MICRO-CRACKING AND BREAKDOWN OF KAISER EFFECT IN ULTRA HIGH STRENGTH STEELS

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Abstract

This study investigates the progression of mechanical damage in ultra high strength steels during tensile testing using acoustic emission technique. We prepared samples from two steels, whose chemical composition are 1.4C-11Cr-0.8Mo-0.23V (JIS SKD11) and 0.8C-8.1Cr-1.9Mo-0.52V (modified SKD11), respectively. We heat-treated the samples to obtain peak secondary hardening. The mean and maximum diameter of primary M7C3 carbide inside the samples was 5.3 µm and 26 µm for SKD11, and 2.5 µm and 14.4 µm for modified SKD11. In SKD11 steel, we detected the AE events starting from 0.6 GPa having the amplitude of 23 - 40 dB. AE events were continuously detected as load increased. Above 1.2 GPa, we also detected many stronger AE events of 40 to 65 dB. In the second or later loading, we observed AE events at higher than 1.1 GPa, and AE persisted even when load was kept constant. Kaiser effect was not observed and AE typically started at 90% of the previous load after the second loading. In modified SKD11, the process of AE generation is similar to that in SKD11 steel. However, the AE initiation stress was higher and the amplitude of almost all the AE events was below 40 dB. The difference in amplitude of AE events between SKD11 and modified SKD11 reflects the size of carbide particles. These findings suggest that detected AE events were classified into two fracture types. One is, in lower stress ranges, AE signals caused by carbide cracking, as reported previously. The other is, in higher stress ranges, AE signals caused by micro-cracking, which propagated from fractured carbides. These micro-crack AE events are responsible for the breakdown of Kaiser effect when these steels are highly stressed. They are also indicative of slow crack growth as the loading approaches fracture criticality.

Keyword: Ultra high strength steel, Kaiser effect, Carbide cracking, Micro-cracks

1. Introduction

Cold-work tool steels form a class of ultra-high strength steels and derive their strength properties from high-carbon martensite with secondary hardening. Another attribute arises from the presence of primary carbide particles, imparting high wear resistance [1-3]. Previously, we showed that acoustic emission (AE) of two such steels (JIS SKD11 and its modification) originates mostly from the fracture of primary carbides and demonstrated by the use of laser-induced impulse method that observed AE signals correspond to the crack-opening mode of fracture in hour-glass tensile sample [4]. These fracture events also have fast rise time of ~1 µs. In connection to the fracture and fatigue strength of the steels examined, AE-start stress was substantially higher for a modified SKD11 steel, which had improved strength properties from the reduction of the average size of the primary carbides [5]. The peak amplitude of AE signals also indicated that the modified steel samples had a lower median value from smaller carbides.
The AE study verified its worth in characterizing the fracture initiation. It is possible that the subsequent progression of fracture and improvement of fatigue properties can be attained with better AE analysis.

In this study, we have extended AE analysis of the same two ultra-high strength steels. Because the modified SKD11 steels produced few or no AE with wide-band 40-dB preamplifiers, even at the stress levels of 1 to 2.5 GPa, we utilized narrow-band instrumentation and captured low-amplitude (20-40 dB) AE activities and analyzed them in detail. We have altered loading schemes and separated AE due to micro-cracking from those of carbide fracture. The breakdown of Kaiser effect was observed once micro-cracking appears to initiate. This points to the progression of fracture although the final fracture is not preceded by any precursor. Amplitude distribution analysis, load holding and brine exposure were also utilized in this study.

2. Materials and Experimental Procedures

A conventional tool steel (1.4C-11Cr-0.8Mo-0.23V, JIS SKD11) and its modification are used in this study. The modification reduced Cr and C content and increased Mo and V, resulting in the optimized composition of 0.8C-8.1Cr-1.9Mo-0.52V (modified SKD11). The chemical composition, heat treatment and other details are given in reference [4]. We heat-treated the samples to obtain peak secondary hardening effects (tempering at 773 K for SKD11 and 793 K for modified SKD11). The mean and maximum diameter of primary M2C3 carbide inside the samples was 5.3 µm and 26 µm for SKD11, and 2.5 µm and 14.4 µm for modified SKD11. The shape of specimens was hour-glass type. The specimen has the reduced section of 5-mm minimum diameter with the radius of 30 mm. The stress concentration factor is 1.406. It has shoulder flat on both side of the reduced section. Tensile tests utilized a floor-model Instron and the crosshead speed was 0.01”/min. We typically conducted the loading-unloading cyclic test. AE instrumentation is basically identical to that used in the previous study with AET MAC375L sensors, except the bandwidth was reduced to 250-500 kHz and 60-dB preamplifiers (AET 160) were used. This allowed us to set the threshold level at 23 dB (equivalent to 14 µV at the preamplifier input).

