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Discrimination of Nuclear Grade Steel Samples Subjected to Different Heat Treatment Procedures Based on Acoustic Emission (AE) Data Profiling at Crack Initiation

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Abstract

The present work is an attempt to study crack initiation in nuclear grade, 9Cr-1Mo ferritic steel using AE as an online NDE tool. Laboratory experiments were conducted on 5 heat treated Compact Tension (CT) specimens made out of nuclear grade 9Cr-1Mo ferritic steel by subjecting them to cyclic tensile load. The CT Specimens were of 12.5 mm thickness. The Acoustic emission test system was setup to acquire the data continuously during the test by mounting AE sensor on one of the surfaces of the specimen. This was done to characterize AE data pertaining to crack initiation and then discriminate the samples in terms of their heat treatment processes based on AE data. The AE signatures at crack initiation could conclusively bring to fore the heat treatment distinction on a sample to sample basis in a qualitative sense. Thus, the results obtained through these investigations establish a step forward in utilizing AE technique as an on-line measurement tool for accurate detection and understanding of crack initiation and its profile in 9Cr-1Mo nuclear grade steel subjected to different processes of heat treatment.

Keywords: *Acoustic emission (AE), Crack initiation, Heat treatment*

1. Introduction

For the purpose of fail-safety of structures and components, an extensive study of material behavior under service loading is very essential. Further, the study of behavior of structures in the presence of cracks is also essential and is an interesting topic for experiments. It may be shown that different materials behave differently in the presence of cracks, depending upon mechanical properties, loading conditions and environment. The theoretical prediction of the behavioral pattern of a component may not comply when tested under simulated conditions of load and environment. There might be a large deviation between theoretical and experimental prediction. Hence, it must be ensured that each material used for fabrication of structural components is tested under simulated load conditions and environment at coupon level.

The present work is an attempt to study crack initiation in nuclear, grade 9Cr-1Mo ferritic steel using AE as an online NDE tool. The study was confined to the crack initiation profile pertaining to the pre cracking of the material.

The nuclear grade aspect is of particular importance, since the material is required to be exploited in nuclear plants where, maintenance is always difficult due to inherent radiation hazards. All the same, the material is expected to exhibit incomparable resilience to any expected/unexpected service condition deviations over the entire life cycle and should represent epitome of reliability. This is warranted due to radiation hazards and international norms of safety and standards.

2. Experimental Details

2.1 The Specimen

During the present work, crack initiation studies were done on five Compact Tension (CT) specimens of 12.5mm thickness and 60mm width, fabricated out of 9Cr-1Mo ferritic steel with a chevron notch. These specimens were bearing identification nos from 913 to 918.

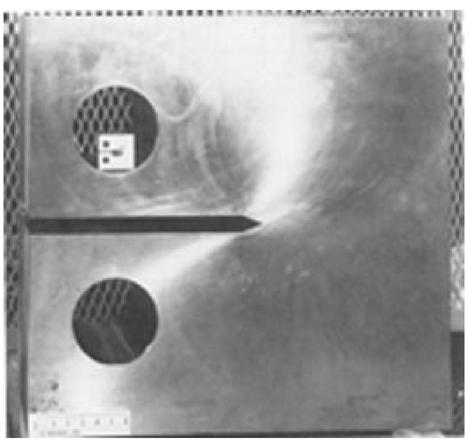

Fig. 1: The Specimen

2.2 Experimental Setup

Fatigue crack initiation tests were done on compact tension specimens in the laboratory. Tests were done on a ±100 kN load rating, ±50mm stroke, servo-hydraulic testing machine (Instron 1341).

Figure 2 shows the set up of the test fixture. The specimen was mounted on the machine using a clevis pin fixture. Initially, the machine was calibrated for load and stroke. Also, the machine was tuned to achieve up to 25 Hz frequency for a specified load sequence. The loading cycle was controlled via the software interface through a keyboard input through out the experiment.

