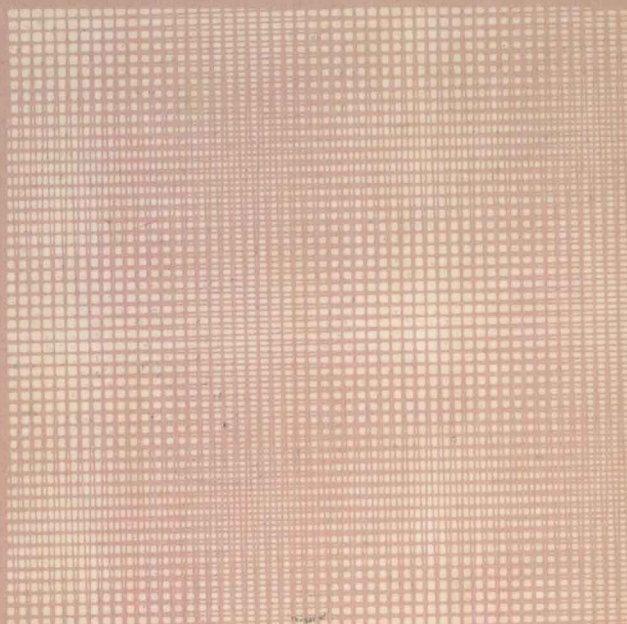


Volume 10

International Advances in  
**NONDESTRUCTIVE  
TESTING**



Edited by Warren J. McGonnagle

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The International Advances in Nondestructive Testing series publishes original research, development and application papers relating material properties to measurable physical phenomena. Special emphasis is given to new methods and techniques resulting from theoretical and laboratory investigations which may as yet have little or no industrial application. Also, papers dealing with the application of nondestructive testing methods and techniques to industrial problems and materials are published. The quantitative aspects of nondestructive measurements will be emphasized.

The series will also publish shorter papers dealing with subjects of general interest to all workers in the field of nondestructive testing, surveys reviewing developments in science and technology as they relate to nondestructive testing, and technical notes concerned with experimentation or instrumentation.

We invite our colleagues to publish in *INTERNATIONAL ADVANCES IN NON-DESTRUCTIVE TESTING*, suggest ways of improving the series, and communicate with us freely concerning its editorial policy.

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THE ACOUSTIC EMISSION RESPONSE  
of Ti6AL4V  
IN METHANOLIC MEDIA

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**Abstract:** The stress corrosion cracking (SCC) of Ti6AL4V in a methanolic/HCL solution was monitored with acoustic emission. Tests were performed on flat-plate, single edge-notched (SEN) samples under tensile loading in air and in the stress corrosion environment. A clear distinction in emission behavior was observed for the appropriate conditional states. Primary incubation periods were noted for the SCC samples. Data for the various tests is also presented in cumulative counts versus stress intensity factor.

**Introduction:** The use of acoustic emission (AE) as a non-destructive inspection technique has been under continuous development for over twenty years. The technique involves detection of elastic energy which is spontaneously released when a material undergoes specific changes. AE measurements are especially useful in studying the kinetics of complex fracture processes discontinuous in nature, such as stress corrosion cracking and hydrogen embrittlement. It is established that single events of discontinuous crack propagation can be localized in time and the relative amplitudes of the crack steps evaluated. Thus, acoustic emission constitutes a unique nondestructive inspection method in that the material during dynamic response, transmits its own signal, with the sensor serving as the receiver. For example, a material undergoing crack growth both generates and transmits the AE signal which can then be detected by suitable instrumentation.

The purpose of this investigation was to demonstrate the suitability of acoustic emission monitoring for the detec-

tion and characterization of stress corrosion cracking in a widely used titanium alloy-Ti6AL4V. The potential that AE offers in terms of correlating emission characteristics to load level, solution concentration, and periods of crack growth in materials during SCC may soon be realized. For this test program, samples were placed in two conditional states: (1) tensile loading with stress the main driving force, and (2) a combination of conditions which included tensile loading and the presence of a mildly corrosive solution. In addition, this work attempted to develop relationships between the AE response characteristics, the metallurgical state of the samples, and the mechanisms interactive in failure. Various notch sizes were used for experiments representing both conditional states.

