Acoustic Emission and Microcracking during Aging of Fe-C-Ni Martensite*

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Synopsis

The aging of Fe-C-Ni martensite was studied using acoustic emission and optical microscopy. It was observed that the bursts of acoustic emission were very discrete. The cumulative counts of acoustic emission as a function of the logarithm of the aging time increased linearly. The slope of the previous curves decreased as the grain size of austenite, or the test temperature decreased. The pre-aged martensite greatly influenced the number and rate of the acoustic emission counts and the number of the microcracks in the specimen.

(Key words: aging of martensite, acoustic emission)

I. Introduction

Since the first report in 1923,¹⁾ a number of further investigations have been undertaken concerning microcracking of high carbon martensite. Two models have been proposed to explain the mechanisms responsible for the cracking. In the collision model, it is proposed that cracking occurs during the formation of martensite due to impingements between plates.²⁾ In the aging model two different mechanisms are proposed. First, it is suggested that the aging of martensite itself creates high stresses in individual plates causing cracking. Secondly, the martensite reaction in the retained austenite requires the deformation of the aged martensite and thus creates the microcracks in it.^{3,4)} During the aging of the martensite the volume of the specimen decreases⁵⁾ and acoustic emission occurs.^{6,7)} The reasons for the acoustic emission are unclear.

In this investigation the aging of Fe-C-Ni martensite was studied using an acoustic emission method and optical microscopy to consider the effects of grain size of austenite, aging temperature, carbon content of steels and pre-aged martensite.

II. Procedure

The acoustic emission tests were done using a Dunegan/Endewco commercial instrument 3 000 series, a sensor S140B, a 920 distribution analyser, a 921 amplitude module, 303 counter modules and a 1801 (100H) preamplifier with a 100 kHz~2 MHz bandwidth. The threshold was 36 dB (with reference to 1 μ V at sensor) which was the noise level of the instrument. The instruments were factory calibrated. To check the calibration a 2H PENTAL 0.3 mm dia. pencil lead was broken on the sensor using a special holder recommended by the European Working Group on Acoustic Emission. If the test gave 92 dB the instruments were in calibration. To insulate the sensor from environmental vibrations it was glued

onto the heavy piece of steel which was hung from a cord. The couplant on the sensor was thin mineral oil.

Distribution analyser dead time was 1 ms which was selected on the basis of some preliminary test.

The cumulative ringdown method was used. It is an observed fact that acoustic emission cumulative event amplitude distribution $F(V)$ follows the power law relationship

$$F(V) = F(V_0) \left(\frac{V}{V_0} \right)^{-b,8,9)}$$

where, V : the peak amplitude of an AE event

V_0 : the detection threshold

b : here called, Richter exponent.

In the instrument used a log-log plot of $F(V)$ comes on the screen.

The steels: I 0.44 % C, 23.8 % Ni; II 0.71 % C, 18 % Ni; and III 1.4 % C, 6 % Ni, 1 % Cr (wt%) were used as the test materials. The steels were melted and cast into the chill in the argon atmosphere. The ingots were hotforged to 12 mm thick plates, annealed and machined to 7 mm thickness.

The preparation and heat treatments have been presented elsewhere.^{4,5)} The size of the specimen was 7×7×4 mm³. The specimens were sealed in well-exhausted quartz ampules. The austenitizing temperatures were $T_r=950, 1\ 050, 1\ 150,$ and $1\ 250\ ^\circ\text{C}$, and the time was $t_r=3\ \text{h}$ for the steels I and II and $T_r=1\ 200\ ^\circ\text{C}$ and $t_r=10\ \text{h}$ for the steel III. The grain sizes of austenite were $d_r=0.05, 0.1, 0.6,$ and $1.2\ \text{mm}$ for the steel I and $d_r=0.025, 0.05, 0.1,$ and $0.2\ \text{mm}$ for the steel II corresponding to the austenitizing temperatures, respectively and for the steel III $d_r=2.7\ \text{mm}$. The specimens were quenched to room temperature and maintained there for four to six weeks. Before testing, the specimens were polished and inspected using a magnet to confirm the fully austenitic structure.

To study the effect of the grain size of austenite on the acoustic emission during the aging of martensite, the tests were done at the temperature $T=+15\ ^\circ\text{C}$ with the specimens of steels I and II with different grain size d_r . The specimens were first quenched into liquid nitrogen and then into alcohol whose temperature was $+17\ ^\circ\text{C}$. The specimens were strongly stirred in alcohol. The stirring time was 8 s in which the specimens reached the test temperature with an accuracy of 0 to $-3\ ^\circ\text{C}$. After heating to the test temperature the specimens were placed on the sensor

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with cotton gloves. The time for the heating and the placing was 10 s.

