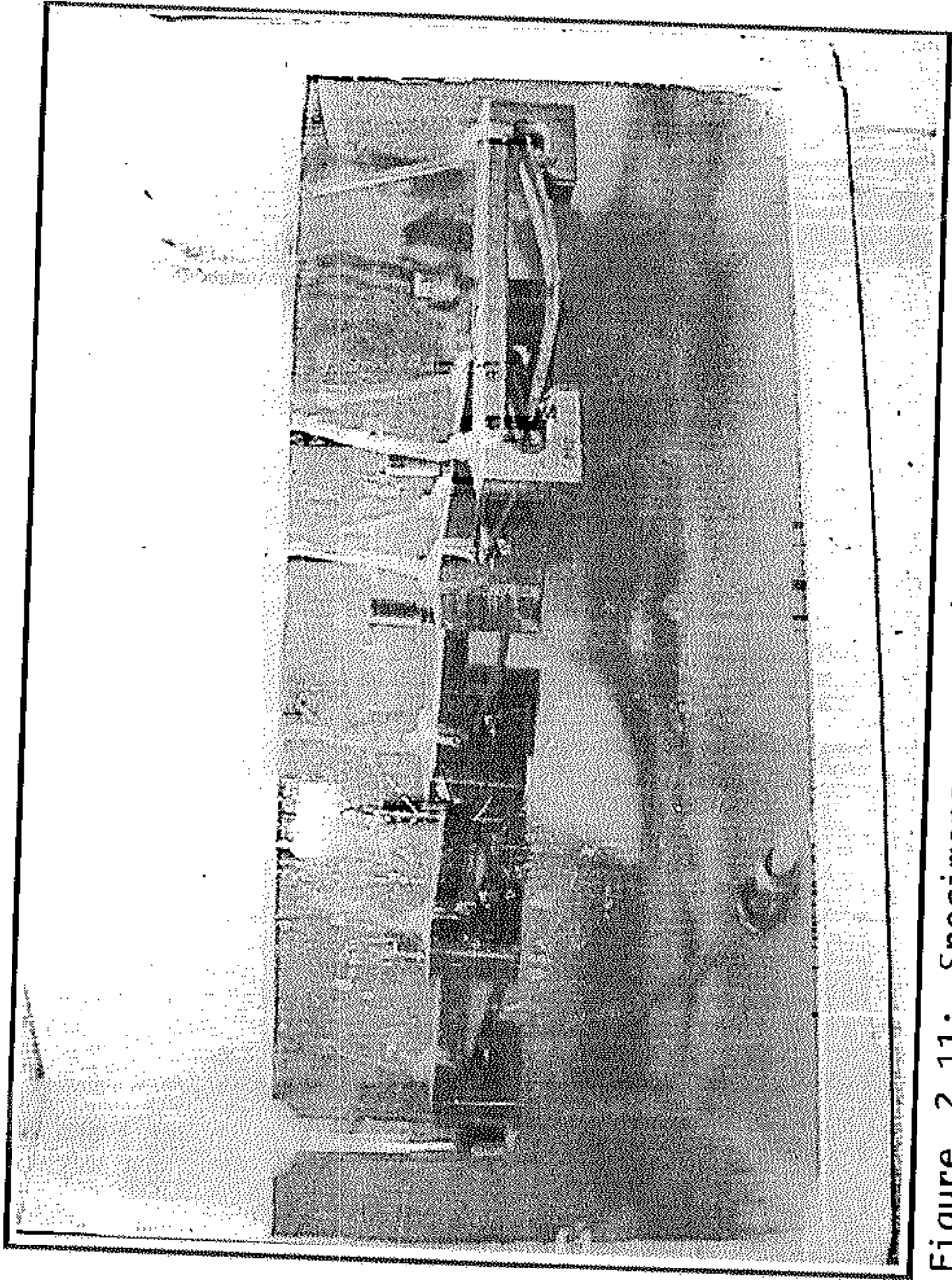


Figure 2.11: Specimens exposed to 3.5% NaCl plus 0.5% Na₂CrO₄

To reveal the plastically deformed layer and the compressive layer produced by shot peening the specimens were etched in 10% aqueous phosphoric acid (H_3PO_4) for 5-20 min at room temperature. The etched samples were then ready for metallographic observation[57].

CHAPTER THREE
RESULTS AND DISCUSSION

CHAPTER THREE

RESULTS AND DISCUSSION

3.1 Selection of Testing Environment

Environments that can promote SCC in laboratory test are several [4]. In this investigation six testing environments (solutions) were tried, these were discussed extensively in chapter two.

The severity of each environment was tested using as-machined (unwelded) 7075-T6 L, LT, and ST constant strain specimens stressed to 25, 50, 75, and 95% of the yield strength.

The effects of each of these environments on the exposed specimens are discussed below:

- 1- The experiments were conducted first in 3.5% NaCl solution, the specimens were exposed to this solution for 1008 hours. All the specimens as well as their stressing fixtures placed in such solution were noticed to suffer severe pitting as shown in figure 3.1. No SCC was detected on any specimen. Thus SCC was inhibited by pitting, i.e.; the free potential of the specimens in this electrolyte might be restricted by pitting potential which is slightly higher than SCC potential as shown in figure 3.2 [4].
- 2- Specimens exposed to the second solution which contained 3% NaCl, 0.5% H₂O₂, 100 ml/l NaOH and 20 ml/l CH₃COOH, experienced only severe exfoliation corrosion along with their fixtures as shown in figure 3.3. No signs of cracks were detected within the exposure period; 672 hours.

It is well known that high strength aluminum alloys; especially 7075-T6 alloy; are highly susceptible to exfoliation corrosion in marine (chloride) environments and its effects are stimulated further by acidic deposits [4,16]. This solution which contained high percentage of chloride was acidified to pH 4. Thus this acidified solution might promote exfoliation corrosion by restricting the free potential of the specimens exposed to such electrolyte to

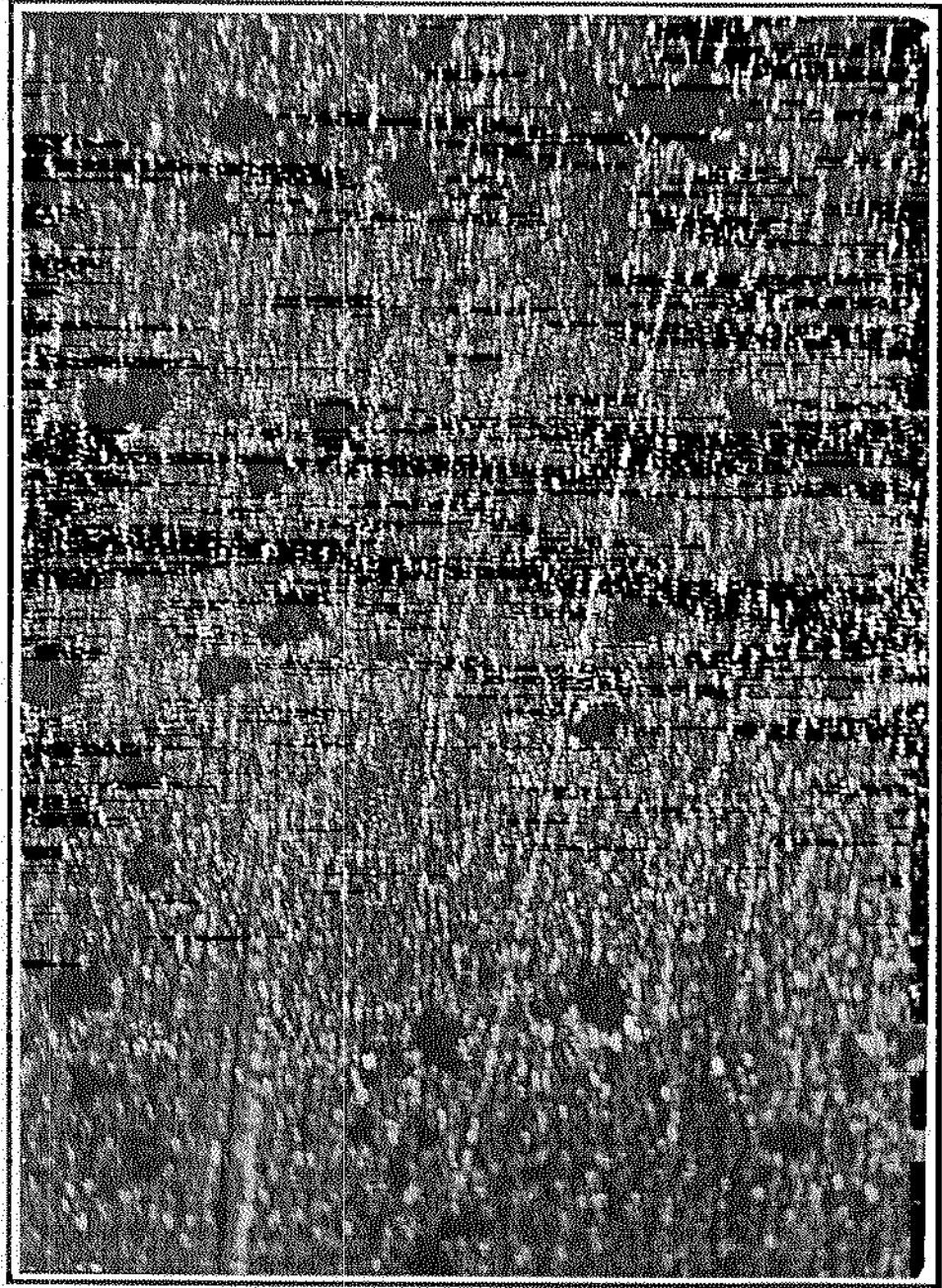


Figure 3.1: Pitting corrosion of an as-machined L specimen exposed to 3.5% NaCl solution for 1008 hours (30X)

