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Study of the oxidation behaviour of 9 Cr–1 Mo steel using the acoustic emission technique

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The onset of breakaway oxidation and *in situ* cracking of the oxide formed on 9 Cr–1 Mo steel in air at 900 and 950° C has been detected by acoustic emission (AE) technique. AE parameters, i.e. AE counts and AE event counts, show negligible increase during isothermal heating at these temperatures, until at a point, where a sudden increase in AE activity is found. This point corresponds to the onset of breakaway oxidation. An enormous increase in AE activity after the start of cooling has been attributed to the separation of scale from matrix as a result of thermal stresses arising during cooling. Peak amplitude distribution is measured and *b* values are calculated which help in distinguishing the phenomenon at 900 and 950° C in terms of the amplitudes of the AE emission released.

1. Introduction

Acoustic emission is a technique which is capable of detecting the transient elastic waves generated by rapid release of energy due to dynamic structural alterations in the material. The structural alterations can be of the type of microstructural change, plastic deformation, cracking of material or of the surface film and many others. The transient elastic waves thus generated are detected by a piezoelectric transducer and analysed.

Detection of cracks in the surface films or in the oxide layers formed during high-temperature oxidation is the recent field where AE technique is finding wide application. Studies on the cracking and spalling of the oxide layer formed on niobium [1], titanium [2], zirconium [3], copper [4] and steels [5] have been carried out by several workers. In an earlier work [5, 6] we have successfully used this technique to detect the breakaway oxidation of oxide layers formed on $2\frac{1}{4}$ Cr–1 Mo steel by continuously heating the sample from ambient to the set temperature. In the present work, isothermal oxidation of 9 Cr–1 Mo steel samples have been carried out for AE monitoring to detect the breakaway oxidation. In addition, an important parameter known as the *b* parameter, calculated from the measurement of peak amplitude distribution of AE events, has been used to characterize and compare the AE signals released at 900 and 950° C.

2. Experimental techniques

The chemical composition of the steel used is given in Table I. Specimens of about 1 cm^2 were cut from a 1 mm thick strip, obtained after rolling 12 mm thick plate followed by annealing at 950° C for 2 h in vacuum. The specimens were polished up to 600 grit finish, degreased and finally washed in alcohol before use.

TABLE I Chemical composition of steel

Cr	Mo	Mn	Si	P	S	C	Nb	Fe
8.46	1.02	0.46	0.41	0.001	0.004	0.083	0.072	Bal

A 1 mm diameter platinum rod, half a metre in length was spot welded to the specimens at one end and at the other end to a stainless-steel plate to which a transducer was attached. The integral assembly of platinum rod and stainless-steel plate acted as an efficient transmitter of AE signal from the specimen hanging inside the furnace to the transducer positioned outside (Fig. 1). The platinum rod was surrounded by a glass sheath sealed to a lid which sits on a quartz chamber. The lid could be moved up and down as and when desired in order to position the specimen in the furnace. This helped in achieving isothermal heating during oxidation.

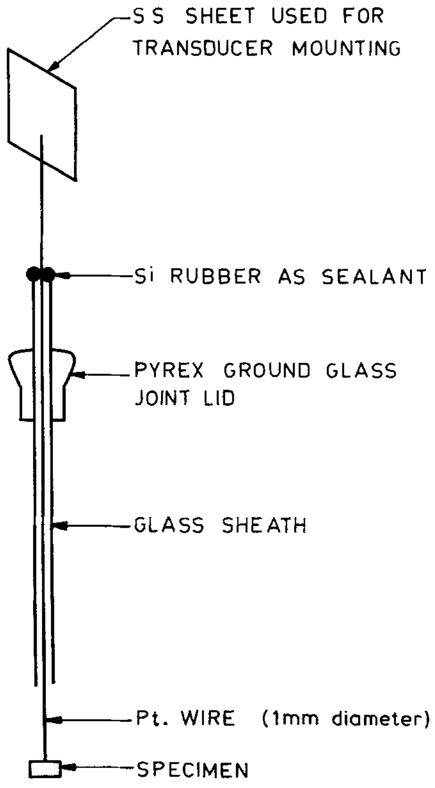

Figure 1 Schematic illustration of the wave-guide.