To study the effect of the test temperature on the acoustic emission during the aging of martensite the tests were done at temperatures $T = +15$, -17 , and -33 °C. The steel III specimens were first quenched into liquid nitrogen and then into alcohol whose temperature was 2 °C above the intended temperature. Otherwise the tests were as above.

To study the effects of the pre-aged martensite on the acoustic emission and the number of microcracks, different amounts of martensite were formed in the steel III specimens by quenching them to different pre-quenching temperatures T_{q1} . After quenching, the specimens were pre-aged by bringing them to room temperature $T = 15$ °C, holding there for $t = 17$ h and the martensite contents and the number of microcracks in the martensite were counted. After pre-quenching and pre-aging the specimens were quenched into liquid nitrogen of the temperature $T_{q2} = -196$ °C, then heated to room temperature $T = 15$ °C and placed on the sensor. The heating and the placing procedure was as above. After the acoustic emission test the martensite contents and the number of microcracks across the plates and the total number of microcracks in martensite were counted. The number of microcracks was counted using an optical microscope. The magnification used was $800\times$. The diameter of the field of vision of the microscope was measured using an objective micrometer. The field of vision of the microscope was moved stepwise around the specimens taking 20 views. The total number of microcracks observed in the consecutive fields was divided by their aggregate area to obtain the number of microcracks per mm^2 .³⁾ The specimens were polished using an automatic vibratory polishing machine and aluminium oxide slurry and then etched with nital.

III. Results

The cumulative counts of the acoustic emission created during aging of the martensite corresponding to different austenite grain size of the steel II as a function of the logarithm of aging time are shown in Fig. 1. The true recorder curves were like the broken line curve in Fig. 1. The curves were smoothed down so that the curves in Fig. 1 represent the average run of the true recorder curves. The bursts of emission were discrete so that with the instruments used it was not feasible to measure the rate of the bursts. The peak heights of the bursts fluctuated from a few counts to some exceptionally large bursts *ca.* 8×10^3 counts. The exceptionally large bursts brought about non-continuity into the count-curve. The number of bursts fluctuated in the repeated tests, being some tens when the total number of cumulative counts was low and a few hundreds when it was high. Any actual regularity was not seen in the number and peak height of the bursts. One can see that the decrease of the grain size of austenite decreases the rate of acoustic emission. With the grain size $d_f = 0.05$ mm the emission was very little and with $d_f = 0.025$ mm it was

Fig. 1. The cumulative counts corresponding to different austenite grain size of the steel II as a function of the logarithm of aging time.

Fig. 2. The cumulative counts created during the aging of steel III martensite *vs.* the logarithm of the aging time at the different aging temperatures.

totally lacking. The emission was also totally lacking with steel I.

The cumulative counts of the acoustic emission created during the aging of steel III martensite *vs.* the logarithm of the aging time at the different testing temperatures are shown in Fig. 2. The repetition of tests expressed a good deal of variation which is presented by brackets. As in Fig. 2 the general run of the curves straightened reasonably well in the logarithmic scale. One sees that the rate of count decreases as the test temperature decreases. When one of the specimens aged at $T = -33$ °C was further heated to the temperature $T = +15$ °C the enormous rate and number of counts were observed (the test is marked in Fig. 2 with a broken line).

The amplitude distribution in log-sum form for one of the characteristic $T = +15$ tests in Fig. 2 is shown

Fig. 3. The amplitude distribution in log-sum form for one of the characteristic $T = 15^\circ\text{C}$ tests in Fig. 2.

in Fig. 3. The curves 1~6 in Fig. 3 were taken at 3×10^1 , 6×10^1 , 1.2×10^2 , 9×10^2 , 3.7×10^3 , and 6×10^3 s, respectively. Richter's exponent in the large amplitudes b_a was about -2.1 and that in the small ones b_b was about -1.25 .

In Fig. 4 one has the cumulative counts after quenching steel III into liquid nitrogen T_{Q2} corresponding to the aging time points $t_{\text{aging}} = 1$ min denoted in Fig. 4 $C-C_{t=1}$ and $t_{\text{aging}} = 20$ min denoted in Fig. 4 $C-C_{t=20}$ of the cumulative count curves at the test temperature $T = 15^\circ\text{C}$ for pre-quenched and pre-aged specimen as a function of the pre-quenching temperature T_{Q1} .

