Indentation Loading Studies of Acoustic Emission from Temper and Hydrogen Embrittled A533B Steel

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Isothermal tempering at 500 °C (within the region rendering low alloy steels susceptible to reversible temper embrittlement) induced acoustic emission activity in A533B steel during indentation loading. Samples, when sectioned, were found to contain small (~10 μm long) MnS inclusions, some of which had debonded from the matrix material when they were near the indentations. Hydrogen charging prior to testing greatly enhanced the acoustic emission activity. It also resulted in the formation of small (~20 to 200 μm) microcracks in samples tempered at 500 °C. These microcracks, when examined by optical metallography, appear to have propagated along prior austenite grain boundaries, consistent with fractographic observations of temper embrittlement in other low alloy steels. Many were nucleated by MnS inclusion debonding and all were confined to within a few hundred micrometers of the sample surface and within two or three indenter diameters from the indent. It is proposed that trace impurities (P, As, Sb, Sn) diffuse during the 500 °C temper to both the MnS inclusion interfaces and the prior austenite grain boundaries, reducing local cohesive strength. The tensile field created by the indenter debonds inclusions to form crack nuclei. Moderate acoustic emission results. In the absence of hydrogen these void nuclei may grow but do not coalesce to form observable cracks. The prior austenite grain boundaries, which in contrast to the dispersed inclusions can provide continuous crack paths, are not sufficiently temper embrittled to fracture without the assistance of hydrogen at these stresses. Hydrogen charging induces a high hydrogen concentration in a surface layer of the sample. This reduces further the grain boundary cohesion, and cracks initiated at inclusions are able to propagate along continuous grain boundary paths, generating additional energetic acoustic emission signals. This process can continue after unloading the indenter due to hydrogen diffusion to the residual stress field.

1. INTRODUCTION

REVERSIBLE temper embrittlement is the phenomenon whereby a quenched and tempered low alloy steel, upon tempering in (or slowly cooling through) the temperature range 300 to 600 °C, suffers an upward shift in ductile to brittle transition temperature, a reduction in upper shelf toughness, and a transition of low temperature fracture mode from transgranular cleavage to intergranular cracking.1,2 The effects have been linked to the co-segregation, during tempering, of certain trace impurities (P, Sn, Sb, As) with alloying elements such as nickel. It is a potential concern in all low alloy steels that are tempered in the critical temperature range during either fabrication or service. Temper embrittlement can also enhance considerably the susceptibility of a steel to hydrogen embrittlement.

Acoustic emission is the name given to the elastic waves generated by certain types of deformation, fracture, and phase transformation processes in solids. The elastic waves are generated by sudden local rearrangement of the internal stress (or strain) field. The properties of the generated elastic wave field (e.g., amplitude and frequency spectrum) are controlled by the magnitude and dynamics of individual deformation or fracture events. In particular, it has been shown that for crack growth sources, crack increments of ~2 to 3 μm at average speeds of ~500 ms⁻¹ are required to generate detectable signals.3 In A533B in the quenched and 650 °C tempered state, it has been found that almost no detectable acoustic emission is associated with the propagation of ductile dimple fracture (some is detected from MnS debonding/cracking).4 In other low alloy steels, temper and hydrogen embrittlement has been found to generate extremely energetic acoustic emission signals indicative of many quasi-brittle microfractures prior to failure.5 It would thus seem that monitoring acoustic emission activity should be a very sensitive method of detecting the development of temper embrittlement in A533B.

In the majority of acoustic emission experiments, acoustic emission has been observed during simultaneous measurement of mechanical properties on standard tensile or crack growth geometry samples. The advantage of this is that the macroscopic stress state at the moment of emission is fairly well characterized in these samples, making it somewhat easier to interpret the emission data. However, because of the large volume of material undergoing deformation it is very difficult to correlate, using metallographic techniques, a particular acoustic emission with a particular source. A major step in understanding acoustic emission would be independent observation of the emission source. The development of an indentation method of acoustic emission testing5,9,30,31 in part allows us to do this. With this method, a plate or small sample is indented under controlled loading conditions, and the acoustic emission is monitored as a function of time. The region around the indent can then be serially sectioned and the emission generating processes deduced. The method has been used to study brittle fracture in high hardness (Rc > 40 to 50) tool steels,5,30 in indentation fatigue and stress corrosion cracking of aluminum alloys,7 during hardness testing of aluminum alloys,31 and glass.8,9,10
Table I. Chemical Composition of A533B Plate