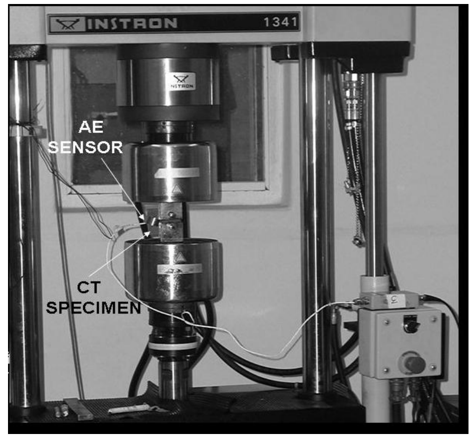

Fig. 2: Laboratory fatigue test set up

2.3 AE Data Acquisition

An acoustic emission system was set up with the sensor (Micro 30D Differential sensor operating in the band of 20-1200 KHz) mounted on the specimen surface using spring clips. The sensor was interfaced to an acoustic emission data acquisition system (Mistras 2001). The acoustic data was continuously captured and displayed on the monitor for online monitoring of the acoustic emissions.

The acoustic emission system included hardware, data acquisition controller and software with options for selecting sampling rate, AE parameters, gain offset as per application requirement. For ensuring that the data was reliably acquired, special care was taken to calibrate the AE sensor response and checked on dummy specimen so that all AE parameters were being reflected in the desired manner. Appropriate .INI files were written in to the AE data acquisition system and compatible. DTA files were played out as the acoustic emission data was logged in real-time continuously during the conduct of fatigue crack initiation.

The main shortcoming of the AE system is that, the sensor also picks up background noise along with emission due to crack initiation. Background noise mainly includes hydraulic noise in laboratory experiments done on servo-hydraulic testing machines. One had to carefully separate noise and the signal by setting a proper threshold value. The signal below the threshold would be neglected in AE data analysis. By a trial and error method, the threshold values were set between 54 to 58 db.

Along with the acoustic emission parameters, load output from machine console was also recorded by connecting the load channel output as one of the external inputs to the acoustic emission system. The data acquisition was started as soon as fatigue cycling on the specimen was started, and acoustic emission data were recorded continuously throughout the test. Various options for filtering data by inputting lower and upper limits were available in the post processing software of the acoustic emission system. This helped in separating emission corresponding to peak load and filtering signals corresponding to lower load levels.

2.4 Fatigue Test summary

5 Samples of 12.5 mm 9Cr-1Mo Ferritic steel were subjected to cyclic fatigue. All the samples were loaded on the Instron (1341) m/c one at a time after calibrating the m/c for the experiment. Each fatigue experiment was carried out after placing an AE sensor so as to capture and record acoustic emission data. The acoustic emission data in respect of each of the samples was later replayed and analyzed to study the crack initiation. In each of the sample, Liquid dye penetrant (red) was applied at the notch to facilitate visual observation of the crack initiation (identified by bubbling at the notch tip).

The summary of cyclic fatigue loading is given at Table1 as under. The loading conditions were kept identical with maximum load, P_{max} of 10 Kn and an average stress ratio of 0.14 being maintained through out the loading.

Table 1: Summary of fatigue tests

Sample	P_{max} (Kn)	Cumulative Fatigue load (Cycles)	Freq (Hz)
913	10	2.5 lac	25
915	10	5 lac	25
916	10	4.3 lac	23
918	10	2.5 lac	23
919	10	5.8 lac	25

3. AE Data Analysis and Results

3.1 AE Data Profiling

AE profile for crack initiation was categorically discriminated from the complete AE data profile in the play back mode. This was done primarily on the basis of the cumulative counts plot against the time scale (indirectly reflecting the fatigue cycles). Fig. 3 below clearly shows the localization of the crack initiation zone in one of the samples. This is repetitive in each of the samples, as in each one of them; it was possible to characterize the AE data as belonging to the crack initiation.