In Fig. 5 one has the martensite contents $M_{T_{Q1}}$ and the number of microcracks in the martensite $N-M_{\text{tot } T_{Q1}}$ after the pre-quenching and the pre-aging; the martensite contents $M_{T_{Q2}}$, the number of microcracks across the plates $N-M_{\text{acr } T_{Q2}}$ and the total number of microcracks $N-M_{\text{tot } T_{Q2}}$ after the quenching into liquid nitrogen and the acoustic emission test, the results of which are in Fig. 4, as a function of the pre-quenching temperature T_{Q1} . The steel III was used for the tests. In Fig. 5 one has also a schematic picture of microcracks across a plate (arc) and the touch point of these ($\times$).

One can see in Fig. 4 that when T_{Q1} decreases, the cumulative counts for both points of the cumulative count curves $C-C_{t=1}$ and $C-C_{t=20}$ first increase and then decrease so that at $T_{Q1} = -55^\circ\text{C}$ the emission is quite small. In Fig. 5 the martensite content $M_{T_{Q1}}$ and the number of microcracks in it $N-M_{\text{tot } T_{Q1}}$ increase when T_{Q1} decreases. Martensite content $M_{T_{Q2}}$ and the number of microcracks across the plates $N-M_{\text{acr } T_{Q2}}$ have a maximum at $T_{Q1} = -60^\circ\text{C}$ and -44°C , respectively. The total number of microcracks increases quite evenly (notice $10 \times$ scale for $N-M_{\text{tot } T_{Q2}}$). One can see that the greatest number of microcracks

Fig. 4. The cumulative counts after quenching into liquid nitrogen T_{Q2} corresponding to the aging time points $t_{\text{aging}} = 1$ min denoted $C-C_{t=1}$, and $t_{\text{aging}} = 20$ min denoted $C-C_{t=20}$, of the cumulative counts curves at the test temperature $T = 15^\circ\text{C}$ for the pre-quenched and pre-aged specimen as a function of the pre-quenching temperature T_{Q1} .

Fig. 5. The martensite contents $M_{T_{Q1}}$ and the number of microcracks in the martensite $N-M_{\text{tot } T_{Q1}}$ after the pre-quenching and the pre-aging, the martensite contents $M_{T_{Q2}}$, the number of microcracks across the plates $N-M_{\text{acr } T_{Q2}}$ and the total number of microcracks $N-M_{\text{tot } T_{Q2}}$ after quenching into liquid nitrogen and the acoustic emission test, the results of which are in Fig. 4, as a function of the pre-quenching temperature T_{Q1} .

is in touch points ($\times$).

IV. Discussion

One can watch the curves in Figs. 1 and 2 and dilatometry curves in Fig. 6 and see that they all are

Fig. 6. Relative length change vs. time for 100 % martensite of different aging temperatures (T_a) for steel III (after Ref. 5)).

nearly straight. In both cases, Figs. 1, 2 and 6, the rate of change goes down with temperature. There are, however, two big differences between them. As one can see in Fig. 1 the rate of change goes down with the grain size of austenite or in other words with the lens size of martensite. We have not observed that this happens in the case of dilatometry curves. It seems that the rate of change also goes down with the carbon content in the case of the acoustic emission. There was no emission with steel I. This is on the contrary with the dilatometry results where the rate of change was the largest at low temperatures with the low carbon content steel.⁵⁾ However, in the case of acoustic emission one counts only those pulses which go over the threshold and so there can be emission also in the case of steel I but its amplitude is so small that it is under the threshold used.

The test, marked with a broken line in Fig. 2, gives an impression that in the low temperature aging, something happened which made the following high temperature aging very noisy. This low temperature aging effect may be one reason for the variations which can be seen in Fig. 2. A little difference in the heating makes a big difference in the emission.

In the specimen with aging temperature $T = +15^\circ\text{C}$ of Fig. 2 (which is the same as the specimen $T_{Q1} = 15^\circ\text{C}$ in Figs. 4 and 5) there were $ca. 8 \times 10^5$ microcracks after two weeks aging at $T = 15^\circ\text{C}$. This number is large compared with the total number of cumulative counts in Fig. 2. If the log-sum curves in Fig. 3 are examined, one can see that curves straighten and follow the direction of the tangent a , as the aging time increases. If one draws a line tangential to the curve 6 in Fig. 3 and continues it up to the $F(V)$ axis at 0-amplitude it cuts the axis at $ca. 6 \times 10^5$ events. This is a very good approximate value of the number of microcracks in the specimen. In spite of the fact that the drawing of the tangent of curves was some-

what indefinite it gave good approximate values of the number of microcracks also in the other test. In the above tangent construction, it was presupposed that the power law relation⁹⁾ extends as far as 0-amplitude.