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>P</th>
<th>S</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Cu</th>
<th>Sn</th>
<th>As</th>
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<tr>
<td>Wt Pct</td>
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<td>0.011</td>
<td>0.007</td>
<td>1.39</td>
<td>0.20</td>
<td>0.58</td>
<td>0.23</td>
<td>0.51</td>
<td>0.12</td>
<td>0.024</td>
<td>0.017</td>
</tr>
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</table>

In this study, we have used the indentation method of acoustic emission testing to investigate systematically the effect of both tempering at 500 °C and hydrogen charging on A533B pressure vessel steel.

II. EXPERIMENTAL PROCEDURE

A. Materials

A533B is a MnMoNi low alloy steel containing ~0.2 pct carbon; it is usually supplied as hot rolled plate. The material we have used was supplied by Lukens as 100 mm thick Class I nuclear grade plate in the normalized and stress relieved condition. Its composition is given in Table I.

Samples 7.2 × 2.2 × 0.9 cm were cut from the plate as shown in Figure 1. In a deliberate attempt to study coarse grained material, samples were austenitized one hour at 1100 °C and oil quenched resulting in a prior austenite grain size of ~20 μm. Two samples were retained for testing in the quenched state; the remainder were tempered for one hour at 650 °C. Of this remainder, two were tested in this tempered state while the rest were given isothermal tempering treatments in pairs at 500 °C to promote temper embrittlement. The samples were electropolished after heat treatment to remove scale and surface damage.

One of each pair of samples was given a hydrogen charging treatment just prior to testing. Hydrogen was introduced by cathodic precharging in 10 pct H₂SO₄ solution that was poisoned with 144 mg/l of arsenic (in the form of arsenic trioxide). Samples were each charged for 12 hours at a current density of 10 mA cm⁻².

B. Acoustic Emission Measurements

Acoustic emission activity was measured as a function of time during indentation loading using the experimental configuration shown in Figure 2. A “Vickers” pyramidal diamond indenter was loaded against the sample at a rate of 17 N s⁻¹ up to a preset maximum load of 1000 N. The resulting acoustic emission activity was detected with a model S140B Dunegan Endevco transducer* (resonant frequency

*Certain commercial equipment, instruments, or materials are identified in this paper in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

at ~140 kHz) was situated 2.5 cm from the indenter. The signal was then amplified 75 dB and bandpass filtered at 100 to 300 kHz, and the count rate was measured above a 20 μV threshold as an arbitrary, but standard, estimate of acoustic emission activity using a Todyne 7500 system.

We recognize the acoustic emission source is a rapid localized change of stress distribution caused, as determined here, by the propagation of a crack through an elastically stressed solid. The electrical signal parameter recorded is the number of times voltage exceeds a preset threshold value (predetermined by background noise). This count will be greatly influenced by the elastic properties of the solid, the source-receiver distance, the sample size and geometry, the precise transducer and couplant used, and by the background noise environment. Consequently, it is, at present, quite impossible to relate directly the count of an emission signal to physical properties of its source. However, by standardizing the factors that influence count, we have attempted here to use this easy to measure parameter to give an indication of the relative changes of emission sources of various heat-treated A533B samples.

To reduce background noise and radio frequency interference, the sample and loading device were enclosed in an aluminum Faraday cage. Additionally, as a check that no spurious signals were generated by the loading system,
acoustically inactive as-received A533B samples were tested. These generated no detectable signals, confirming the low acoustic emission response of this steel and the quietness of the loading system.

Following indentation, samples were serially sectioned and polished to reveal the effects of indentation and aid deduction of the emission generating sources.

III. RESULTS

A. Microstructure Characterization

The continuous cooling transformation (CCT) diagram for A533B steel is shown in Figure 3. The cooling path of one of the samples quenched in oil was measured using an imbedded thermocouple and is shown in superposition. The diagram shows that the bainite “nose” of the diagram is barely intersected so that a substantial amount of martensite is formed. The relatively high martensite transformation temperature (Mₜ) of ~390 °C could allow some autotempered carbides to be formed. The microstructure of the quenched condition is shown in Figure 4, and from the presence of small carbides in some laths it can be seen that an autotempered martensite/bainite microstructure had indeed formed.