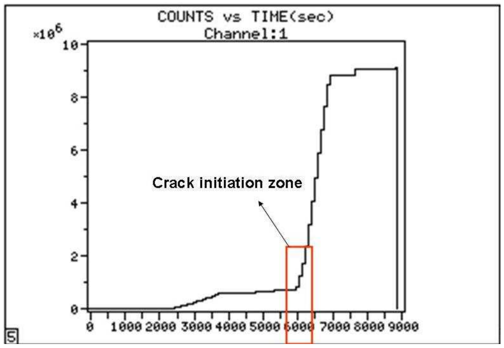

Fig. 3: Plot isolating crack initiation in 12.5 mm sample

Necessary filters were later executed on the AE files for bringing better clarity to the data and drawing conclusions to attribute the qualitative differences in the crack initiation profile to the differences in heat treatment. After studying the AE data files based on generated graph sets (based on post filter criteria), Crack initiation profile and trends were inferred. Comprehensive analysis of all the AE parameters was done to take cognizance of the significant changes in terms of Hits, Counts, Energy, Duration and Rise time. The interpretation of these changes was done based on cumulative, non cumulative and average values that have been recorded during the conduct of the experiment. Steep changes in slope or in trends over the complete recorded file were interpreted after considering the experimental observations. The aim was to generate appropriate graph sets that would effectively and absolutely associate the AE activity as being related to crack initiation. Further, consistency/repeatability of various parameters was essential for the accurate characterization of AE data. Based on these, time windows for each sample were estimated so as to apply a second string of filters which clearly brought out the crack initiation profile for each sample. The crack initiation profile for each sample was essential since they would clearly bring out the difference between each sample in terms of the Heat treatment. Each sample of 9Cr-1Mo ferritic steel of 12.5 mm thickness had undergone a different heat treatment and this was amplified adequately by the experiment.

3.2 AE Data Characterisation for Crack Initiation

The filtered data file that picked up the AE data after post processing from the time window pertaining to the crack initiation clearly brings out the characteristics of AE data associated with crack initiation in the material. The profile AE data for crack initiation is significantly different due to difference in the heat treatment even though the material composition is the same in the sample set. The AE data characterizing the crack initiation in each sample is tabulated in Table 2.

The AE activity identifies each sample in terms of avg counts, avg rise time, avg duration, avg energy and avg amplitude. The acoustic emission that characterizes crack initiation is unique to each sample because of the fact that each sample has undergone a different heat treatment that would have lend to the material a different hardness and microstructure variation at grain orientation level. Figs. 4 & 5 bring about these aspects in an explicit manner by categorically discriminating AE between one sample and another.

4. Conclusion and Discussion

The data in respect of the heat treatment is presented in table 3

The heat treatment data (Table 3) and the AE data/crack initiation data of samples do not have one on one correlation in terms of the hardness. It is not possible to establish any meaningful trend taking the avg AE parameters and the hardness no. as a reference to say that a particular hardness would exhibit a particular nature of AE activity that would uniquely identify with the hardness no. However, it is reasonably evident that there is a sample to sample distinction in terms of AE data at crack initiation. This distinction is primarily qualitative than quantitative. The qualitative discrimination of acoustic emission data seems to be intricately related to the process of the heat treatment rather than the avg hardness achieved. The reasoning for the same can be further extended with the knowledge that the heat treatment process alters the microstructure and grain orientations in the material. Therefore, the release of the acoustic energy at the time of crack initiation exhibits trends that mimic the associated microstructure.

The heat treatment profile clearly indicates three different heat treatment processes that these samples were subjected to. In fact, the AE data qualitatively discriminates these trends in all acoustic emission parameters at the time of crack initiation. While, sample 913 was subjected to furnace cooling, samples 915 & 916 were subjected to short-term aging in the order of 2 & 10 hrs respectively during the last step of the heat treatment. Similarly, samples 918 & 919 have been subjected to long term aging (200 & 500 hrs respectively) in the last step of heat treatment. Thus, it can be concluded that, at crack initiation in 12.5 mm 9 Cr-1Mo nuclear grade steel, the AE parameters manifest themselves qualitatively so as to discriminate different heat treatment procedures that the samples are subjected to. Also, the No. of fatigue cycles required to initiate a crack is discriminated. The same are objectively amplified in the Fig. 6. In terms of no. of cycles for fatigue crack initiation, there is a decreasing trend as the aging time decreases, for both long/short term aging. However, the AE parameters such as Avg Energy/Avg duration & Avg rise time show opposite trends of increase /decrease for short term and long term aging respectively.