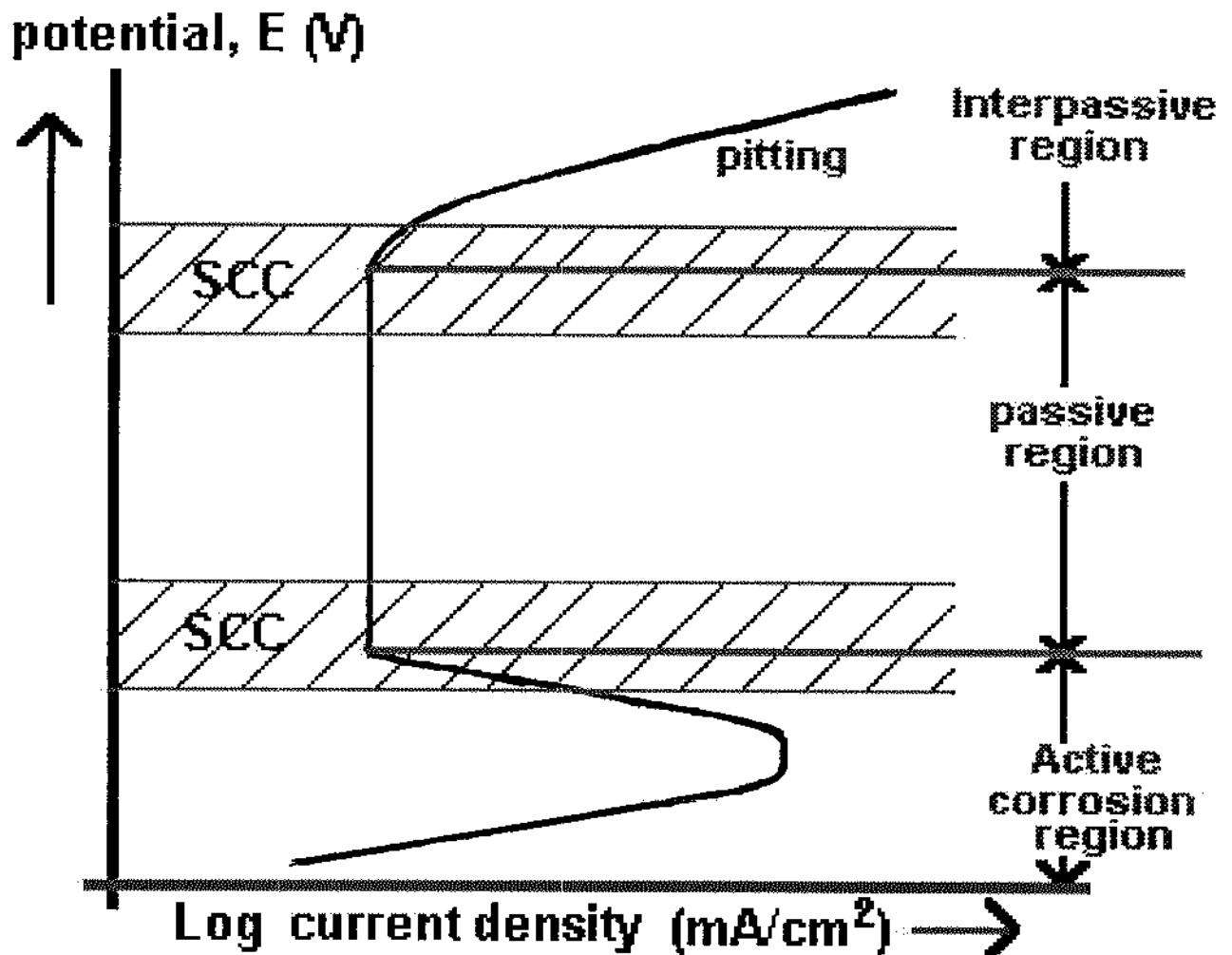


Fig 3.2: Potentiokinetic polarization curve & electrode potential values at which SCC appears [4]

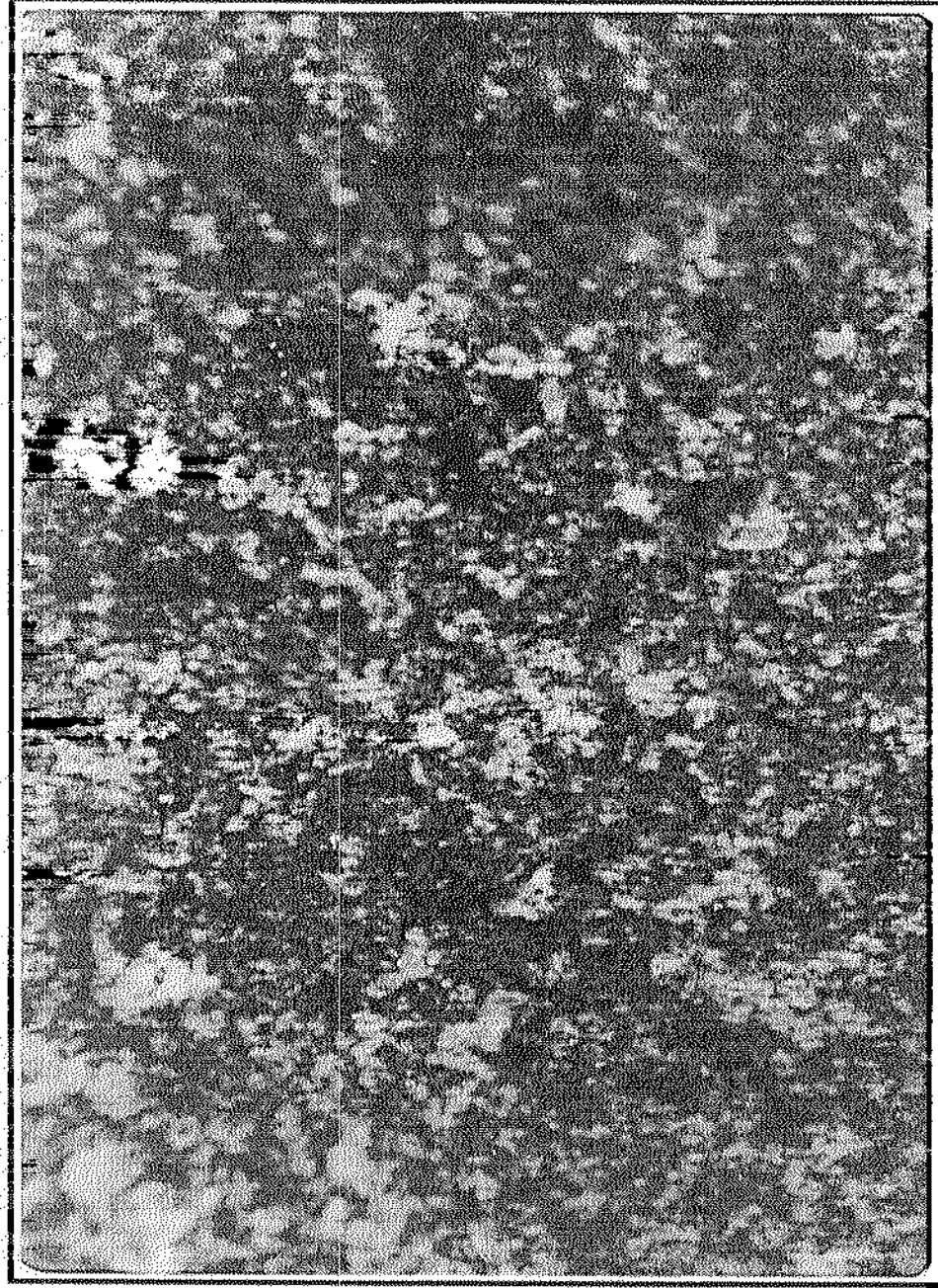


Figure 3.3: Exfoliation corrosion of an as-machined specimen exposed to 3% NaCl, 0.5% H₂O₂, 100ml/l CH₃COOH for 672 hours (30X)

the active potential which is usually lower than the SCC potential as shown in figure 3.2.

The specimens exposed to the first and the second solutions developed potential in the interpassive and active regions respectively in which SCC was inhibited and another unwanted types of corrosion was produced. Thus these two solutions were excluded.

3- The severity of four other types of solutions was tested, these are;

- * 1.0 % NaCl plus 2% $K_2Cr_2O_7$, exposure time 168 hours.
- * 2.0 % NaCl plus 0.5% Na_2CrO_4 , exposure time 672 hours.
- * 3.5 % NaCl plus 0.5% Na_2CrO_4 , exposure time 336 hours.
- * 5.0 % NaCl plus 0.5% Na_2CrO_4 , exposure time 168 hours.

All specimens exposed to these solution were unattacked by any kind of corrosion. An example is shown in figure 3.4.

It is well known that all chromates and dichromates are excellent passivating inhibitors used mainly to prevent pitting and exfoliation corrosion of high strength aluminum alloys by shifting the specimens potential to the passive potential [4,13]. Since all of the above mentioned solutions contained chromate or dichromate, it is believed that the specimens exposed to such solutions did not corrode since they were passivated, i.e. isolated from the aggressive solutions by passive films.