Tempering for one hour at 650 °C resulted in carbide precipitation and transformation of martensite (Figure 5). Many of the carbides appeared to have nucleated at the prior austenite and ferrite subgrain boundaries. Scruby et al. have used convergent beam microdiffraction techniques to analyze the carbides in A533B composition steel formed during tempering at 650 °C. They found that the majority of the carbides were of the M₇C₃ type where M was predominantly Fe, Mn, and Mo. Short tempering times resulted in a Mn:Fe ratio of 0.15 ± 0.03 and a Mo:Fe ratio of 0.04. Longer tempering times resulted in the appearance of M₇C₃ carbides containing a higher Mo concentration that could render the steel more prone to temper embrittlement.

Additional tempering at 500 °C (Figure 6) had very little detectable effect upon microstructure of 650 °C tempered samples. The most noticeable effect appeared to be a slightly denser distribution of carbides at prior austenite and ferrite grain boundaries.

B. Acoustic Emission Measurements

Acoustic emission was monitored during indentation loading of samples of A533B which had been given temper and hydrogen embrittlement treatments. There were small variations from one indentation to another on the same sample, but there were clear trends as can be seen in the following typical results.

In the as-received condition, A533B emitted no detectable acoustic emission when loaded with the indenter to 1000 N. Oil quenching resulted in the generation of a few (~4) small burst emissions during loading (Figure 7(a)). Hydrogen charging greatly increased the acoustic emission activity during loading and caused generation of very large amplitude signals both while a constant 1000 N load was applied to the indenter (Figure 7(b)) and even after removal of the applied load (this we will discuss in more detail later).

Tempering at 650 °C for one hour affected the acoustic emission very little in uncharged material (Figure 8(a)).
Fig. 6 — Microstructures of samples tempered at 500 °C for (a) 8 h and (b) 20 days.

However, it significantly reduced the acoustic emission activity of the hydrogen charged state (Figure 8(b)). Additional tempering at 500 °C greatly increased the acoustic emission activity of uncharged samples (Figures 9(a), (c), and (e)) with the activity increasing with tempering time. Hydrogen charging tended to increase further this acoustic emission activity (Figures 9(b), (d), and (f)). The levels of acoustic emission activity in the hydrogen charged, 20 day tempered sample was comparable to that emitted by borosilicate glass under similar loading conditions.

Acoustic emission was also observed for some specimens during unloading from both charged and uncharged samples (Figure 10). In addition, it was observed that hydrogen containing samples continued to generate acoustic emission signals after unloading and this persisted for periods of several days at room temperature (Figure 11). The rate of emission decrease after load removal could be greatly accelerated by increasing the temperature of the sample while in a water bath; annealing for as short as one hour at 100 °C resulted in a disappearance of this acoustic emission activity.
approximately penny-shaped and were generally coplanar with the surface. Some were not coplanar and intersected the surface outside of the indentation.

It was observed that:
1. The microcracks were up to several hundred microns in length.
2. They were confined within a layer ~500 μm of the surface.
3. The microcracks were predominantly intergranular with isolated areas of cleavage (Figure 14).
4. Many of the microcracks had apparently nucleated at MnS inclusions (Figures 14 and 15).

A horizontal microcrack was sometimes present at the bottom of an indent. This is a region of compressive stress while the indenter was under load; it becomes tensile only upon unloading.10 We expect cracks at this location to have occurred during or after unloading. To examine this in more detail one sample was indented prior to hydrogen charging. It was sectioned and no visible cracking was found. It was then hydrogen charged and the cracks shown in Figure 16 were observed to form, presumably under the action of the residual stress field.

IV. DISCUSSION

We have observed the acoustic emission from A533B to show a great sensitivity to 500 °C tempering and to hydrogen charging. This must be due to changes induced in either the mechanisms of dislocation motion or fracture that operate to accommodate the deformation around the indenter. Tests of as-received A533B (which produces no detectable emissions) clearly showed that friction or other spurious noise sources were not present in these experiments.