Table 2: AE data characterizing crack initiation of 12.5 mm samples

	No. of cycles at which crack initiation occurred	Avg Duration (µs)	Avg Enrgy (counts)	Avg Amp (db)	Avg Counts (no.)	Avg Rise Time (µs)
913	18,750	2000	100	62	40	160
918	40,000	600	25	59	8	60
916	50,000	150	10	60	4	30
919	90,000	1200	40	62	14	200
915	39,000	300	15	61	14	110

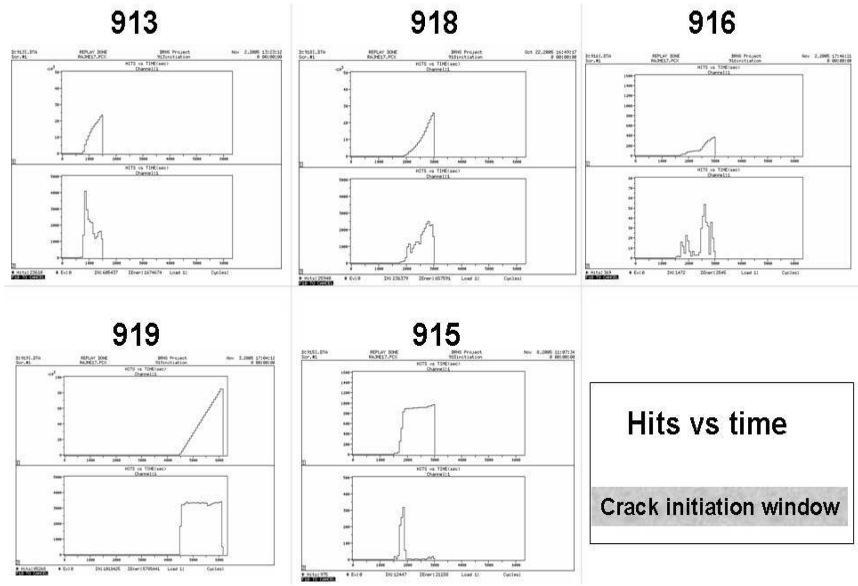

Fig. 4: Acoustic emission hits profile at crack initiation

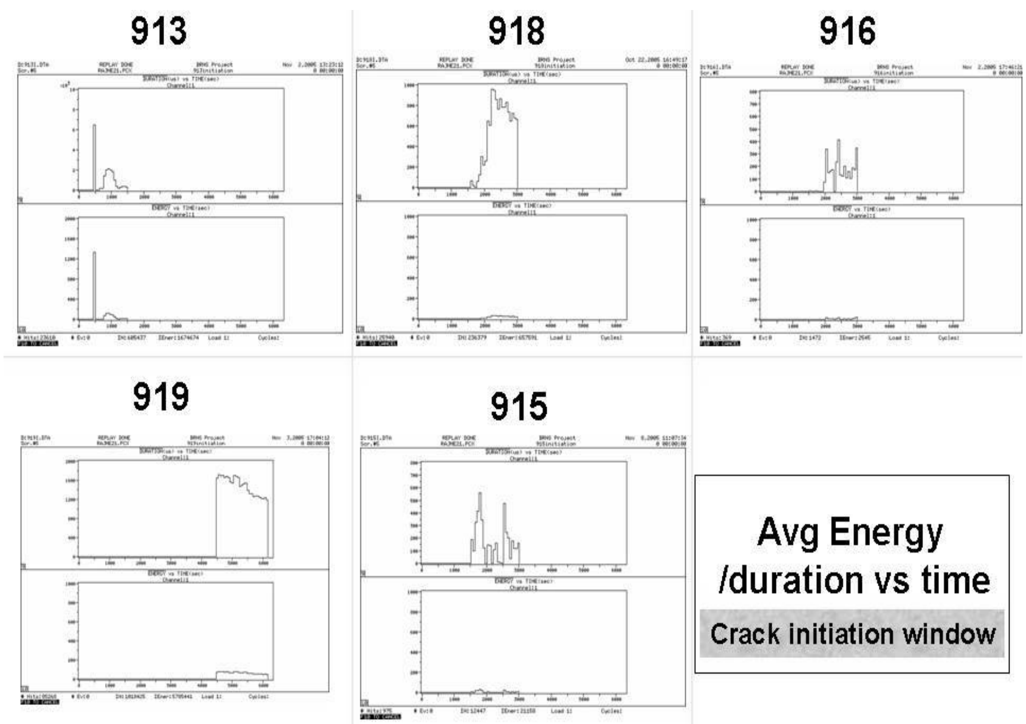

Fig. 5: Acoustic emission energy/duration profile at crack initiation

Table 3: Data in respect of the heat treatment

Sample (12.5 mm, 9 Cr-1 Mo ferritic steel)	Avg Vickers hardness taken at 10 Kg load	Heat treatment