From the standpoint of material studies, when a stress pulse is generated, the disturbance travels through the material as an elastic stress wave. The stress wave may be detected by a transducer at the material surface, which is sensitive to frequencies higher than those encountered at the background noise level. In order to further emphasize the separation of background noise from the desired acoustic emission, often a threshold limit is set such that a counter registers one count every time the signal exceeds the threshold. The obtained signals are electronically processed and converted to analog form to be displayed as a plot versus time or other variables. Such a display denotes the level of acoustic emission activity.

When a stressed material contains even a submicroscopic flaw, the zone of the stress concentration rises in the vicinity of the flaw. If the stress being applied is continuous in nature, these submicroscopic flaws will eventually coalesce to form bigger cracks. During the process of crack initiation and propagation two forms of energy are then released: (1) low level energy over a long period, generated by the creation of a plastic zone at the crack tip, and (2) high level bursts of energy of relatively short duration which result from fracture occurring in the plastic zone (2). Other acoustic emission sources, previously observed during corrosion and stress corrosion cracking, would include gas evolution from anodic/cathodic surfaces, oxide film breakdown, and chemical dissolution (3,4).

Potentially, one of the more important observations derived from acoustic emission technology is that there exists a strong relationship between the emission characteristics and the stress intensity factor. In an attempt to further detail this relationship, this study was designed to identify how the stress intensity value ( $K$ ) was affected by stress corrosion cracking. AE was the continuous monitoring method used to explore further the relationship herein described.

SCC of alpha-beta titanium alloys in methanolic environments has been subject of scant study in the past decade, so that unequivocally established theories concerning this topic do not yet exist. However, certain phenomenological features have already been established (5). The fracture mode, for example, has been found to be transgranular in nature for Ti6AL4V exposed to methanol. The difficulty in establishing the fracture behavior in a given material arises from the fact that the transitions in fracture mode occur depending upon a number of variables. These may include: stress level, heat treatment, corrosive media, testing method, and temperature. Another interesting characteristic of this alloy is the occurrence of transgranular fracture in the presence of halide ions.

**Experimental Procedure:** Although there does not yet exist a test standard for stress corrosion cracking, those conditions normally used for fracture toughness testing are considered to be applicable. For this testing, rectangular specimens, cut from sheet material, were single edge notched to one of three notch sizes: 0.062, 0.124, and 0.186 inches. The geometrical configurations used for the specimens were selected on an arbitrary basis and were not intended to fulfill the specimen size requirements suggested ASTM. Because the sheet material was not available in sufficient thickness, the calculated stress intensity values are given in terms of plane stress.

After machining, the samples were heat treated at 950°C for one hour in argon and furnace cooled. This procedure normally provides an equiaxed alpha-beta structure. A light purplish-colored oxide ( $TiO$ ) was also obtained.

The mildly corrosive solution employed was methanol with

HCl controlled to a pH of  $1.7 \pm 0.2$ . An environmental chamber was built for the SCC tests that maintained the specimen's notch submerged in the liquid environment. A tensile machine was used to load the specimens, and to obtain an accurate constant load plot. The loading rate used for all tests was 0.02 inches per minute.

Commercially-available instrumentation was used to monitor the AE activity in the appropriate test conditions. The equipment included a differential piezoelectric transducer having a frequency response in the range of 0.2-0.4 MHz. The transducer preamplifier and amplifier had a combined range of 80 dB and a band width of 100-300 KHZ. The primary AE parameter measured was total counts. After testing, metallographic and fractographic examinations were conducted.

Results: The calculated stress intensity values for the three notch sizes are plotted in Figure 1. These data also establish a critical stress intensity band for SCC ( $K_{SCC}$ ) for this alloy. As noted, variations in test duration and plane stress conditions have caused deviation from a fixed  $K_{SCC}$  value. Components loaded to K-values at or above  $K_{SCC}$  would conceivably show crack growth to failure. Figures 2-5 correlate the cumulative counts during tensile and/or SCC testing for two sets of samples. These figures demonstrate that most of the emission activity shown originated from pre-yield loading. Perhaps also, as expected during the initial stress relaxation period or constant load, only a minimal amount of emission was recorded. Thus, for the air samples, the cumulative count remains fairly constant, even during a 24 hour monitoring period. This is in contrast to those samples tested under near constant load in the presence of the methanolic environment. As noted, varying count rates were observed for the corrosive-interactive phase of testing.

The relationship between cumulative counts (N) and the stress intensity value (K) can be seen in Figures 6 and 7. A clear indication of the amount of AE energy that can be expected for any of the three notch sizes is shown. The curves obtained can be approximated to a power relationship (6). A straight line relationship is shown for the SCC samples.