A. Mechanisms of Acoustic Emission Generation

Acoustic emission in A533B steel has been found to arise at room temperature from both plastic deformation5,13 and a range of microfracture processes5,13,16 which include debonding of inclusions14 (and possibly carbide precipitates in pearlitic microstructures) as well as alternating shear fracture1,15 and cleavage. It is also known that the occurrence of these processes is dependent upon heat treatment.3,17 Published evidence indicates3 that in quenched and ~200 °C tempered A533B of low S content (0.0045 wt pct) containing few inclusions, signals during yielding are caused by the rapid motion of groups of dislocations whose integrated slip distance exceeds about 100 μm. At larger strains in this material, additional signals may be emitted by cleavage at ambient temperature. Higher temperature tempering (>600 °C) leads to a ductile room temperature fracture mode and a loss of emission. The ductile dimple fracture process itself (the internal necking of intervoid ligaments) does not produce detectable emission.3

Ono et al.18 have studied the effect of preferred orientation in A533B on acoustic emission, flow stress, and fracture. Their work shows that much greater acoustic emission activity is observed when tensile stresses are applied through the thickness of rolled plate material. This anisotropy in acoustic emission events is similar for both low
Fig. 9—Load and acoustic emission count rate vs time for temper embrittled (left column) and temper embrittled and hydrogen charged (right column) samples.
(0.005 wt pct) and high (0.021 wt pct) sulfur steels but is of greater magnitude in the high sulfur material. Such a scale factor suggests a direct correspondence between sulfur content (number of inclusions) and the number of acoustic emission events. It strongly suggests that MnS inclusion debonding (and possible fracture) is the main source of acoustic emission in A533B tempered above ~600 °C.

Fig. 10 — Load-unload cycle indicating load and AE count rate for sample in the quenched condition showing acoustic emission response on unloading.

Fig. 11 — Decay of acoustic emission rate with time after removal of load on indenter.

Fig. 12 — SEM micrograph of debonding of MnS inclusion due to indentation of uncharged, temper embrittled A533B.

Fig. 13 — Successive section views of an indentation showing system of approximately penny-shaped cracks (arrows). Material had been temper-embrittled for 10 days at 500 °C and was hydrogen-charged immediately prior to indentation.
There is also anisotropy in fracture properties (i.e., reduction of area and strain to fracture), that is related to the inclusion distribution and morphology. Plastic flow itself is more isotropic. Ono's results suggest that the predominant, burst-like acoustic emission, being anisotropic, is not linked with isotropic plastic flow, but inclusion debonding/cracking.

Other evidence supporting this view comes from the relatively small variation in hardness during tempering at 500 °C (Figure 17) while there is a continual increase in acoustic emission (Figure 9). Since hardness is limited by plasticity in the matrix, this invariance supports the view that acoustic emission in this case is not directly controlled by plastic properties of the matrix material. In addition, observations of debonding cracks at inclusions, shown in Figure 12, strongly suggest inclusion debonding as the origin of burst acoustic emission during indentation loading of temper embrittled materials, in agreement with Ono.¹⁸

Moreover, it appears to be promoted by the presence of oxides associated with the MnS inclusions.

From the above, the following picture emerges. During tempering at 500 °C in A533B, segregation of tramp elements to the prior austenite grain boundaries occurs, as established by Hasagawa et al.¹⁹ and Durey and Edwards.²⁰ Segregation reduces the cohesive strength of the prior austenite boundaries, but not enough to induce grain boundary fracture at room temperature. In uncharged material, microcracks form at the MnS inclusion interfaces but are ~10 microns in size and give only moderate levels of "burst" acoustic emission activity. These microcrack events are greater in temper embrittled samples and become progressively more numerous as aging continues. This could be accounted for if segregation to inclusion interfaces causes the cohesive strength of the inclusion interfaces to be lowered during aging. Very little study has been made of such embrittlement; attention is usually focused on the grain.
boundaries. But it may be consistent with observed reductions in upper shelf toughness by allowing void nucleation at smaller strains.

The question next arises as to the cause of the effects which hydrogen charging produces in the temper embrittled materials. Metallography (Figures 13 to 16) showed that in hydrogen charged material, shallow intergranular cracks are formed during loading, and that the inclusions can act as nuclei. Hydrogen primarily has two effects. First, it changes the source from small debonding microcracks in the temper embrittled material (<10 μm) to longer intergranular cracks (20 to 200 μm), thus producing larger emissions. Second, it tends to produce shallow or surface connected cracks. This latter effect is probably related to the enhanced concentration of hydrogen near the specimen surface due to limited time for diffusion before testing.

