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ACOUSTIC EMISSION FROM HYDROGEN SATURATED TYPE 304L
STAINLESS STEEL

by

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INTRODUCTION

Effects of hydrogen on the deformation and fracture of austenitic stainless steels vary widely, depending upon the steel composition and treatment, hydrogen exposure, and loading conditions (1-5). The steels are divided into two groups based on ductility loss and fracture mode: a small ductility loss with no change in fracture mode, or a large ductility loss with a change from ductile rupture to transgranular cleavage (6). In addition, intergranular fractures are observed in sensitized stainless steel (7). Alloys such as Type 310, Type 316, and A286 stainless steels are in the first group and Type 304L, 21-6-9, and Tenelon* are in the second.

The areal fraction of transgranular cleavage that is observed following hydrogen exposure varies with test temperature, hydrogen concentration, and grain size. Microcracks form preferentially along coherent twin boundaries throughout the deformed metal following a 12-14% plastic strain (6). Microcracks along grain boundaries occur less frequently. The microcracks and consequent cleavage failure are related to the stability of the austenite and ϵ -phase as measured by stacking-fault energy, but the mechanism of nucleation and growth is not certain.

* Registered trademark of U.S. Steel Corporation

Acoustic emission is attributed to energy release within a material body by localized plastic deformation or failure processes (8). The elastic stress waves may come from slip band formation, mechanical twinning, martensite transformation, or crack propagation. Each of these processes has slightly different acoustic characteristics allowing for easy identification.

Acoustic emission was monitored during tensile tests of Type 304L austenitic stainless steel to explore the applicability of the technique to hydrogen-assisted fracture.

ACOUSTIC EMISSION

Acoustic Emission Tests

Smooth bar tensile specimens, 0.48 cm diameter by 2.5 cm gauge length were machined from bar stock of types 304L and 316 austenitic stainless steels (Table I). The 304L specimens initially had a grain size of $\sim 10 \mu\text{m}$; several specimens were annealed at 1270 or 1470 K for 24 hours to increase the grain size to ~ 100 and $400 \mu\text{m}$, respectively. All the 316 specimens tested had a grain size of $\sim 30 \mu\text{m}$. Tensile tests were conducted in air at 200-250 K on specimens exposed to hydrogen at 60 MPa pressure at 620 K for three to four weeks. This exposure yielded a hydrogen concentration in the alloys of about 200 moles/m^3 based on previous experiments (9). The test temperature is near the ductility minimum for hydrogen-charged Type 304L steel and where microcrack frequency is greatest.

Acoustic emission was monitored with an Acoustic Emission Technology Corporation model 204A system. The PZT sensor with a 175 KHZ resonant frequency was attached to the upper grip with a constant force clamp and silicone grease couplant. The amplified and processed signal was monitored directly on an oscilloscope. Simultaneously the instrument presented the instantaneous RMS value of the signal including background noise and an analog voltage proportional to the event, or event rate of acoustic emission. This physical arrangement (Figure 1) reduced the sensitivity of the technique because the transducer was not in direct contact with the specimen. Results were satisfactory at a high gain (80-100 dB) as judged from observation of acoustic emission from an aluminum specimen and comparison with published information.

Type 304L Stainless Steel

Acoustic emission from Type 304L steel depended on grain size and hydrogen. Neither event count nor signal intensity displayed any significant response from hydrogen-free specimens with 10 or 100 μm grain size. For the largest grain size, there was a small increase in AE intensity at yield. The cumulative event count reached ~ 100 at fracture and was distributed evenly over the deformation except for an initial rapid rise at yield.

Substantial acoustic emission activity was detected in specimens saturated with hydrogen prior to test. Details of the emission differed among the specimens, but the same general

patterns of event rate vs. deformation occurred in all three (Figure 2). After acoustic emission began at yield, the event rate rose to a maximum and then declined until fracture. This pattern was independent of amplification for gains of 80 and 100 dB, although absolute values of event rate differed. Acoustic emission was not detected at a gain of less than 70 dB. The maximum event rates were 330-400 sec^{-1} for an 80 dB gain and 1040-1260 sec^{-1} for a 100 dB gain. As the strain to fracture increased, the peak in acoustic emission event rate shifted to higher strains also. There was never any necking of the specimens, so the decline in event rate took place while the applied load was rising. Based on limited observations, the maximum in the event rate does not correspond to the onset of visible microcracks in the specimens although there is a strong temptation for such a correlation.

An increase in grain size reduces the acoustic emission rate. The maximum event rates for specimens of 100 and 400 μm grain size were 1135-1330 sec^{-1} and 1040-1260 sec^{-1} , respectively. The cumulative acoustic emission is the summation of the rate over the time of the test and was about the same for the two grain sizes despite the differences in event rate and plastic strain at failure.