Independent of the notch size and the stress intensity

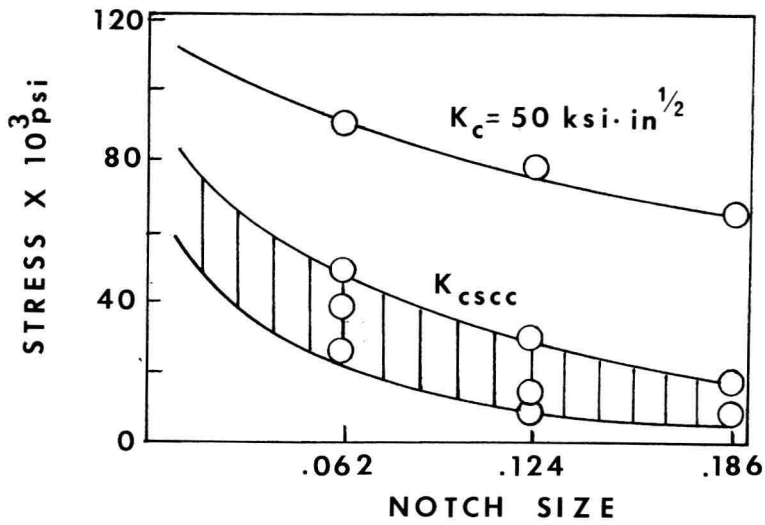


FIGURE 1. The stress intensity relationship for samples tested in air and methanolic media

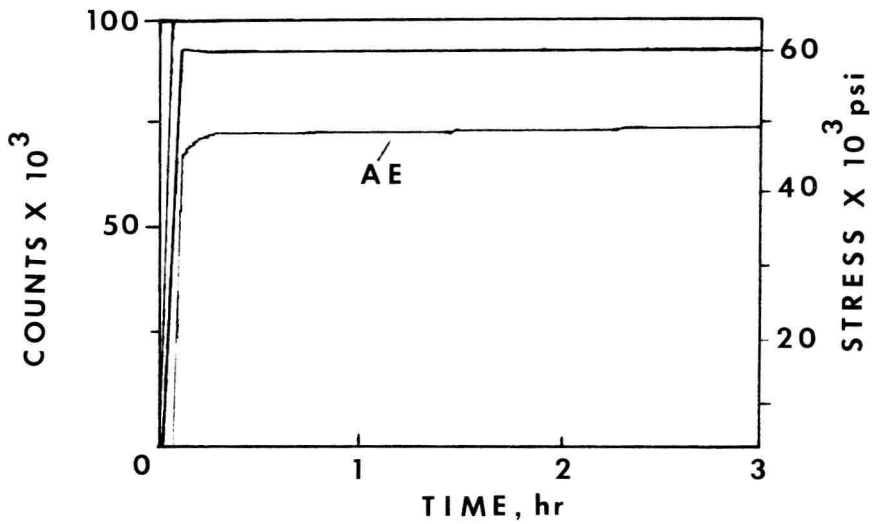


FIGURE 2. Applied stress and cumulative counts vs time for the 0.062 in. notch size

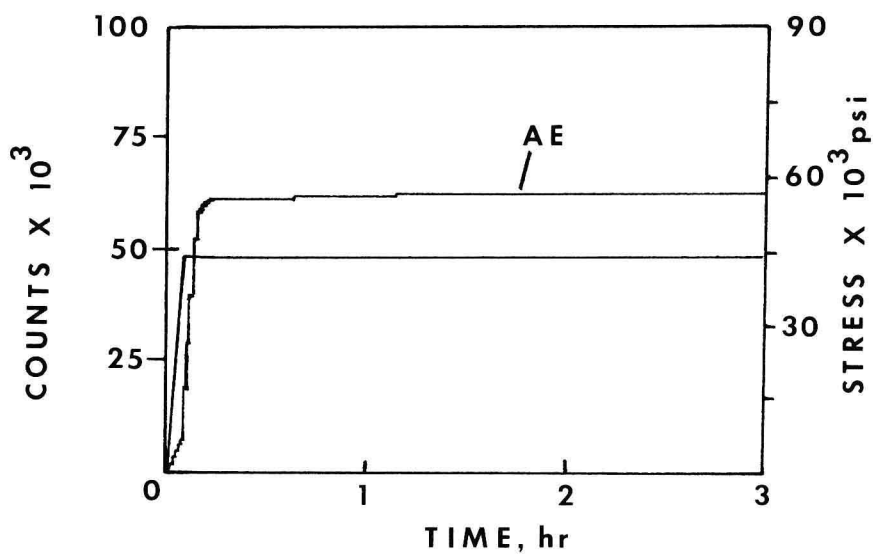


FIGURE 3. Applied stress and cumulative counts vs time for the 0.124 in. notch size