In the hydrogen charged samples, cracks from regions of low tensile stress around the indenter can act as local stress raisers. The synergistic interaction between hydrogen and segregated trap elements at prior austenite grain boundaries then results in brittle intergranular fracture as the local tensile stress exceeds the reduced grain boundary cohesive stress. These cracks are able to propagate considerable distances because of the relative weakness of grain boundary triple points as crack arresters.

B. Fracture and Indentation Mechanics

The acoustic emission produced by indentation in most structural materials will depend on the relative contributions made by plastic flow and fracture. Plastic flow usually dissipates mechanical energy at a lower rate than fracture because fracture is more localized in steels. If large AE signals are to be generated in a structural medium strength steel, a material is needed which, under indentation loading, fractures rather than flows plastically (e.g., glass). This implies that a high hardness is required to sustain sufficiently high stresses for fracture and that similarly the fracture toughness ought to be low. Lawn and Marshall have proposed the use of the ratio of hardness to toughness as an index of material "brittleness". Both of these can be determined from a single indentation by the indentation diameter and associated crack size. Such a ratio might also have merit as a predictive index of "acoustic emmissability" of structural materials. Previous work using indentation on steels hardened by various heat treatments indicated that increasing hardness favored AE but no systematic study of fracture toughness was attempted.

Indentations can produce highly concentrated stress fields which can initiate and propagate cracks. Crack initiation and propagation is in turn controlled by the stress field and material properties. The indenter shape is likewise important, e.g., a "sharp" indenter produces a more highly concentrated field which is generally more likely to produce fracture (see Lawn and Wilshaw for a review of geometrical conditions). Generally, a deformation zone surrounds the indenter contact area and deformation processes and inhomogeneities are generally responsible for nucleating cracks in structure metals. In glasses, preexisting flaws can also cause premature fracture. Much work has been done recently on the fracture mechanics aspects of this phenomenon. In particular, it has been found that residual stresses associated with the elastic/plastic deformation field play a vitally important role in propagating any crack nuclei.

From detailed fracture mechanics studies of such cracking, it can be shown that the extent of fracture can be used to determine fracture toughness. Notably, in glasses and ceramics there is one crack system which develops in a surface radial direction from the impression corners of the Vickers indentations which can be used to provide simple, quantitative information on this parameter. In our experiments such cracks are not as well developed as they apparently are in more brittle materials (i.e., ceramics), but we may nevertheless use the formulation to gain a crude estimate of $K_C$. The formulation for the Vickers indenter is

$$K_C = 0.016 \left( \frac{E}{H} \right)^{1/2} \left( \frac{P}{D^{3/2}} \right)$$

where $P$ is the indenter load, $D$ is the crack size (the radial length of the cracks measured from the impression center), $E$ is Young's modulus, and $H$ is the hardness. For Vickers indenters, $H = 3\sigma_y$, where $\sigma_y$ is the yield stress; we take $H = 2100$ MPa and $E = 2.0 \times 10^5$ MPa. Taking $P = 1000$ N and $D = 700 \mu$m (Figure 13) we calculate $K_C = 26.6$ MNm$^{3/2}$. This is a reasonable value for an embrittled steel.

In the present work, as described above, acoustic emission was produced by the nucleation of small inclusion debonding microcracks in temper embrittled materials and their larger scale extension along continuous embrittled grain boundary paths in hydrogen charged material. We turn now to this problem of crack nucleation in relation to indentation mechanics to examine why AE activity varied with time at 500 °C for material that was only temper embrittled.

Indenter shape determines the geometry of the stress/strain field in the presence of plastic deformation in a given material. Samuels and Mulhearn have shown that there are essentially two types of strain field produced by wedge-shaped indenters. Indenters with an included angle less than a critical value (between 100 to 140 deg., depending on the material) produce a cutting or V-shaped type of strain field; those with an included angle greater than this value produce an encapsulating tensile (or expanding cavity) type of strain field. Atkins and Tabor later determined that this critical angle is about 105 deg. for a mild steel. The Vickers indenter used here has an included angle of 138 deg. between faces so that we expect an expanding cavity type of strain field. In addition, Mulhearn demonstrated that the strain field is relatively independent of the indenter shape for radial distances greater than about one indenter diameter away from the indenter point. It is then approximately hemispherical in shape.