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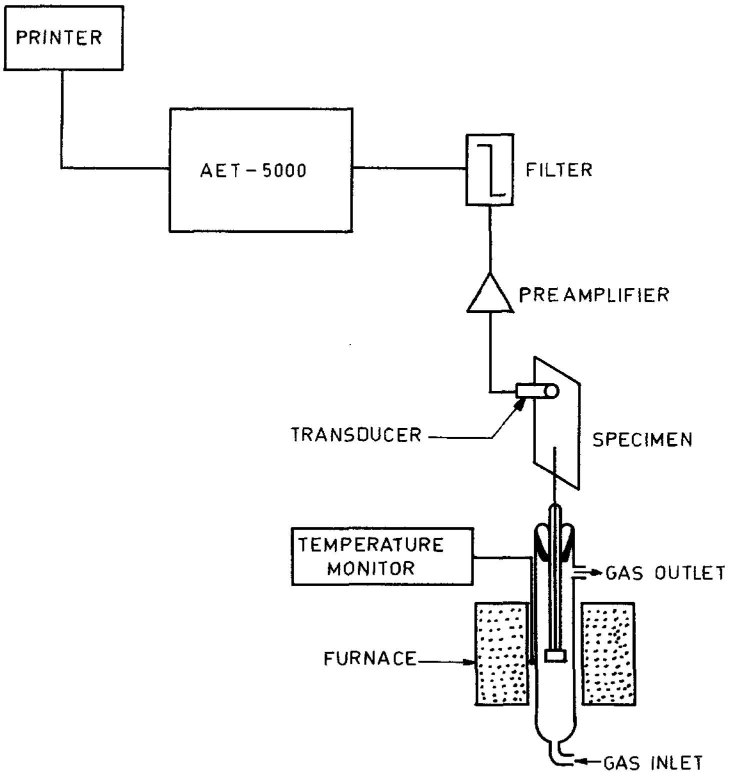

Figure 2 Schematic illustration of the experimental arrangement.

AE monitoring was carried out using an AET-5000 system supplied by Acoustic Emission Technology Corporation, USA. A piezoelectric transducer having a resonant frequency at 175 kHz was used together with band pass filter (125 to 250 kHz) and 60 dB fixed-gain amplifier. The total system gain of 100 dB and threshold voltage of 0.68 V was maintained throughout the test to eliminate the noise level. A schematic illustration of the experimental arrangement is shown in Fig. 2. Acoustic emission parameters such as AE event counts, AE counts and peak amplitude distributions were used for measuring AE activity.

3. Results

Fig. 3, gives the results obtained at 900° C. Both AE counts (Fig. 3a) and AE event counts (Fig. 3b) show negligible AE activity up to about 51 min exposure. At 51 min, a sudden increase in AE counts level and AE event counts level is noticed. Furthermore, an enormous increase in AE activity is seen at the end of 92 min when the furnace was switched off.

An alternative way to show the change in AE level at various points mentioned above is by plotting the cumulative AE activity, i.e. total cumulative AE counts (Fig. 3c) and total cumulative AE event counts