Type 316 Stainless Steel

There was no significant acoustic emission from Type 316 stainless steel even when saturated with hydrogen. The ductility and fracture mode of this steel were unaffected by hydrogen, but the strength increased slightly.

DISCUSSION

The simultaneous appearance of acoustic emission and a change in fracture mode (Figure 3) implies that the processes causing cleavage fracture are also responsible for the acoustic activity. If this correlation is valid, an analysis of acoustic emission should assist identification of the mechanism of microcrack formation.

The acoustic activity could result from energy releases accompanying microcrack formation, or from plastic deformation processes (8). In the first case, the cumulative event count would be expected to correlate with the number of microcracks and vary inversely with the grain size, which is not the case. Furthermore, acoustic emission commences with the onset of plastic deformation at <1% strain and precedes observable microcrack formation at 12-14% strain. The considerations suggest that dislocation activity is the more likely source of acoustic emission.

Plastic deformation of Type 304L stainless steel occurs primarily by slip, which is mostly coplanar, because of the low stacking-fault energy. In addition, deformation twinning and strain-induced transformation to ϵ -phase and α' -martensite occur and contribute to the plastic strain. None of these processes produced acoustic emission detected in hydrogen-free specimens except for the very largest grain size where a small

amount of activity was noted. This observed grain-size effect agrees with the notion that the amplitude and therefore the detectability of acoustic emission increases as the mean free path of dislocation motion increases with grain size. A mean free path of several hundred micrometers is needed apparently to generate a detectable acoustic signal in Type 304L stainless steel. If a large mean free path is necessary for acoustic emission, α' -martensite formation is not a likely emission source because dislocation motion is restricted to distances of less than a micrometer. Slip, ϵ -formation, and deformation twinning on the other hand are all associated with dislocation motion over distances comparable to the grain size. In alloys of low stacking-fault energy, these three processes are interrelated and inseparable. For example, ϵ -phase and deformation twins are generated by coplanar motion of partial dislocations on every other slip plane and on every slip plane, respectively.

A model was developed by Frederick and Felbeck to explain dislocation motion as a source of acoustic emission (10). Application of this model to Type 304L stainless steel suggests that the ratio of slip region diameter to source length is about three for specimens free of hydrogen and about 20-50 for hydrogen saturated specimens. Furthermore, hydrogen would have to reduce the source length from $\sim 10^{-4}\text{m}$ to $\sim 10^{-5}\text{m}$ to be consistent with the model and data, assuming that the slip region remained constant. Conversely, if the source length remained constant,

the slip region would have to increase. The former alternative is reasonable as an explanation of how hydrogen evokes acoustic emission in this alloy, and is consistent with dislocation damping measurements on stainless steel (n).

SUMMARY

Acoustic emission accompanies plastic deformation of hydrogen saturated Type 304L stainless steel at 200-250 K, but is absent in specimens that are not charged. The pattern of emission and peak event rate depend on grain size. Strain dependence of acoustic emission does not correlate with the presence of observable microcracks.

The results suggest that dislocation motion is the most likely source of acoustic emission. Microcrack nucleation from coalescence of a relatively few dislocations and incremental propagation are consistent with the observations.

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TABLE I

Stainless Steel Compositions

	<u>Type 304L^a</u>	<u>Type 316^b</u>
Chromium	18.35	16-18
Nickel	10.29	10-14
Manganese	1.57	2.0 max.
Molybdenum	0.17	2-3
Carbon	0.03	.08 max.
Nitrogen	-	-
Silicon	0.43	1.0 max.
Phosphorous	0.015	.045 max.
Sulfur	0.008	.03 max.
Copper	-	-
Cobalt	0.09	-

a. Analysis of heat.

b. Nominal composition.

TABLE II

Mechanical Properties of Type 304 Stainless Steel
Tested at ~200 K

<u>Grain Size, μm</u>	<u>Hydrogen Exposure</u>	<u>Strength, MPa</u>		<u>Elongation, %</u>		<u>Red. in Area, %</u>
		<u>Yield</u>	<u>Ultimate</u>	<u>Uniform</u>	<u>Total</u>	
10	None	520	1300	66	73	82
100	None	340	1200	60	72	92
400	None	250	1130	55	63	81
100	69 MPa	370	750	30	30	28
400	69 MPa	280	540	20	20	23
400	69 MPa	270	470	17	17	15

FIGURE 1. Specimen and Acoustic Emission System.

FIGURE 2. Acoustic Emission Frequency of Type 304L Stainless Steel Saturated in D₂ at 69 MPa.

Hydrogen Embrittled

Normal Fracture

Hydrogen Embrittled

Normal Fracture

FIGURE 3. Type 304L Stainless Steel.