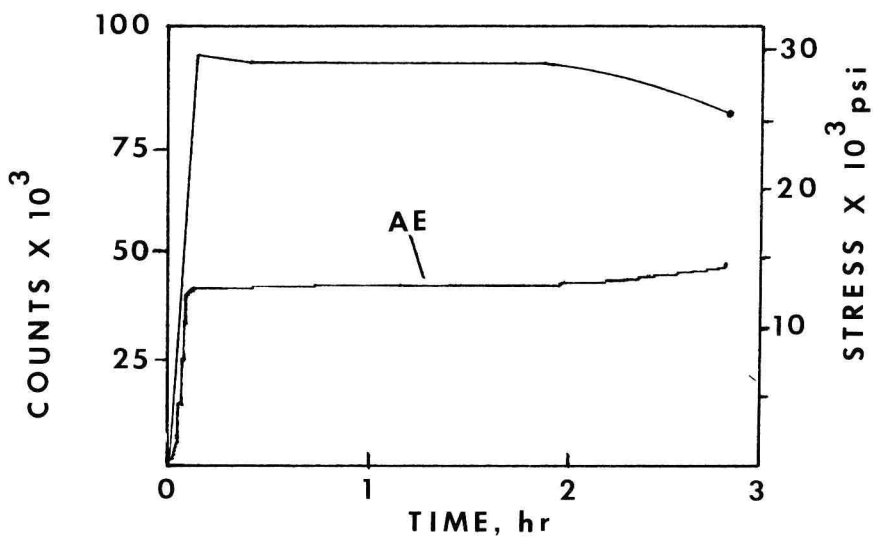


FIGURE 4. Applied stress and cumulative counts vs time for the 0.124 inch notch size in test solution

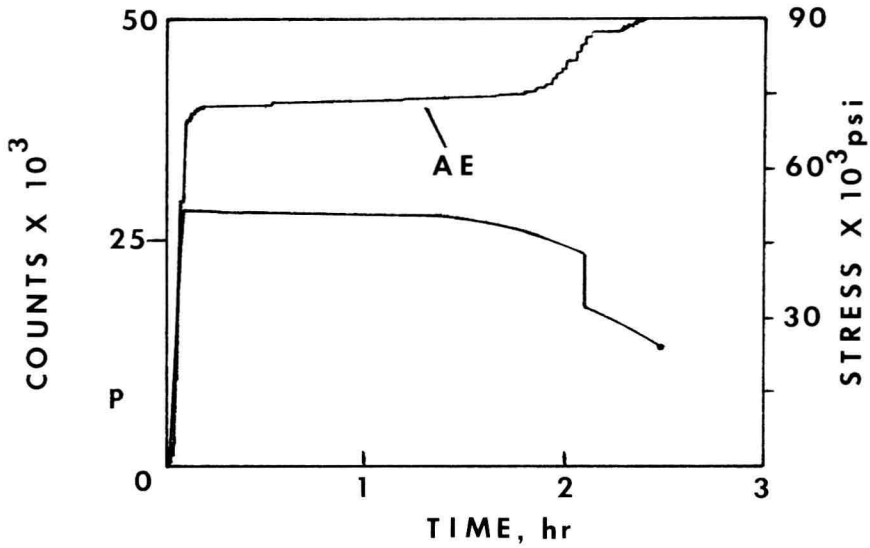


FIGURE 5. Applied stress and cumulative counts vs time for the 0.062 in. notch size in test solution