It is well established that many of potentials within which SCC occurs are the lower and higher passive regions as shown in figure 3.2. [4,5,6]. Thus since as-machined specimens exposed to the above four solutions had developed potentials in the passive range it was suggested that such solutions could be SCC promoters. Nevertheless it was believed that the applied elastic stress levels were not high enough to produce SCC, so welded specimens were tested in such solutions. The severity of each of the last four solutions was tested again using welded L, LT and ST stressed to 95% of the yield strength, test results are as follows;

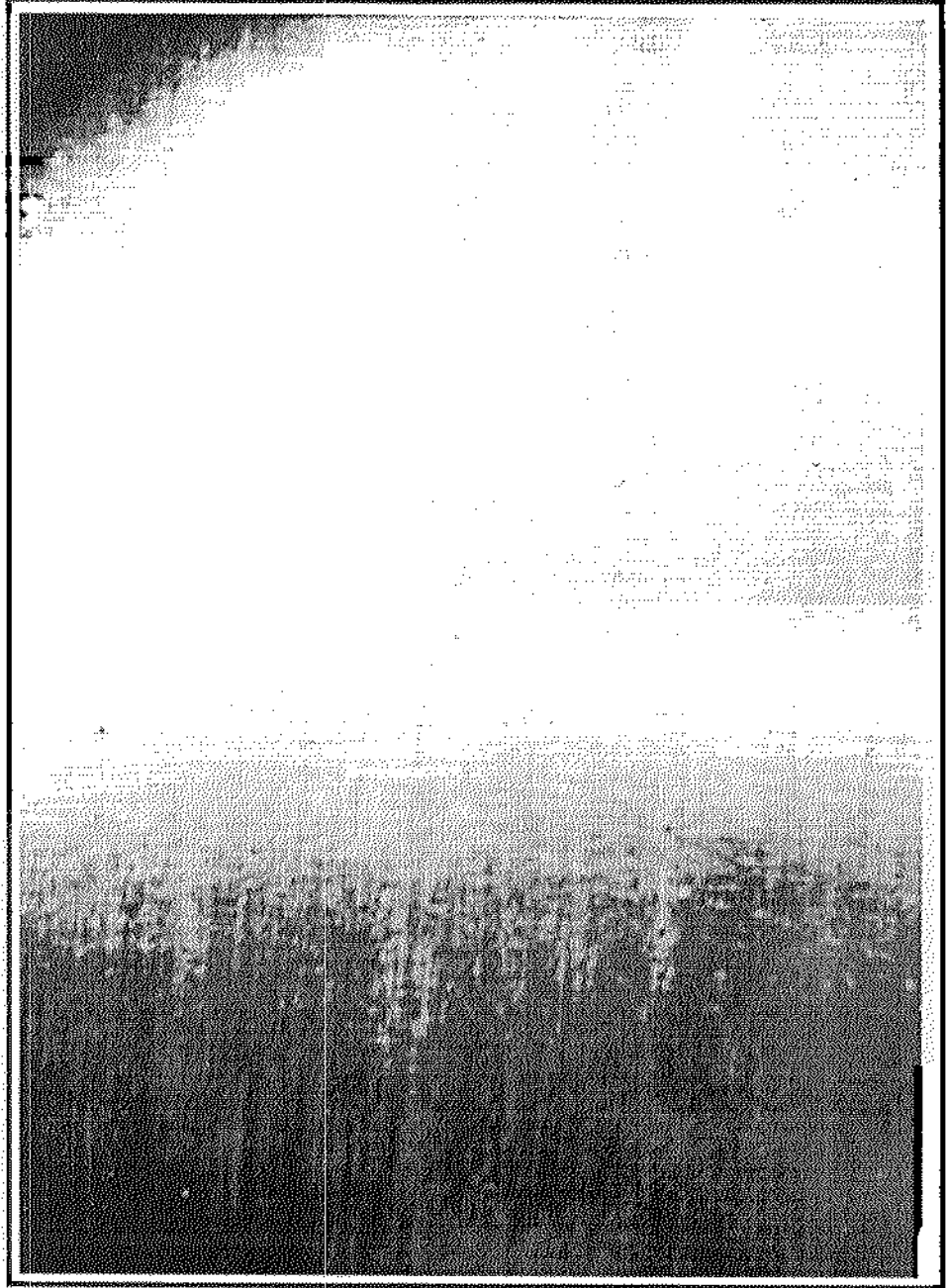


Figure 3.4: Surface appearance of an as-machined specimen exposed to 1% NaCl plus 2% K₂Cr₂O₇ solution for 168 hours (30X)

- A. Welded specimens exposed to 1% NaCl and 2% $K_2Cr_2O_7$ for the maximum exposure period; i.e. 168 hours, did not corrode. Given that this solution contained low percent of NaCl and high percent of $K_2Cr_2O_7$; the dichromate was very effective in preventing any type of corrosion by passivating the aluminum specimens and preventing breakdown of the passive film. Thus preventing SCC.
- B. ST specimens exposed to 2% NaCl plus 0.5% Na_2CrO_4 all failed within 672 hours. L and LT specimens did not fail.
- C. Specimens exposed to 3.5% NaCl plus 0.5% $K_2Cr_2O_7$ all failed within 17 to 48 hours, depending on the grain orientation which will be discussed later in details.
- D. Specimens exposed to the non-standard solution and tested in this work which contained 5% NaCl plus 0.5% Na_2CrO_4 all failed within very short time.
- * L specimens failed after 24 hours.
 - * LT specimens failed after 14 hours.
 - * ST specimens failed before 7 hours, since the cracks were detected after 7 hours, it is therefore suspected from the cracks appearance that failure occurred before 7 hours.

These excellent results were not expected from such solution since the chloride percentage in such highly acidified solution (pH 3) is very high. Thus there was a risk of exfoliation corrosion, but apparently the low percent of chromate was very effective in preventing such risk.

From the above it was found that the most convenient environment for the experimentation purposes in this work was that containing 3.5% NaCl plus 0.5% Na_2CrO_4 which caused SCC failures within reasonable time varying from 17 to 48 hours.

All the results that are going to be discussed further on were obtained using this solution.

3.2. Selection of Testing Environment Conditions

3.2.1 Selection of Testing Environment pH

It was observed that when the solution pH was initially adjusted to 3.0 (conforming [37]), it raised to approximately 4.5 due to the aluminum corrosion products. It was also found that at the latter pH, the cracks propagation was retarded. Thus the pH was adjusted to 3.0 daily.

Nevertheless the SCC susceptibility decreased significantly when the solution became highly acidic, i.e. when pH was around 1.5 to 2.0.

There is no agreement in the literature about the bulk solution pH effect on SCC susceptibility of aluminum alloys [12,15]. Nevertheless there is a general agreement that the solution pH inside corrosion cracks in 7075-T6 falls near 3 in chloride-containing environment [12,15].

3.2.2. Selection of Testing Environment Temperature

It was observed that the SCC susceptibility of the specimens increased significantly with temperature increase. Thus solution temperature was adjusted to 40 C to accelerate the cracks initiation and propagation. It is well established that temperature increase makes the diffusion of solution faster. Thus the rate of species transport and diffusion to the crack tips is increased [4,12]. Moreover there is general agreement in literature that SCC performance of 7XXX aluminum alloys significantly improves at temperatures around 40 C [12].

3.3 Effect of Grain Orientation on SCC

When the welded L, LT and ST specimens stressed to 95% of the yield strength were exposed to the solution, the following results were obtained:-

- * L specimens failed after 48 hours.
- * LT specimens failed after 36 hours.
- * ST specimens failed after 17 hours.

From the above it is noticed that 7075-T6 alloys have the highest SCC resistance in the longitudinal direction, a moderate resistance in the longitudinal transverse direction and the lowest resistance in the short transverse direction. There is general agreement in the literature that 7075-T6 is highly susceptible to SCC in the short transverse direction and relatively immune to SCC in the longitudinal direction [1,3,4,13].

3.4. SCC Behavior

SCC occurred in all the welded specimens on the three orientations at different times but always in the same manner.

When the specimens are exposed to the solution, gas bubbles immediately started to accumulate on two narrow bands of the heat affected zone (HAZ) a few millimeters (5 to 10mm) from the line representing the center of the weld. These bubbles were localized on edges, upper and lower surface confined on these narrow bands. It is suggested that these bubbles are hydrogen bubbles since the solution used is an acidic solution.

After few hours aluminum corrosion products started to accumulate on the two narrow bands where the bubbles were appearing forming two well defined bands of yellow color as shown in figure 3.5a. During specimen inspection when the corrosion products were removed these distinct regions appeared to have a surface texture as shown in figure 3.5b, i.e; dimple like structure. This dimple layer was very loose, so it peeled off the surface in form of very fine flakes as shown in figure 3.5c.

Then the surfaces within these distinct regions would be full of cracks which propagated very fast and peeled off in form of large fragments of metal. This is demonstrated in figures 3.5d, 3.5e, 3.5f and 3.5g.

Hence these distinct regions appeared to be highly susceptible for SCC and prolonged exposure in the testing solution produce severe crackings confined to

these regions as shown in figures 3.5h and 3.5i. No cracks propagation was in the weld, the HAZ immediately adjacent to the weld metal, or the base metal.

The explanation of these results can be done by examining the microstructure of the specimen, such examination revealed that the HAZ susceptible to SCC has a different microstructure from that of the HAZ immediately adjacent to the weld and the base metal. It is well known that in general, the HAZ is divided into two regions; the high temperature region which is beside the weld and the lower temperature region which is few millimeters away from the weld [58].

The high temperature HAZ has a microstructure almost similar to the base metal as shown in figures 3.6 and 3.7. Because during welding this region was subjected to high temperature similar to that needed for solution annealing, also this region cooled down very quickly because welding always produce high rate of cooling in aluminum alloys due to the high coefficient of thermal conductivity for these alloys [16]. Accordingly the structure of this region actually is solution annealed then quenched, aging may also be produced during the welding process. Thus the structure of this region resembles the structure of the base metal at T6 condition.

This means that the high temperature HAZ is subjected to the same thermal treatment that the base metal usually produced at, i.e. T6 condition. Thus it can be explained why such region did not suffer from any type of corrosion especially SCC, this is due to the good resistance of the T6 condition to SCC [16].