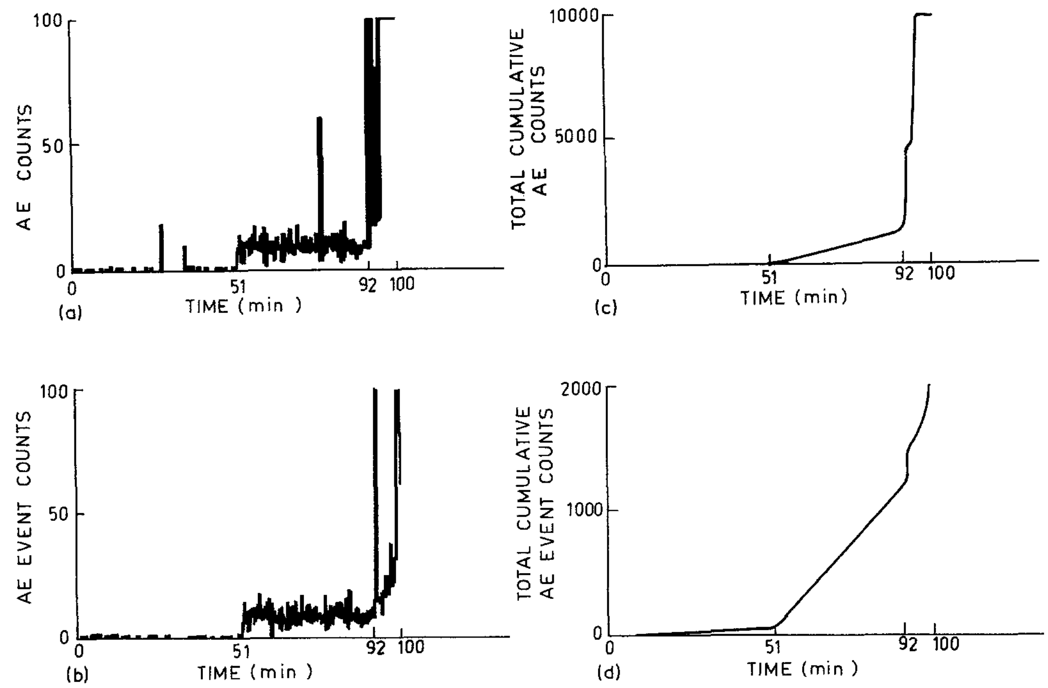

Figure 3 (a) AE counts; (b) AE event counts; (c) total cumulative AE counts, and (d) total cumulative AE counts plotted against time for 9 Cr–1 Mo steel oxidized in air at 900° C.

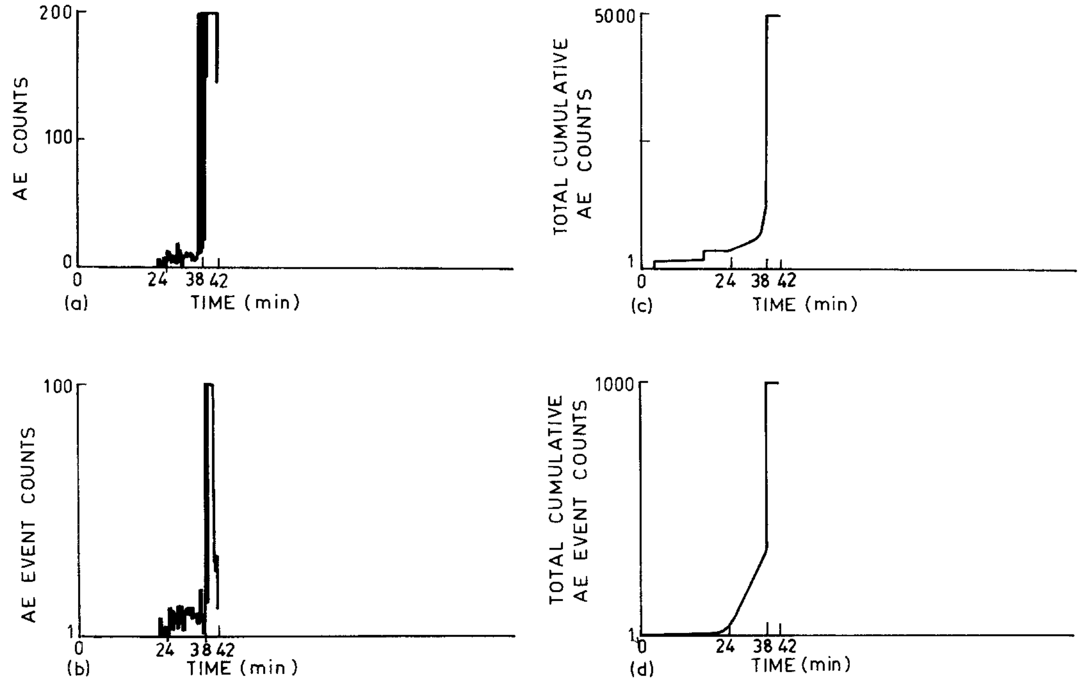

Figure 4 As Fig 3, but at 950° C.

(Fig. 3d) as a function of time. These plots very clearly show the change in the slope of curve at the beginning of the 51st minute (where AE activity was released during heating) and at the beginning of the 92nd minute (when cooling started).

Similar results were obtained at 950° C shown in Fig. 4. There is, however, a difference in the timings of the spurt in AE activity released during heating as well as during cooling, i.e. at the beginning of the 24th minute during heating and at the beginning of the 38th minute when the furnace was switched off.

A further insight into the comparison of the extent of AE activity released at 900 and 950° C can be made from a plot of cumulative distribution AE events by peak amplitudes as given in Fig. 5. At first instant it can be seen that AE activity released at 950° C (Fig. 5a) is of a more severe nature than at 900° C. This has direct relation to the energetics of the phenomenon as will be discussed below.

4. Discussion

Oxidation of 9 Cr–1 Mo steel has been extensively studied in air [7]. The steel undergoes breakaway oxidation at 900 and 950° C in a very short time after reaching these temperatures, i.e. in 15 min after reaching 900° C and in 10 min after reaching 950° C. The experiments were carried out using a Mettler TAI thermobalance, where a fast heating rate of 25° C min^{-1} was used to reach the desired temperature. It is, therefore, expected that an oxidation test performed under isothermal conditions as carried out in the present studies, the time to the onset of breakaway will be more than that reported in the dynamic experiments.