The stress field associated with this deformation field has been discussed by Evans. A spherically symmetric stress field can be associated with a pressurized, expanding spherical cavity, for which an analytical solution has been found by Hill. This expanding cavity model is used to approximate the stresses induced in a half-space by indentation. Experimental results and numerical calculations generally support such a model (except near the free surfaces, where all normal stresses must be zero). The stress state in the spherically deformed region is triaxial, which tends to promote fracture due to reduced plastic flow. The inclusion
debonding cracks were radial fractures, for which the tangential stresses are of most importance. In an ideal elastoplastic material (i.e., no work-hardening), the tangential stresses during loading from Hill’s solution are

\[ \sigma_t(r) = \sigma_y \left[ -2 \ln\left(\frac{r_p}{r}\right) + 1/3 \right] \quad (r \geq r_p) \quad [2] \]

where \( r_p \) is the plastic zone radius and \( \sigma_y = 3\sigma_t(r_p) \) is the uniaxial yield stress. The tangential elastic stress outside the plastic zone is tensile:

\[ \sigma_t(r) = \frac{1}{2} \sigma_y \left( \frac{r_p}{r} \right)^3 \quad (r \geq r_p) \quad [3] \]

Figure 18 shows the stress field of a loaded indentation and its variation in \( r \) in the approximation of the expanding cavity model. Here the plastic zone radius corresponds to the locus of maximum tensile stress \( \sigma_t(r_p) = \sigma_y / 3 \) and is given in Hill’s analysis to be

\[ r_p = r_i (E / (3(1 - \nu)\sigma_y))^{1/3} \quad [4] \]

where \( r_i \) is the radius of the indenter, \( E \) is Young’s modulus, and \( \nu \) is Poisson’s ratio.

For brittle inclusion interfaces, fracture can occur during loading in a region where a critical stress \( \sigma_c \) (the strength of the interface) is exceeded. As shown in Figure 18, this corresponds to the zone \( r_p \leq r \leq r_c \). We let \( \sigma_c / \sigma_y = \alpha \). (Note that \( \sigma_c \leq \sigma_t / 3 \) so that \( \alpha \) is limited to \( 0 \leq \alpha \leq 1/3 \).)

From Eq. [2] we have

\[ r'_c = r_p \exp(\alpha / 2 - 1/6) \quad [5] \]

and from Eq. [3] we have

\[ r_c = r_p (1/3 \alpha)^{1/3}, \quad \alpha \neq 0 \quad [6] \]

so that the fracture zone \( r_c - r'_c \) is defined by the ratio of the critical stress and the yield stress.

The increase in acoustic emission with temper embrittlement alone can be explained according to our above observations only by enhanced microcrack nucleation at inclusion interfaces, since no larger scale defects could be found. This can be related to indentation mechanisms in the following way. As discussed above (Eqs. [5] and [6]), there is a fracture zone from \( r'_c \) to \( r_c \) (Figure 18) surrounding the indentation which is determined by \( \alpha = \sigma_c / \sigma_y \), and the plastic zone size \( r_p \). At a given flow stress, this fracture zone increases in size with temper embrittlement due to the decrease in \( \sigma_c \), according to our interpretation, with impurity segregation to the inclusion interfaces. We can then expect a larger number of inclusions to be critically stressed, generating a larger number of AE events. Let \( p(r) \) be the probability that a crack nucleation site (inclusion interface) lies between concentric radii \( r \) and \( r + dr \). The total number of critically stressed sites (potential acoustic emission sources) will then be

\[ N_c = \int_{r_c}^{r'_c} p(r) dV(r) \quad [7] \]

where \( dV(r) \) is an increment of volume. For a uniform density of inclusions \( \rho \) we take \( p(r) = \rho \) and \( dV(r) = 2\pi r^2 dr \) (because we consider only the hemisphere) so that

\[ N_c = \rho \int_{r_c}^{r'_c} 2\pi r^2 dr = (2\pi/3) (r^3_c - r^3) \quad [8] \]