The low temperature region; i.e. the susceptible region to SCC, appeared to have a completely different microstructure from that appeared in the high temperature HAZ. This region is also affected by the welding; however lower heat dissipated through this region and different value of cooling rate exhibited in this region. During welding the alloying elements at this region will be partially dissolved and during cooling they start to precipitate in a random

Figure 3.5: Stages of SCC development in the welded L specimens exposed to 3.5% NaCl plus 0.5% Na₂Cro₄ for 576 hours

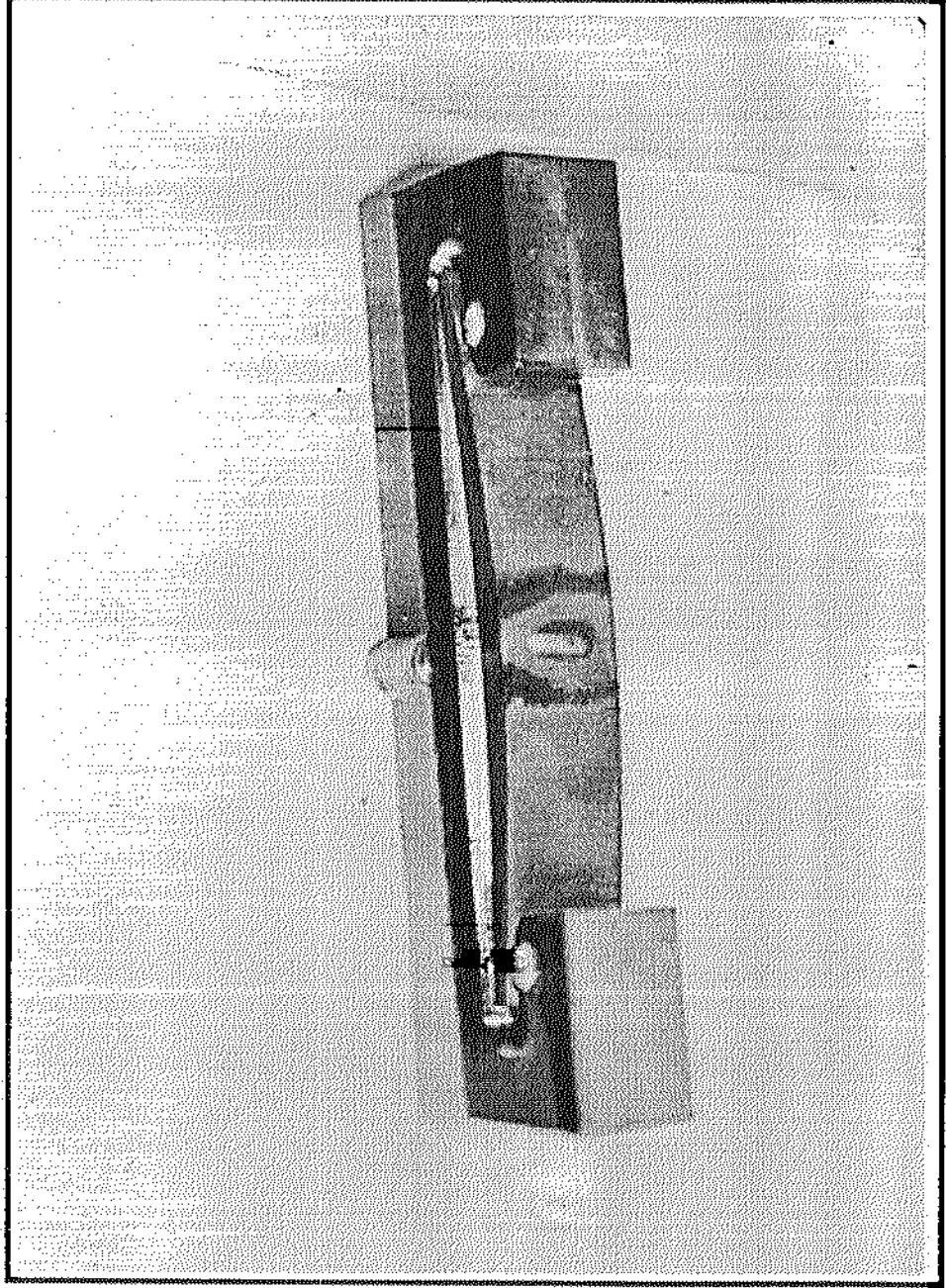


Figure 3.5a: the formation of two well-defined narrow bands



Figure 3.5b: Dimple like structure formed beneath the narrow band susceptible to SCC (30X)



Figure 3.5C: Peeling of the dimple layer in form of fine flakes (30X)

Figure 3.5d-3.5h: Show crack propagation stages uptill complete fracture



Figure 3.5d: Peeling of large fragments off the surface (30X)



Figure 3.5e: Peeling of layer fragments of metal off the surface (30X)

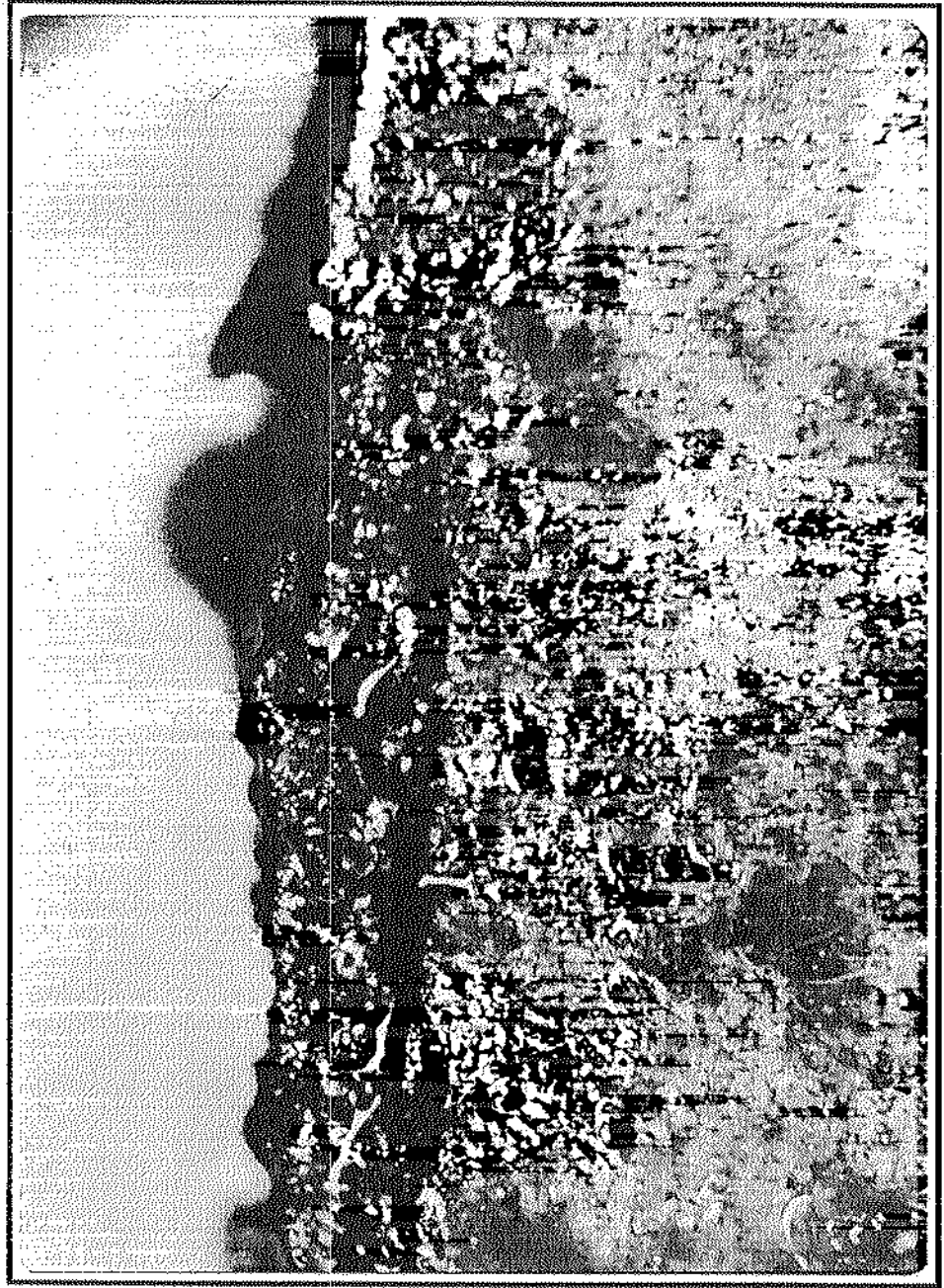


Figure 3.5f: Cracking of surface edges (30X)



Figure 3.5.g: Micrograph of the SCC (400X)

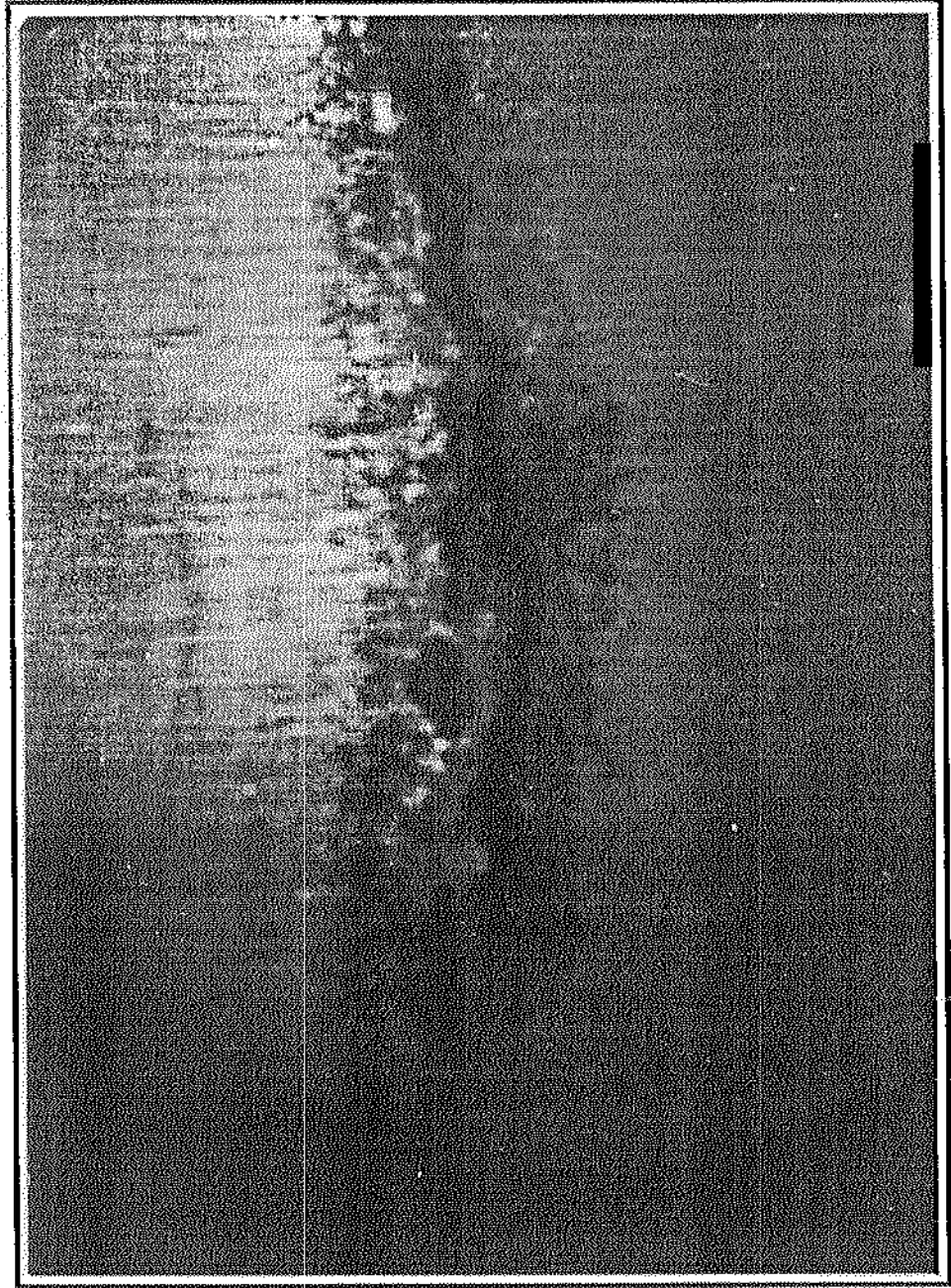


Figure 3.5h: The macrostructure of the fracture surface (15X)