The increase in AE activity during isothermal heating at the beginning of the 51st minute (900° C) and at the beginning of the 24th minute (950° C) is, therefore, due to the onset of breakaway oxidation. This has been further confirmed by terminating the experiment at these points and observing the microstructure using SEM. The observed microstructure (rosette appearance oxide growth [8]) shown in Figs 6a and b for 900 and 950° C, confirm that the steel has undergone breakaway oxidation.

When the transverse section of the scale was observed after mounting, it was found that the scale had become separated from the matrix, and in addition there were voids in between the inner and the outer scale (Figs 7a and b). Thus the enormous increase in AE activity during cooling must be due to this internal cracking or separation of the scale from the matrix. Because of the nature of the phenomenon

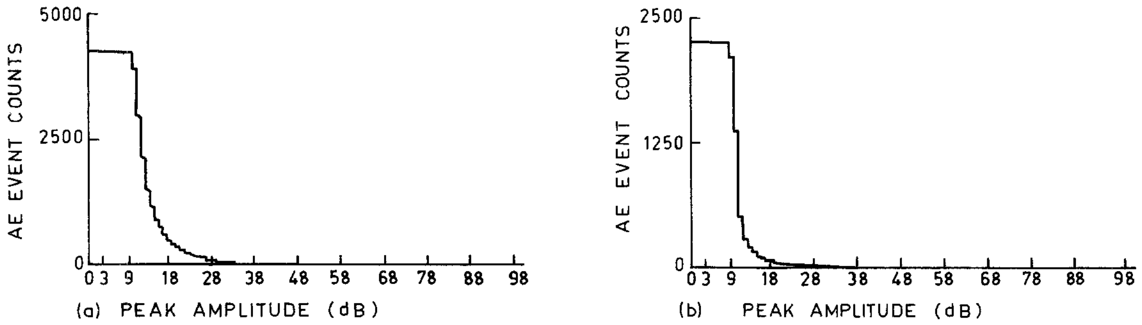

Figure 5 Cumulative distribution of events by peak amplitude at (a) 950° C, (b) 900° C.

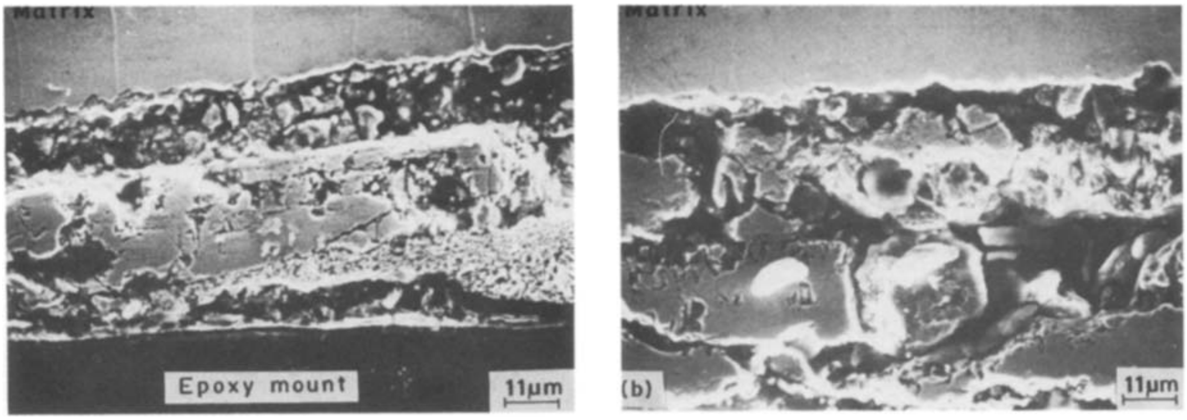

Figure 6 Surface morphology of the oxide scale formed on 9 Cr–1 Mo steel oxidized at (a) 900° C, (b) 950° C.