Combining [5], [6], and [8] we have the number of potential acoustic emission sources,

\[ N_c = (2\pi r_p^3 / 3) [(1/3 \alpha) - \exp((3\alpha - 1)/2)], \quad \alpha \neq 0 \quad [9] \]

and replacing \( r_p \) with \( r_i \) (from Eq. [4]) we have

\[ N_c = (\gamma / \sigma_y) [(1/3 \alpha) - \exp((3\alpha - 1)/2)], \quad \alpha \neq 0 \quad [10] \]

where \( \gamma = (2\pi r_i^3 E / (3(1 - \nu))) \). The magnitude of the second term in [10] varies from 0.606 to 1 so that for purposes of discussion

\[ N_c \approx \gamma \left[ \frac{1}{3 \sigma_c} - \frac{1}{\sigma_y} \right] \quad [11] \]

From Figure 17 it is seen that \( \sigma_c \) and \( \gamma \) do not vary appreciably during temper embrittlement. However, \( \sigma_c \) could decrease due to segregation of tramp elements to inclusion interfaces. Equation [11] would then predict the increase in AE events observed during temper embrittlement. This is actually an upper limit if the inclusion density is used since not all the inclusions will debond at \( \sigma_c \), due to unfavorable orientation, size, shape, or surface chemistry factors which we have not included. Additionally, Hill’s theory assumes no work hardening.

From metallography we estimate the inclusion density to be about \( 2 \times 10^{16} \) inclusions/m\(^3\). At 1000 N, \( r_i \approx 5 \times 10^{-4} \) m, and for this steel we take \( E = 2.0 \times 10^5 \) MPa, \( \nu = 0.28 \) and we take \( \sigma_y = 700 \) MPa. Butcher and Allen’s work\(^1\) suggests we let \( \sigma_y = 150 \) MPa. This gives a value of \( N_c \approx 1200 \) as the potential number of acoustic emission sources at the prescribed loads. Typically, we observe \( \approx 100 \) signals suggesting only \( \approx 10 \) pct of the inclusions debond, broadly in agreement with metallographic observations. The model qualitatively accounts for the increase in AE with temper embrittlement aging for uncharged specimens and focuses interest upon the factors controlling inclusion interface strength. It also highlights the possible value of the AE technique as a method of studying inclusion interface toughness and temper embrittlement.
Finally, while the present analysis is principally concerned with AE produced during loading, AE can also be produced during unloading, as we have noted (Figure 10). The driving force for the generation of this appears to be the residual tensile stresses formed upon unloading the plastically deformed indent. These stresses lead to the development of an enhanced hydrogen concentration at stress concentrations (cracks formed during loading). This lowers the local fracture toughness, and incremental crack propagation occurs generating acoustic emission. The kinetics of the crack growth will be controlled by the spatial extent of the residual stress field and the diffusion of hydrogen to crack tip stress concentrations.

V. CONCLUSIONS

The phenomenon of temper embrittlement in a pressure vessel steel—with and without the presence of hydrogen—has been studied using the indentation method of acoustic emission. The following conclusions can be drawn from this work:

1. Temper embrittlement at 500 °C causes an increased amount of AE activity that increases with tempering time.
2. This activity is greatly enhanced if temper embrittled specimens are also hydrogen charged.
3. The source of AE in the temper embrittled specimens is the debonding of ~10 pct of the inclusion interface, with microcracks (~10 microns in size) distributed radially about the indentation center.
4. The source of AE in the temper embrittled and hydrogen-charged specimens is largely intergranular cracking (up to 250 microns in size); such cracks in many cases are nucleated by the inclusions. These cracks are close to the sample surface, due to the enhanced surface hydrogen concentration.
5. Delayed microcracking can result from residual stress fields of indentations if samples contain hydrogen.

This technique appears useful for correlating microstructure and the AE signal. With further development it might even prove useful as a portable technique for on-site nondestructive evaluation of critical components, such as the heat-affected zone of a weld, where it could give an indication of the presence of local embrittlement.

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REFERENCES

31. S. Carpenter: 22nd Acoustic Emission Working Group Meeting, March 1981, unpublished work discussed the monitoring of AE in aluminum alloys during hardness indentation of the Rockwell B type. (Professor Steven Carpenter, Dept. of Physics, University of Denver, Denver, CO)