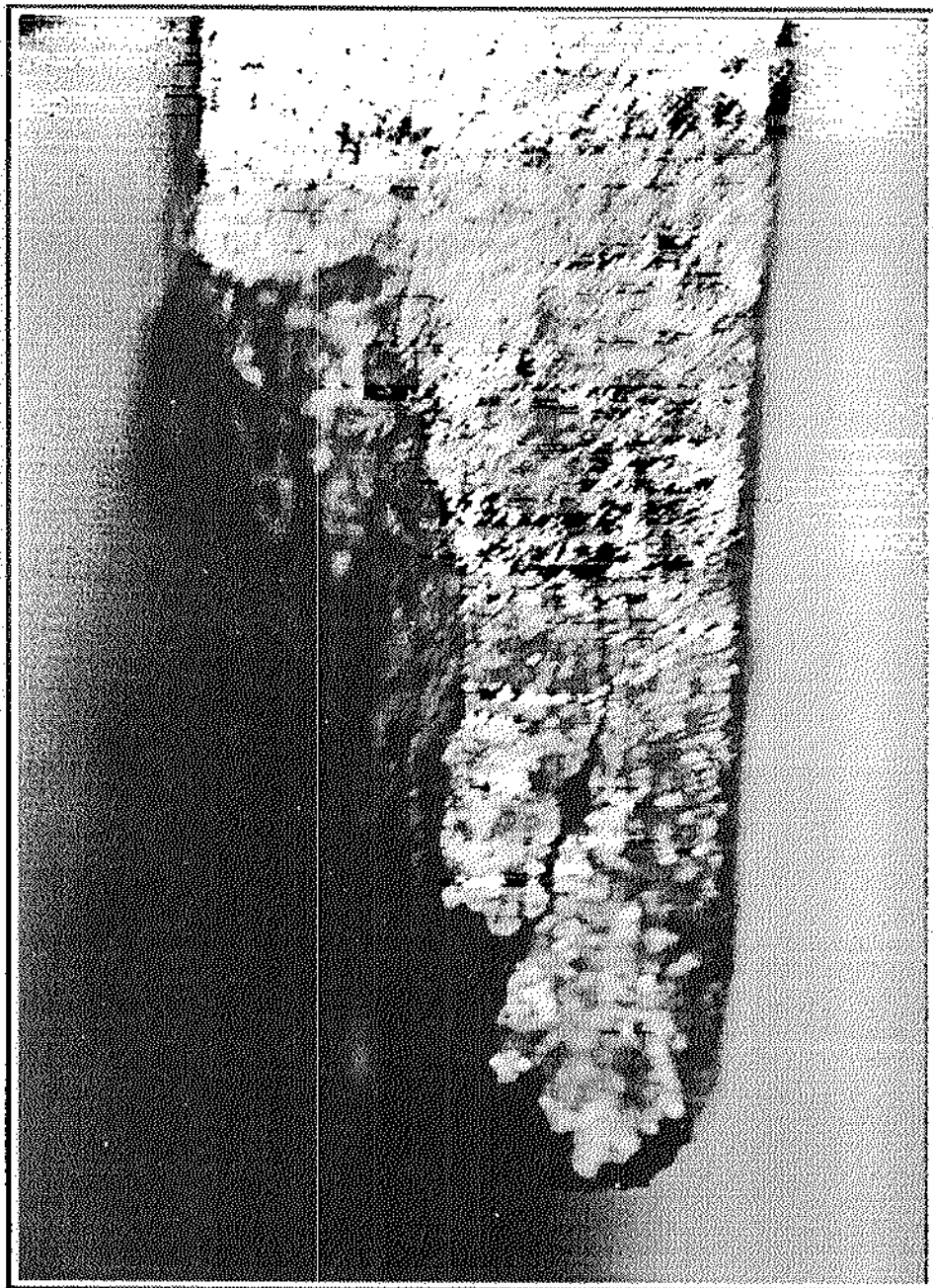


Figure 3.5i: Complete fracture of the specimen due to SCC after 576 hours of exposure to the solution (15X)



Figure 3.6: Microstructure of base metal (grains are oriented in LT direction) (200X)



Figure 3.7: Microstructure of high temperature HAZ, i.e . HAZ immune to SCC (grains are oriented in the LT direction) (100X)

manner across the grain boundaries and within the grains producing non-homogeneous precipitation and agglomeration of large unknown complex intermetallic compounds as shown in figure 3.8. This non-equilibrium structure introduce dislocations and distort the grain boundaries [16].

It seems that different phases from that in the high temperature HAZ and base metal were produced in this region as can be detected from the different colors of the grains in it (figures 3.9, 3.10 and 3.11). These grains obviously have lower corrosion resistance than those grains in T6 condition in the base metal and high temperature HAZ.

Previous investigators [12,16] reported SCC failure in the same region and such failure was attributed to the potential developed in this region which was lower than those of the base metal and the high temperature HAZ and was due to the metallurgical changes experienced by this region during welding. Thus it was relatively anodic with respect to the base metal and the high temperature HAZ. Thus microgalvanic action between this anodic region and the adjacent cathodic regions was produced due to which it was preferentially dissolved in the acidified solution and in the presence of the residual tensile stresses introduced during welding this area became highly susceptible to SCC [4,6,13].

Unfortunately it was not possible in this investigation to analyze the micro-constituents developed in the low temperature HAZ in order to shade the light on the exact mechanism that lead to such phenomenon.

Weld metal exhibited good corrosion resistance because no harmful precipitates or deleterious phases that can produce loss in corrosion resistance were found. This region is an ordinary cast weld as shown in figure 3.12.

3.5. Shot Peening

3.5.1 Optical Metallography

Figures 3.13, 3.14 and 3.15 exhibit clearly the depth of residual compressive stresses obtained at the surface of specimens shot peened to

Non-homogeneous precipitations



Figure 3.8a: Microstructure of the low temperature HAZ, i.e. SCC susceptible region (grains are oriented in the LT direction) (200X)

Random precipitations



Figure 3.8b: Microstructure of low temperature HAZ (grains in LT direction) (100X)

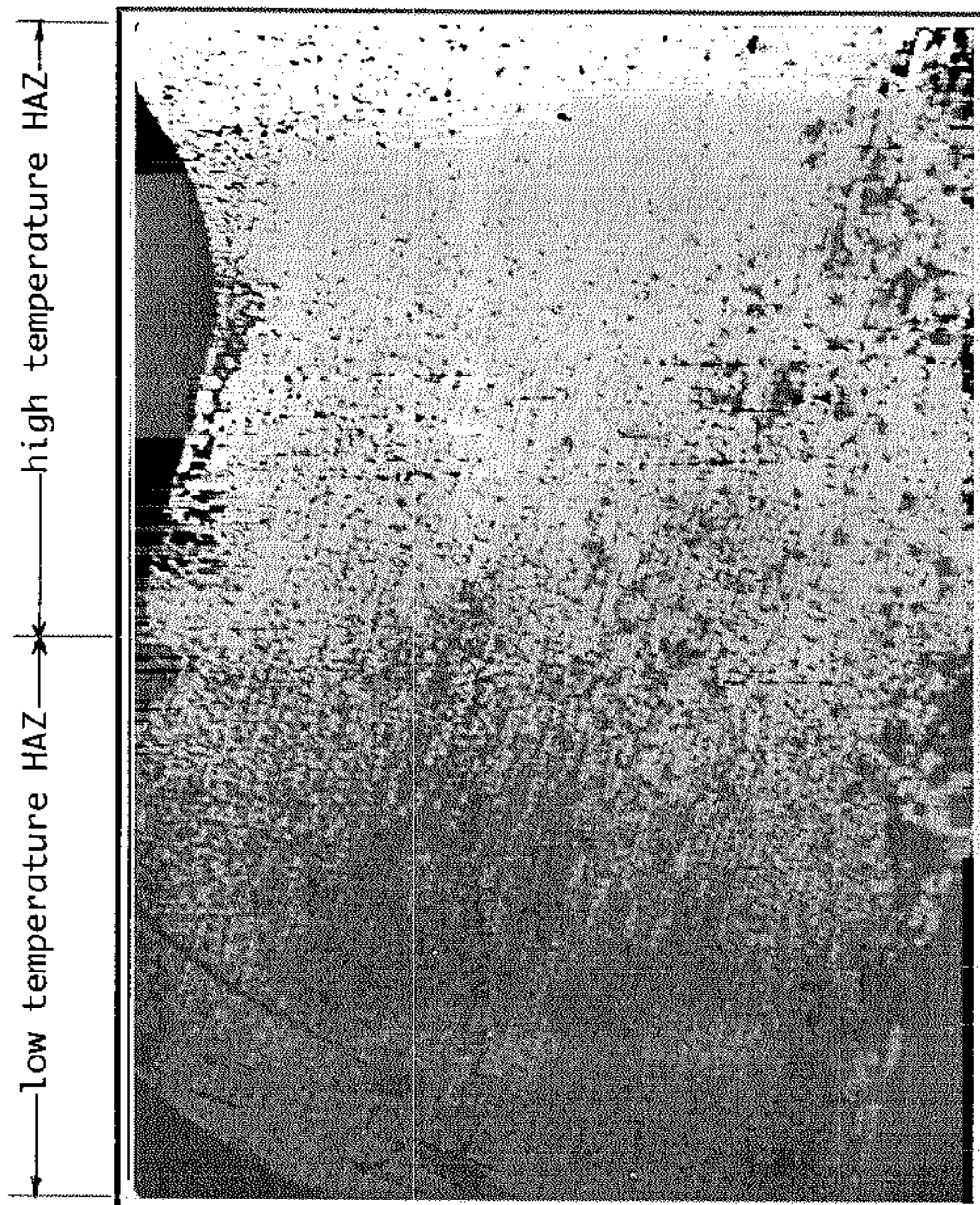


Figure 3.9: Shows the difference of grain structures between low temperature region and high temperature region (grains are oriented in ST direction) (50X)