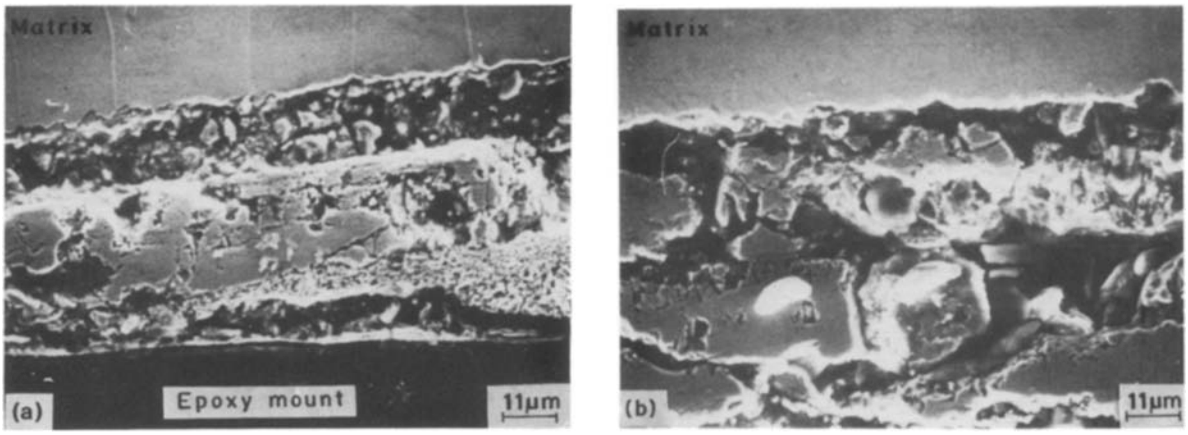

Figure 7 Cross-section of the oxide scale formed on 9 Cr–1 Mo steel oxidized at (a) 900° C, (b) 950° C, indicating cracks, voids and separation of scale from matrix.

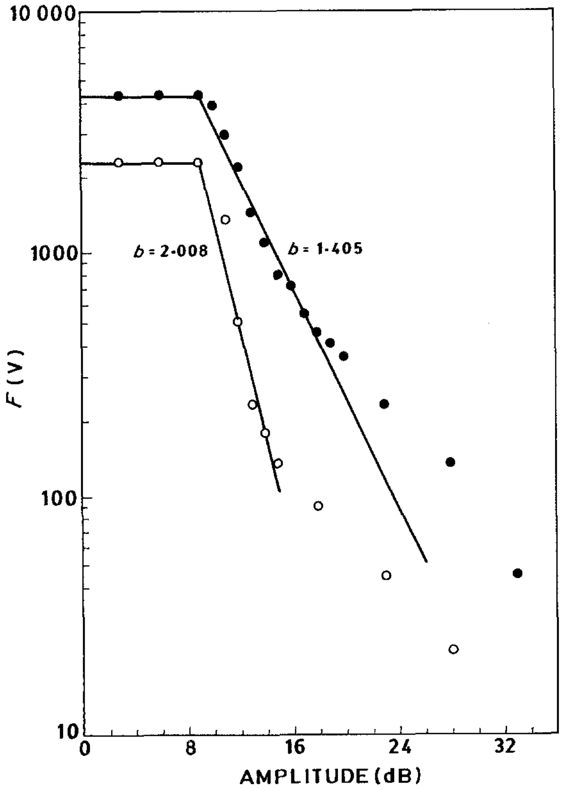

Figure 8 Logarithmic cumulative amplitude distributions for the experiments performed at (○) 900° C, (●) 950° C.

of separation from the matrix or internal cracking as shown in the micrographs, the process seems to be more energetic than the microcracking that is involved during the onset of breakaway oxidation during heating. This has also been indicated by the AE activity level for the two processes (Fig. 3).

Two phenomena, i.e. microcracking in the scale during heating and spalling during cooling, have been observed at these temperatures. The effect of temperature on these phenomena can be elucidated by a parameter known as the b parameter which is related to the peak amplitude distributions (Fig. 5) by the following relationship [9]

$$F(V) = \left(\frac{V}{V_0}\right)^{-b}$$

where $F(V)$ is the number of AE events having an amplitude more than a given amplitude value of V, and V_0 is the lowest detactable amplitude. The value of b can be determined from the slope of the plot of log $F(V)$ against log V/V_0 (Fig. 8). The value of b parameter at 900 and 950° C is 2.008 and 1.405, respectively. A large value of b represents a phenomenon dominated by low-energy emissions while a small value of b indicates the dominance of high-amplitude emissions [9, 10]. Thus the phenomena (microcracking during heating and internal cracking during cooling) at 950° C are more energetic than those at 900° C.

5. Conclusions

In the present study, an isothermal oxidation test and AE monitoring provided the basis of detecting the onset of breakaway oxidation and internal cracking of the oxide scale during cooling, on 9 Cr–1 Mo steels. The *b* value, derived from amplitude distributions, differentiates the phenomena at two temperatures, 900 and 950° C – a small value of *b* corresponds to more high-amplitude events and vice versa.

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