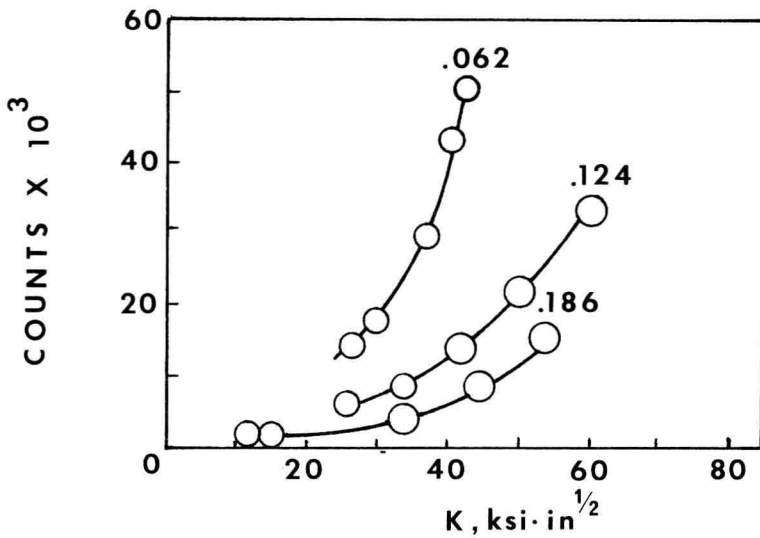


FIGURE 6. Total counts vs K for testing in air

factor, fracture occurred mostly in the transgranular mode through the alpha grains. Macro- and microfractographic analysis revealed that the air tested samples ruptured via ductile separation-dimple formation. Specimens subjected to a stress in the methanol/HCl solution were found to fracture by cleavage. The cleavage mechanism is believed to be the result of a complex interaction between the electrochemical and metallurgical states in the vicinity of the crack tip.

Discussion: There is presently no reliable, fundamental stress corrosion cracking theory for an alloy-environmental system which can be used to predict the performance of components even in environments where conditions are readily defined. However, it is well known that the presence of a corrosive chemical can greatly accelerate crack growth. Crack growth under the combined influence of mechanical stress and chemical attack defines SCC. The main object of these experiments was to determine the role of AE in studying the SCC process.

In the AE analysis of Ti6AL4V several interesting observations were made. Firstly, there are distinct differences in the emission response of the two sets of samples-mechanical stress versus SCC. It was found, for example, that varying notch sizes will influence the number of counts generated from active AE sources inside the material up until the time of constant loading. Normally AE from flawed metals results from a plastic deformation and crack extension. Hence, emission can be observed from a stationary crack only under rising load conditions, and, due to the irreversibility of AE only at stress intensities higher than the crack has been subjected in the past (7). Those tests performed in air, under constant load conditions, should then demonstrate dormancy. As the applied stress was increased (not shown in figures), a crack would begin to propagate from the notch tip. Crack growth was due to the coalescence of plastically induced voids. The fracture surfaces confirmed this by exhibiting shear dimples. Although the continuation of these tests produced weak AE activity, the ultimate failure process was identified by an increasing stress wave count.

Ultimately, the plastic zone as well as crack extension, are controlled by the stress intensity factor. Then it would be



expected that AE from a flawed material would also be controlled by (K). A simple model relating AE and the applied stress intensity factor under rising load conditions was suggested by Dunegan et al (8). The relationship between total counts (N) and (K) is given by  $N=AK^m$ , where A and m are constants. Dunegan et al (8) offered an explanation for this power law where  $m=4$ . Their model assumed that the counts were proportional to the plastic zone volume at the crack tip. Thus, the AE counts of a notched specimen are given as a function of the volume of material that yields plastically. Although some observations of m are close to the above prediction of the plastic zone model, experimental results hardly support this model.

In the case of the specimens exposed to fracture in air, the m-values were 3.2, 2.0, 1.9, respectively for increasing notch size. For the SCC samples, the exponential relationship was not observed. A linear relationship is consistent with other subcritical crack growth studies (9). Materials demonstrating quasicleavage fracture also have an m-value approximately equal to unity (10). Perhaps it is possible to predict failure using AE if the critical stress intensity factor and the relationship between emission counts and the stress intensity factors are known. Hartbower et al (9) believe that crack size can also be calculated provided (K) can be determined at any load.

The criterion used in evaluating the stress corrosion cracking growth process was the primary incubation time as determined by AE. As observed in Figures 3 and 5, contrasting emission rates were identified. Emission commencing after constant load is quite gradual (count rate < one count/second) in contrast to the development of a more rapid emission (count rate > five counts/second). The primary incubation time was taken to be the time to the beginning of the rapidly increasing rate. The correlation of the primary incubation time vs.  $K_C$  is shown in Figure 8. The general relationship revealed is one of shorter incubation times for higher stress intensity values. This may be particularly noteworthy as it substantiates the synergistic relationship of stress and electrochemical interaction theorized for stress corrosion cracking phenomena. Perhaps then, under given conditions, the primary incubation time may be a predictable parameter with the use of