The number of the bursts was a few hundreds in the test in Fig. 3. Because there were $ca. 8 \times 10^5$ microcracks in the specimen it seems that during one burst there was in action, coincident with each other, a large number of emission sources. Dilatometer curves are very even in Fig. 6 and it is natural that the primary cause, carbon diffusion, happens, when thinking of clustering, very evenly. It seems that the deformation requirements, created by the volume changes of microstructure when carbon atoms diffuse into a new order, accumulate and when an avalanche in some place begins it relieves stresses and thus continues through the test piece. The fact that there is a large number of emission sources in action, nearly simultaneously, means also that an interference can take place between vibrations and, in that case, the sensor observes the result as a single large event. That also means that the amplitudes are the result of interferences. Though the tangent construction above gave a good result it is not sure that the sensor can see the true fine structure of a burst.

The maximums in the curves in Fig. 4 mean that the rate of counting increased in the beginning of aging. As one can see in Fig. 5 after pre-quenching and pre-aging there was microcracked martensite in the specimen during the end-aging when the acoustic emission was counted. One can think that a reason for maximums is that this pre-aged martensite made the aging deformations more difficult and noisier than in the case of the without pre-aged martensite.

It is also possible that the microcracks in aged martensite grew during the end-aging. That the curves $C-C_{t=1}$ and $C-C_{t=20}$ go down when pre-quenching temperature T_{Q1} goes down is understandable so that there is less ageable fresh martensite with smaller and smaller lenses (see Fig. 5), so there are less and less deformations.

An interesting phenomenon is the existence of maximum in the number of microcracks across plates, $N-M_{acr T_{Q2}}$, curve in Fig. 5. The causes for that are unclear.

The total number of microcracks $N-M_{tot T_{Q2}}$ increases as the pre-quenching temperature T_{Q1} decreases; in other words, the pre-aged martensite in the specimens increases before the end-quenching. It agrees with previous observations^{3,6)} that the more there is aged martensite the larger number microcracks are created in it by the martensite transformation in the retained austenite.

V. Conclusions

The aging of Fe-C-Ni martensites was studied using acoustic emission and optical microscopy.

It was observed that:

- (i) Acoustic emission happened in very discrete bursts.
- (ii) The cumulative acoustic emission counts vs.

the Logarithm of the aging time curve was a straight line.

(iii) The slope of the previous curves decreased as the grain size of austenite or the test temperature decreased.

(iv) Pre-aged martensite in the specimen strongly influenced the number and rate of the acoustic emission counts and the number of the microcracks of the specimen.

It was deduced that:

(i) Local volume change of martensite may be the indirect cause for the acoustic emission.

(ii) There are large numbers of emission sources in action during a burst.

(iii) Though the tangent construction in the amplitude distribution curve gave a good approximate value of the number of microcracks in the specimen, it is not sure that the sensor can observe the true fine structure of a burst.

(iv) Martensite transformation in retained austenite creates large numbers of microcracks in old aged martensite.

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REFERENCES

- 1) H. S. Rawdon and S. Epstein: U.S. Bureau of Standards Science Paper, **452** (1923), No. 18, 373.
- 2) A. R. Marter and A. O. Bencoter: *Trans. ASM*, **61** (1968), 293.
- 3) T. Vilo and J. Pietikäinen: Proc. ICOMAT-1979, MIT, Mass., (1979), 721.
- 4) J. Pietikäinen: Proc. ICOMAT-1982, Les Éditions de Physique, Paris, (1982), 479.
- 5) M. Kajatkari *et al.*: Proc. ICOMAT-1982, Les Éditions de Physique, Paris, (1982), 461.
- 6) J. Pietikäinen: Proc. Int'l. Symp. in High Strength Martensitic Steels, CSVTS, Havirov, (1972).
- 7) M. M. Shea and D. J. Harvey: *Sci. Met.*, **16** (1982), 135.
- 8) B. Gutenberg and C. F. Richter: *Seismicity of Earth*, Princeton Univ. Press, Princeton, (1949).
- 9) A. A. Pollock: International Advances in Nondestructive Testings, XII, (1980), 215.