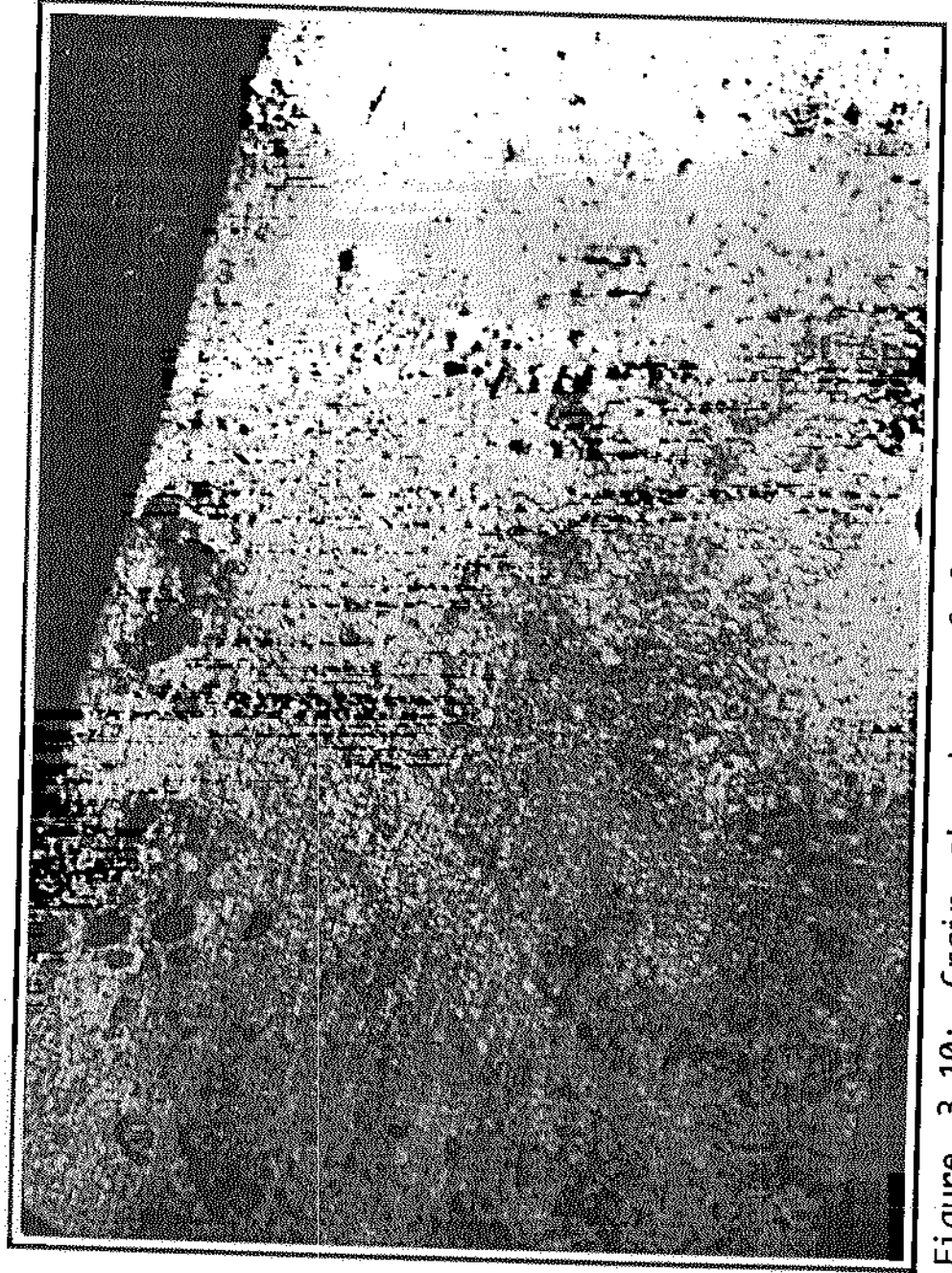


Figure 3.10: Grain structure of low temperature HAZ region (grains are oriented in ST direction) (50X)

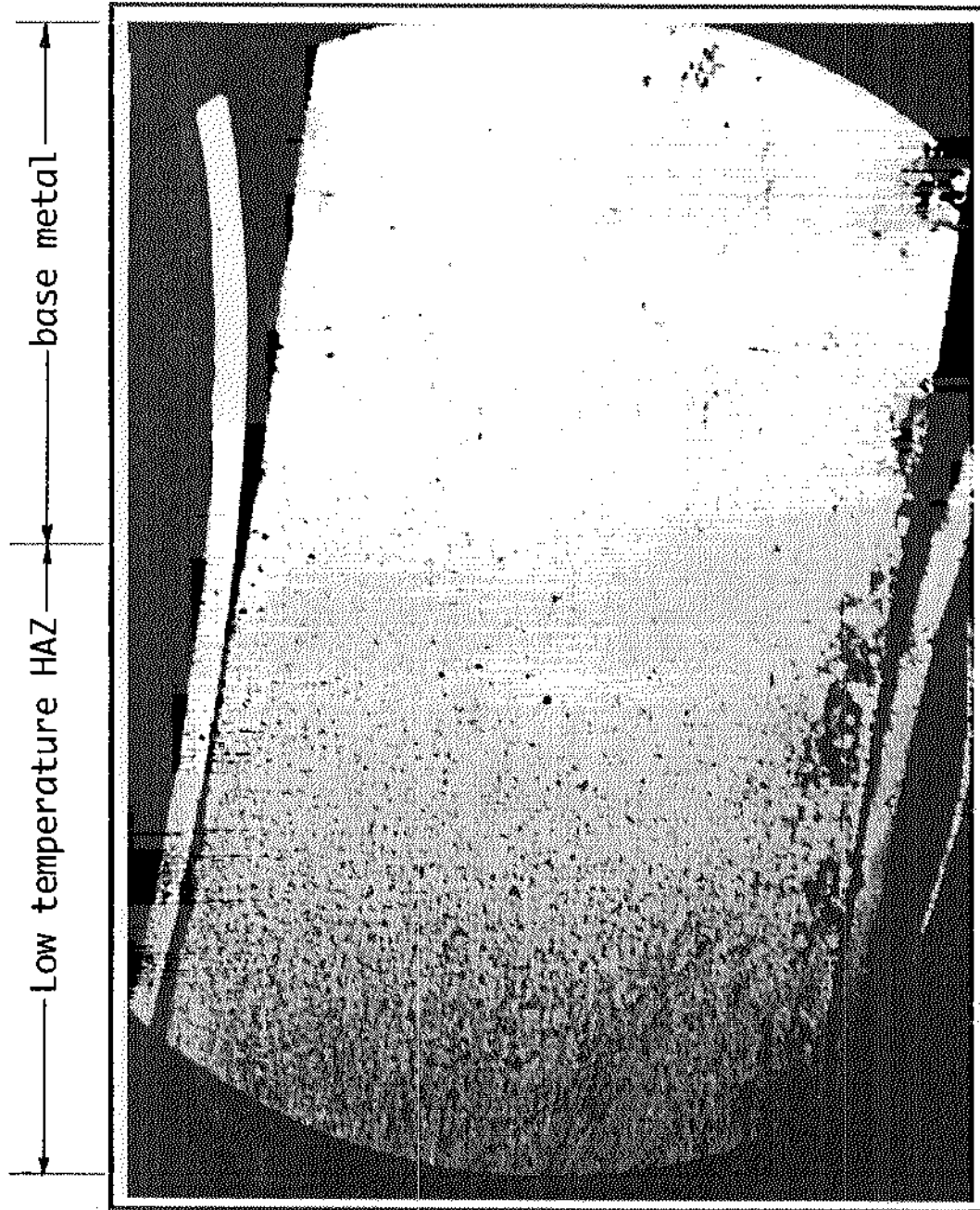


Figure 3.11: Shows the difference of grain structures between low temperature HAZ and base metal (grains are in the ST direction) (50X)

intensities of 6,8 and 10A respectively. The depth of the compressive layer were measured and found approximately as given in table 3.1

Table 3.1: Depth of Compressive Layer

Peening Intensity (A)	Depth of Compressive layer (mm)
6	0.24
8	0.35
10	0.53

From table 3.1, it is noticed that as intensity increases, the compressive layer increases. This result is in excellent agreement with the literature [37,39,59,60] in which it is well established that depth of compressive stresses increases as the intensity increases given that no overpeening effect will occur.

It is evident from figures 3.13, 3.14 and 3.15 the uniformity of the compressive layer obtained for 6, 8 and 10A peening intensities. This also agrees with large number of studies [17,61] in which the compressive layer obtained due to shot peening is reported to be very uniform.

The plastically deformed layer of an 8A peened surface was optically examined as shown in figure 3.16 and was found to be approximately equal to 50 μm , this value falls within the depth of plastically deformed layer range specified by Verpoort and Gerdes [24] who proved that such range can reach a maximum value of 300 μm for some metals in some instances.