# 913	213	1050^0 C/30 min, FC
# 915	185	1050^0 C/30 min, AC, then followed by 750^0 C/ 1 Hr, AC, then heat at 550^0 C for 2 hrs, AC
# 916	145	1050^0 C/30 min, AC, then followed by 750^0 C/ 1 Hr, AC, then heat at 550^0 C for 10 hrs, AC
# 918	228	1050^0 C/30 min, AC, then followed by 750^0 C/ 1 Hr, AC, then heat at 550^0 C for 200 hrs, AC
# 919	226	1050^0 C/30 min, AC, then followed by 750^0 C/ 1 Hr, AC, then heat at 550^0 C for 500 hrs, AC

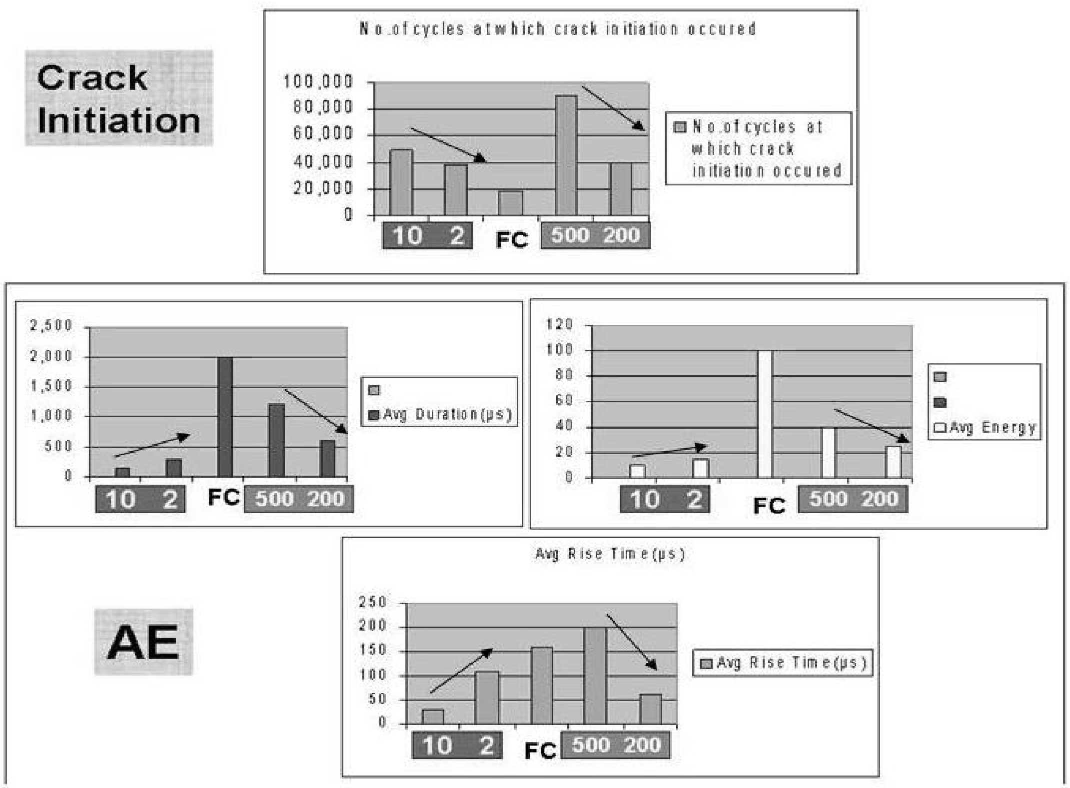

Fig. 6: AE parameters manifest themselves qualitatively so as to discriminate different heat treatment procedures

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