Two parallel data acquisition systems were used. Both are based on PAC Mistras boards, running Mi-LOC or Mi-TRA software. The latter was set to trigger from either of 2 channels synchronously. Since the sensitivity of the two sensors (AET MAC375L) was unmatched, Mi-LOC system recorded about ten times less events. This produced unintentional down-sampling and was useful in rapid data analysis when the hit counts were high. The main part of AE data was obtained from Mi-TRA recordings. All the recognized AE events were visually waveform-matched by viewing the waveforms in “group replay” mode and each set had had less than 5 µs arrival time difference. Signal features of only these events were extracted and subsequently analyzed. This is tedious, time-consuming practice, but produces highly reliable results.

3. Results and Discussion

Tensile and AE test results for modified SKD11 steel (tempered 773 K) are shown in Fig. 1. The sample was loaded to 2.75 GPa and held for 80 s. Two cumulative AE event count plots are given. One is from Mi-TRA (marked syn) and the other from Mi-LOC (ind). The load-displacement curve is essentially linear and no plastic yielding was recognized. AE events started at 1.2 GPa (the fifth event at 1.6 GPa) and the event rates continually increased until reaching the maximum stress.
of 2.75 GPa. The total AE event count was 3100 plus 700 counts during load hold. The waveform was essentially unchanged throughout the test from that reported previously [4]. In Fig. 1, the peak amplitude of independently triggered events is shown by squares. Initial events had a range of 30-35 dB, which broadened later to 25 to 43 dB. Only a few events were detected for similar tests when the threshold was set at 40 dB and after careful noise discrimination.

Fig. 1 AE test results for modified SKD11 steel during the first tensile loading.

Fig. 2 AE test results for SKD11 steel during the first tensile loading.
For SKD11 steel tempered at 793 K, tensile and AE test results are given in Fig. 2 for its first loading. AE events started at a much lower stress of 550 MPa (the fifth event at 680 MPa). About 300 AE events were detected before 1.2 GPa, when the first event was recorded in the m-SKD11 test shown in Fig. 1. The peak amplitude of the observed events, again shown by squares, had comparable range of 23 to 44 dB, as in Fig. 1. Initial events had the amplitude range slightly higher than that of later events. Load hold at 1.25 GPa generated a limited number of AE events. The next loading to the same stress started to generated AE at ~95% of the previous maximum stress; the number of AE events was low or 5% of the previous total. See Fig. 3. Several events were detected during the initial part of unloading.

Fig. 3 AE test results for the same SKD11 steel sample during the second tensile loading.

Fig. 4 AE test results for SKD11 steel during tensile testing with load holds.

Figure 4 shows the results of loading with intermittent load holds. Overall AE behavior in this test is comparable to Fig. 2 without load hold. It is shown, however, that AE during load hold be
gins at 1.1 GPa. Load hold AE continues beyond 10 min at 1.25 GPa. In this test, the first AE was recorded at 530 MPa. Figure 5 summarizes another series of cyclic-load testing with 40-dB threshold setting and 2.1 GPa maximum stress. Kaiser effect breakdown is shown initially with the Felicity ratio of 0.95, but it approaches unity by the fifth loading cycle. AE counts during each cycle are reduced 300-folds. Figure 6 shows similar test results, except during the fourth cycle, the sample was wrapped with cotton cloth saturated with 0.5% brine solution. The first AE was detected at 1.8 GPa or at the Felicity ratio of 0.85 and the total AE counts exhibited 4-fold increase over the previous cycle. This indicated hydrogen-induced cracking. Premature fracture occurred after 15 second load hold.

Fig. 5 Cyclic loading to 2.1 GPa, showing AE start stress and total AE events for each cycle.