3.5.2 Effect of Shot Peening on SCC

Welded LT and ST specimen were shot peened on the weld side to intensities of 6,8 and 10A respectively, then they were stressed to 95% of the yield strength and were exposed to the testing solution for 336 hours. The obtained results were as follows:

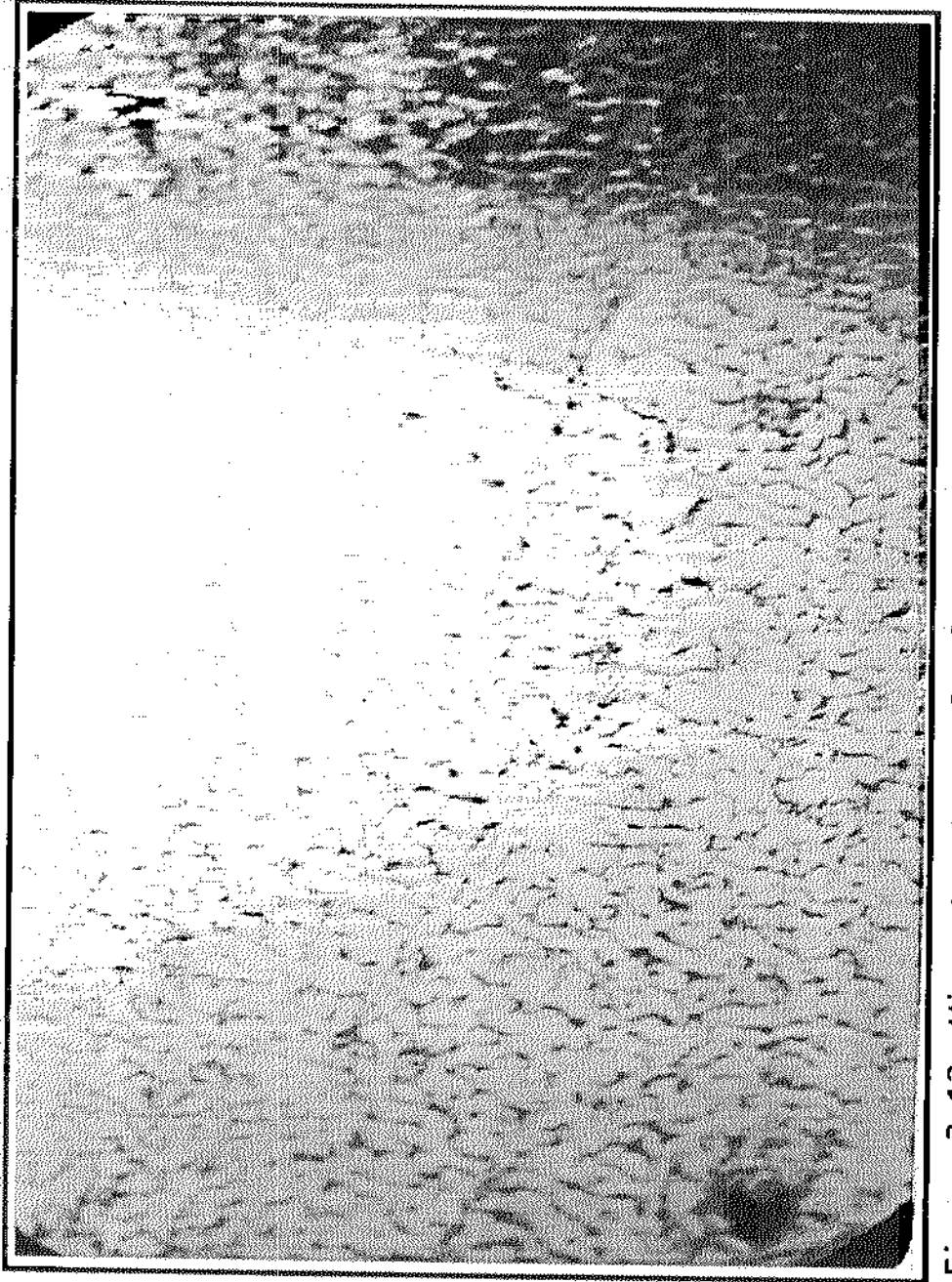


Figure 3.12: Microstructure of weld metal (200X)



0.24mm

Figure 3.13: Depth of compressive layer after shot peening to an intensity of 6A and coverage 200% (100X)



0.35mm

Figure 3.14: Depth of compressive layer after shot peening to intensity of 8A and coverage 200% (100X)



Figure 3.15: Depth of compressive layer after shot peening to an intensity of 10A, and coverage 200% (100X)

1. Shot Peened LT Specimens:

Unpeened surface and edges failed by SCC within 36 hours. Shot peened surfaces to all intensities did not fail within the exposure period; i.e. 336 hours as shown in figure 3.17. Nevertheless peened surfaces to 8A suffered minor pitting but no cracks were detected after 336 hours.

2. Shot Peened ST Specimens:

Unpeened surface and edges failed within 17 hours. Shot peened surfaces to intensities of 6 and 10A did not fail within the test period which was 336 hours.

Surfaces peened to 8A failed after 211 hours, increasing the ST specimens more than 12 times.

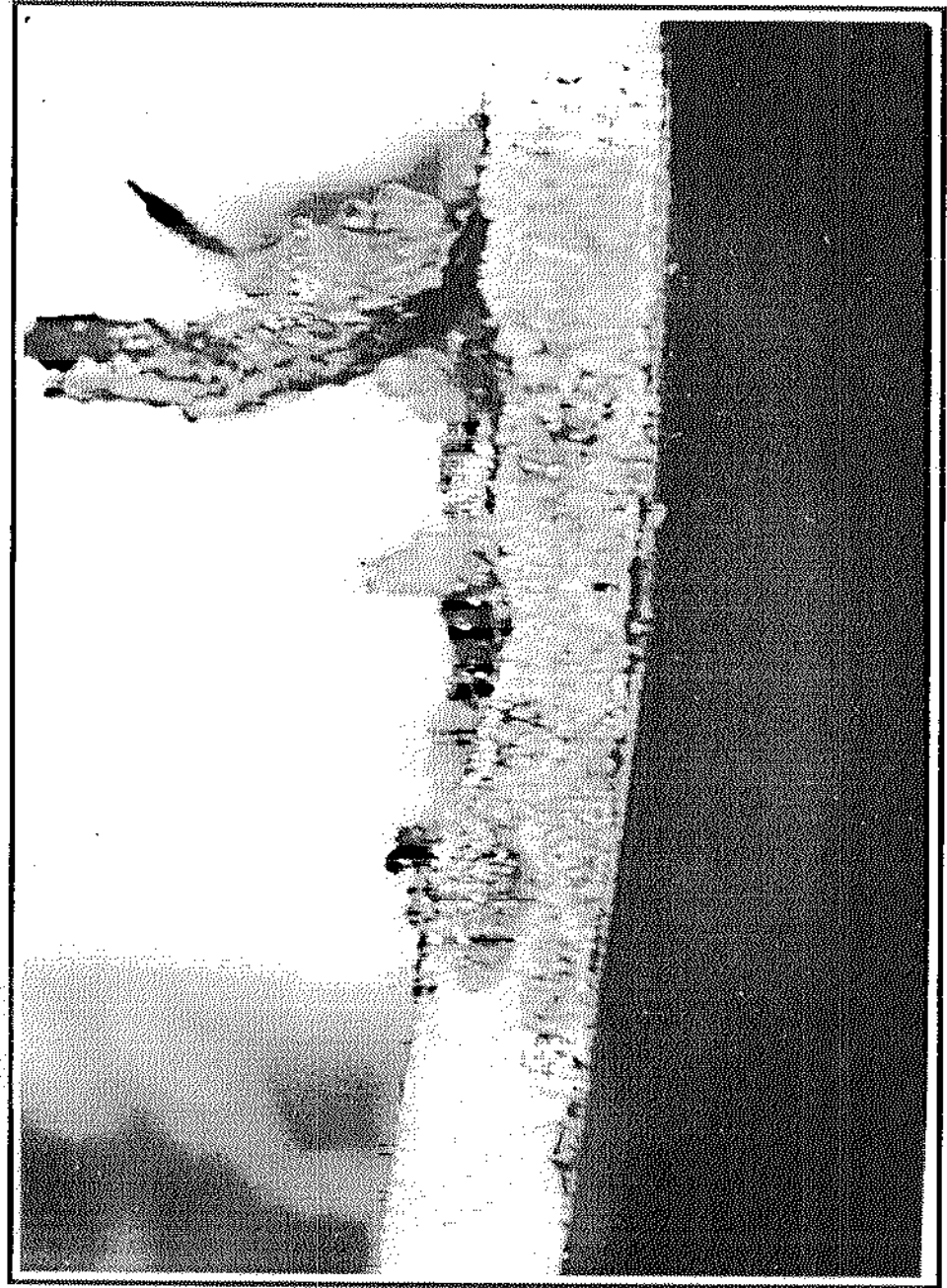
There is no obvious reason why SCC occurred only at 8A intensity in ST specimens. Nevertheless these results agree to some extent with Takemoto et al. results [32]. Thus their findings can be adopted to explain this unexpected result; specimens shot peened to 8A intensity have higher compressive stresses than those peened to 6A, but they might have lower threshold stress than those peened to 6 and 10A [32]. Unfortunately it was not possible to measure the threshold stresses of SCC after shot peening in order to understand clearly the relationship between shot peening intensity and such factor especially that Takemoto et al. did not give a clear explanation for such behavior.

3.5.2.1 Corrosion behavior of Unfailed Shot Peened Specimens

When the shot peened LT and ST specimens to intensities of 6 and 10A were exposed to the testing solution, hydrogen bubbles were observed on all the peened surface especially at the two low temperature HAZs. Then the aluminum corrosion products accumulated on these two regions forming two distinct narrow bands of yellow color as shown in figure 3.18. But when the corrosion products were removed, the surfaces below them were found free of any type of corrosion



Figure 3.16: Depth of plastically deformed layer produced after shot peening to intensity of 8A and coverage 200% (400X)



Unpeened Surface

Peened Surface

Figure 3.17: Shows peened surface and unpeened surface of a LT specimen exposed to the solution for 336 hours (10X)

as shown in figure 3.19. Minor pitting was detected below these distinct bands for LT specimens peened to 8A as shown in figure 3.20. These minor pits may represent the initiation stage of SCC [5]. Thus SCC may occur in such specimens once the compressive layer is penetrated by pitting [2,34,38].

3.5.2.2. SCC Behavior of Failed Shot Peened Specimens

SCC of shot peened ST specimens to 8A intensity was confined to the two low temperature HAZs and was similar to that of unpeened specimens except for the crack initiation which was preceded by severe pitting attack especially near the edges from which cracks initiated and propagated slowly towards the center. This behavior is demonstrated in figures 3.21 and 3.22.

It is suggested that SCC had initiated when the compressive layer was penetrated by pitting [21,34,38].