Fig. 6 Cyclic loading results (to 2.1 GPa) showing AE start stress and total AE events for each cycle. For the fourth cycle, this sample was exposed to 0.5%-brine, reducing the Felicity ratio to 0.85.
The peak amplitude distribution of observed (and discriminated) AE events has been analyzed. All the results can best be modeled by Weibull-type distribution function as shown in Figs. 7-9. Only poor power-law approximation is possible because of systematic curvature exhibited. Here, Weibull-type distribution function for the cumulative event count is given by

\[ N_{\text{cumulative}} = A \exp (-B(V_p)^q), \]

where A and B are constants, \( V_p \) is the peak amplitude (in \( \mu V \)), and q is the Weibull coefficient. Figure 7 is the distribution plot for the entire event data of test shown in Fig. 4. Squares indicate observed data points, while triangles show a Weibull distribution; in this case, q = 0.48. Differential distribution curve is also plotted. Choice of the constant B affects q-values and so B is taken as unity in all the present analyses. These AE events were detected from an SKD11 sample at stress below 1.25 GPa with 20 dB threshold. Figure 8 shows the distribution plot for the AE event data of the same steel, but at stresses of 1 to 2.1 GPa with 40 dB threshold. Even when the low-amplitude data between 20 and 40 dB is absent, a similar Weibull distribution function with q = 0.395 can model the observed data. Figure 9 is a plot for AE when a sample was exposed to brine. The event data represents both during load increase and load hold. This distribution is almost identical to that in Fig. 8 with q = 0.38. These show that the peak amplitude distribution is essentially unchanged.

The Weibull distribution with q = 0.35 – 0.5 was used to describe burst emission data from high-strength low-alloy steels [6,7]. These emissions originated in the decohesion of MnS inclusions, flattened on planes parallel to the hot-rolling plane. The amplitude distribution was correlated to the inclusion size distribution, using a simplified model of source function response [6]. In the case of AE from carbide cracking, a similar modeling may be validated once detailed carbide size distribution is obtained. If micro-cracks in SKD11 steels have fractured carbides as their origin, the same distribution also is likely.

![Fig. 7 Peak amplitude distribution for the entire event data of test shown in Fig. 4. Differential data and calculated Weibull distribution curve are also shown.](image-url)
Fig. 8 Peak amplitude distribution for the event data of SKD11 test with 40 dB threshold stress of 1 to 2.1 GPa.

Fig. 9 Peak amplitude distribution of the fourth loading of SKD11 with brine exposure. This is expected to indicate the distribution of micro-cracking AE.
The results of this study demonstrate the presence of two types of AE. One type is observed starting at low stresses (0.5-0.7 GPa for SKD11 and 0.7 to 1.6 GPa for m-SKD11), having amplitude of 20 to 45 dB. This is consistent with the carbide fracture origin, as previously postulated [4]. The other type is detected during load hold above 1.1 GPa in SKD11 and at 1.8-2.1 GPa in m-SKD11, with higher amplitude events and the sensitivity to brine environment. These emissions also do not exhibit Kaiser effect. This appears to be originated from micro-cracking. Internal carbides cannot be affected by short-term brine exposure. In SKD11 steel, many emissions have 40-55 dB amplitude, as shown in Fig. 8. Very few emissions of such amplitude were found in m-SKD11. Thus, micro-crack size appears to be related to carbide size.

One aspect is puzzling. All the elasticity calculations predict larger energy release when a micro-crack or carbide fracture occurs at a higher stress [8]. No apparent correlation between the stress level and observed AE amplitude is evident. This needs to be explored further.

4. Conclusions

1. Carbide fracture and micro-cracking produce detectable AE in the ultra-high-strength steels examined.
2. Peak AE amplitude, however, is relatively low, in the range of 20-65 dB.
3. No significant distinction in signal waveforms exists. Weibull-type amplitude distribution was found for the two types of AE. However, the amplitude range for carbide fracture is 20-45 dB, while micro-crack AE ranges up to 65 dB.
4. Micro-crack AE is associated with the breakdown of Kaiser effect, load-hold AE, and sensitivity to brine exposure.

References

5) K. Fukaura, Y. Yokoyama, D. Yokoi, N. Tsujii and K. Ono (to be published).