SCC failure was reported only for ST shot peened specimens since 7075-T6 alloys are highly susceptible to SCC when stressed in the short transverse direction [1,3,4,13].

It's worth stating that the SCC severity on the unpeened edges of the peened specimens was higher than that on the as-welded specimens. This can be attributed to the offsetting tensile stress at the subsurface which usually balances the compressive stress layer as shown in figure 3.23.

Thus complete coverage of all exposed surfaces is essential if shot peening is to be used as a preventive measure against SCC, since incomplete coverage may accelerate SCC due to the tensile stresses which usually balances the compressive stresses.



Figure 3.18: Formation of well-defined narrow band on shot peened surface (10X)

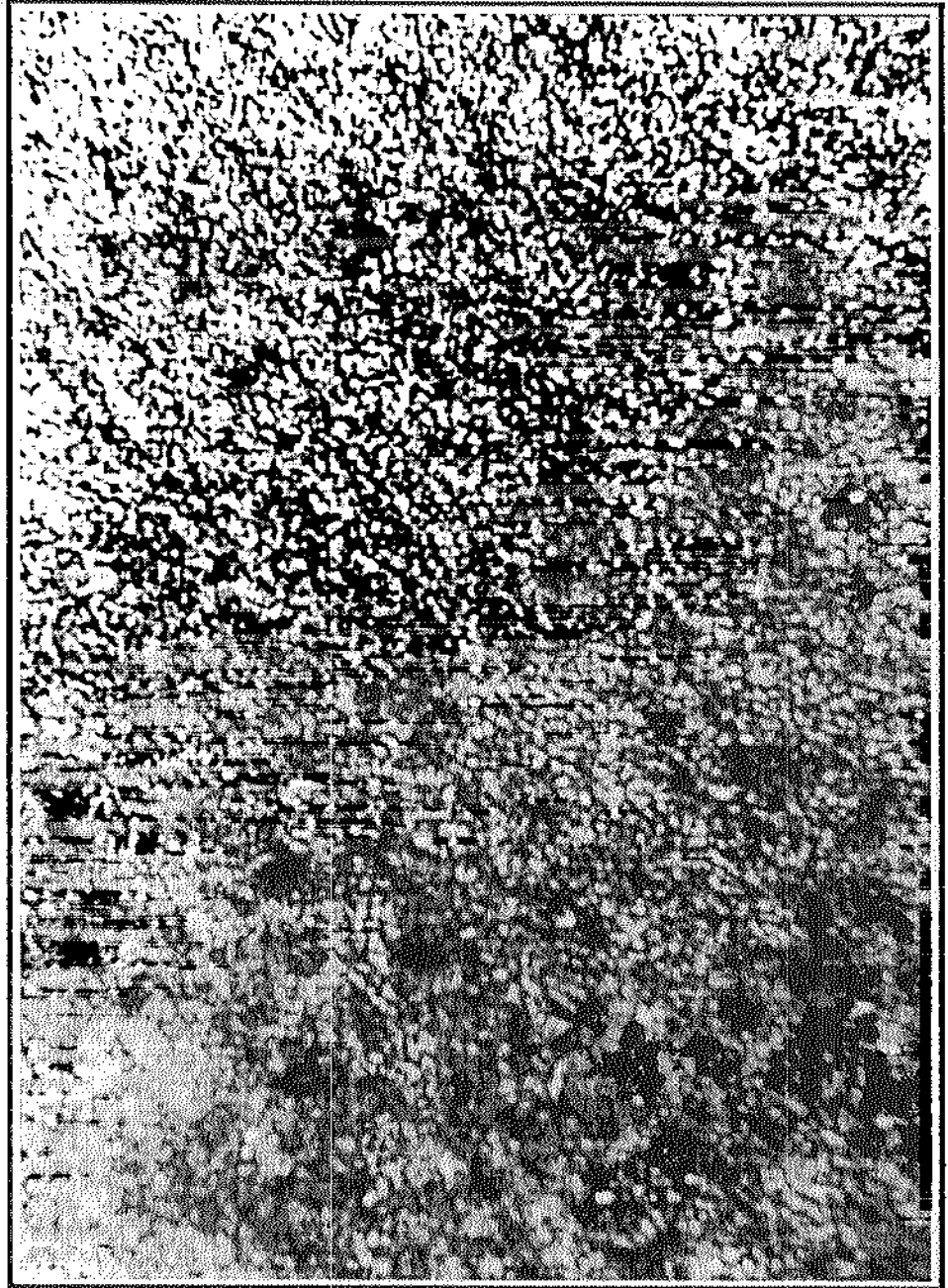


Figure 3.19: Appearance of shot peened surface beneath the low temperature HAZ (30X)



Figure 3.20 : Minor pitting corrosion of welded LT specimen shot peened to intensity of 8A, coverage 200% (30X)



Figure 3.21 : Severe pitting corrosion of welded ST specimen shot peened to intensity of 8A, coverage 200% (30X)



Figure 3.22 : SCC of welded ST specimen shot peened to 8A, coverage 200% (30X)

The beneficial effects of shot peening on SCC behavior are due to the followings:

1. The microstructure change induced on the peened surface [37,43]. It is well known that shot peening produces a homogeneously deformed surface layer with no sharp slips and high dislocation density which retards the crack initiation and prevents the breakdown of the passive film [17,24,37,43].
2. The compressive residual stresses introduced during shot peening which retards cracks initiation and propagation [21,24,31,32].

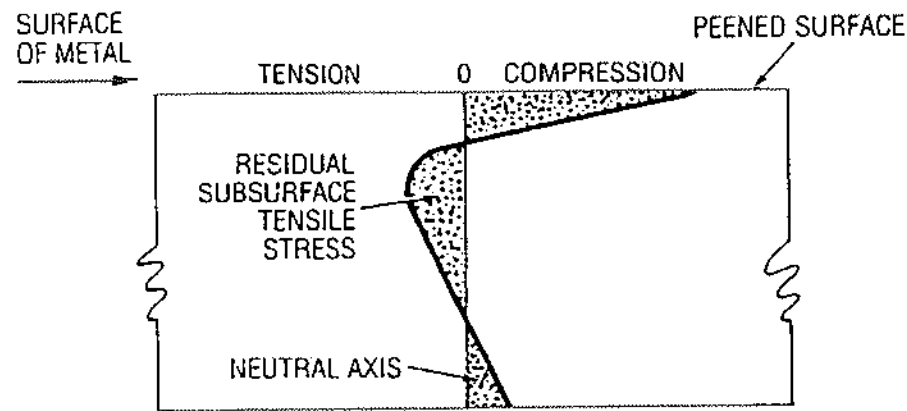


Figure 3.23: Induced Compressive and tensile stresses in a shot peened thin plate [39]

CHAPTER FOUR
CONCLUSIONS

CHAPTER FOUR

CONCLUSIONS

From the obtained results the following conclusions are drawn:

1. Welded 7075-T6 alloy is highly susceptible to SCC in the short transverse direction.
2. The dramatic loss in SCC resistance of the low temperature HAZ is attributed to the metallurgical change in such region introduced during welding.
3. Shot peening improved SCC resistance of the welded 7075-T6 alloy due to the homogeneous cold-worked surface layer and the compressive residual stresses produced during shot peening.
4. Shot peening is very beneficial in preventing or delaying SCC of 7075-T6 welded alloys given that:
 - i. The resultant compressively stressed layer is not penetrated by pitting.
 - ii. All exposed surfaces are shot peened to attain complete coverage. Thus a safety factor of 2 is usually used to guarantee 100% coverage.
5. Shot peening is superior in improving the SCC resistance of 7075-T6 welded alloys, since even in its worst conditions it has improved the alloy life to more than 12 times.

CHAPTER FIVE
RECOMMENDATIONS

CHAPTER FIVE

RECOMMENDATIONS

1. Micro-constituents of the SCC susceptible region, i.e. low temperature HAZ, are recommended to be studied extensively in order to shade the light on the exact mechanism that leads to such phenomenon.
2. Compressive stress profile introduced during shot peening need to be measured.
3. SCC threshold stresses before and after shot peening are recommended to be measured to know the exact effect of shot peening on such parameter.
4. Since shot peening is known to introduce microstructure change on the surface layer, such change is needed to be investigated metallurgically and electrochemically.
5. Other aircraft aluminum alloys (e.g. 2XXX and 6XXX) is suggested to be studied in the same manner conducted during this work.

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الملخص

تأثير الاجهادات الضغطية السطحية على مقاومة التآكل الاجهادي لسبائك الالمنيوم العالية المقاومة في محيط كلوريدي

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سبيكة الالمنيوم العالية المقاومة (7075-T6) هي سبيكة لها استخدامات واسعة في صناعة الطائرات، ولكن هذه السبيكة عرضة للتآكل الاجهادي خصوصاً في حالة اللحام. تم في هذا البحث وضع برنامج اختباري لتحسين مقاومة هذه السبيكة للتآكل الاجهادي في اوساط كلوريدية باستخدام السفع بالكريات. السفع بالكريات هي عملية اصطدام كريات متعدد على سطح مرن أو لدن مما يؤدي الى اجهادات ضغطية، مسببة نشوء التشققات وبالتالي تمنع التآكل الاجهادي. لقد ثبت أن اجهادات الشد السطحية هي العامل الرئيس الذي لا بد من وجوده لحدوث التآكل الاجهادي، لهذا فإن الاجهادات الضغطية الناشئة عن السفع بالكريات تؤدي إلى اعاقا التآكل الاجهادي في المعادن. ابتدأت التجارب المخبرية أولاً لاختيار أفضل محلول كلوريدي محفز للتآكل الاجهادي. كذلك تمت دراسة التراكيب الميتالورجية للعينات الملحومة التي أبدت قابلية شديدة للتآكل الاجهادي في بعض المناطق وعزيت هذه القابلية إلى التغير الميتالورجي الذي تعاني منه العينات عند اللحام.

وفي النهاية، تمت دراسة أثر السفع بالكريات على مقاومة العينات الملوحة للتآكل
الاجهادي باستخدام قيم مختلفة لشدة السفع حيث أظهرت هذه العملية ضمن حدود الدراسة منعاً
للتآكل الإجهادي عند قيم معينة لشدة السفع وتأخيره عند أخرى.