Proc Third Internat Conf on
Effects of Hydrogen on Behavior of Materials
TMS-AIME, Warrendale, PA, in press
DOE/EY-76-C-02-1198/1325 +10 reprint 10/14

STUDIES OF HYDROGEN EMBRITILEMENT AND STRESS-CORROSION
CRACKING IN AN ALUMINUM-ZINC-MAGNESIUM ALLOY*

S. W. Ciaraldi, J. L. Nelson, R. A. Yeske, and E. N. Pugh**
Department of Metallurgy and Mining Engineering
University of Illinois
Urbana, Illinois 61801

MASTER

Tensile tests have been carried out on a high-purity Al-5.6 Zn-2.6 Mg alloy hydrogenated by exposure to moist air. The results indicate that internal hydrogen embrittlement occurs by the formation and rupture of a stress-induced hydride at the grain boundaries. The hydride, identified by electron diffraction as AlH_3 , is shown to be unstable in laboratory air, reverting to aluminum. The hydride phase was not detected in specimens failed by SCC, despite evidence that hydrogen is transported ahead of advancing stress-corrosion cracks, and this leads to the possibility that a basically different mechanism may be responsible for SCC in this alloy.

DISCLAIMER

This book was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

*This work was supported in part by the U.S. Department of Energy, Contract DOE EY-76-C-02-1198, and by the Air Force Office of Scientific Research under Grant AFOSR 77-3175.

**The authors are now at Amoco Research Center, Naperville, IL (SWC); INCO Research and Development Center, Suffern, NY (JLN); Westinghouse Research and Development Center, Pittsburgh, PA (RAY); and National Bureau of Standards, Washington, DC (ENP).

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Introduction

Following the work of Gruhl and Brungs (1), it has become well established that hydrogen introduced into aluminum-zinc-magnesium alloys leads to significant embrittlement (2-12). Hydrogen has been introduced electrolytically (2,3,7,10), by exposure to moist air at room temperature (the pre-exposure effect) (4,6,8) or at 70 °C (5,9,12), by hydrogen-ion bombardment (11), and by polishing with aqueous slurries (12), and the path of fracture is generally intergranular. The mechanism of embrittlement has not been established, and is a primary objective of the present work. In particular, the present studies were aimed at determining the nature and role of a fragmented layer which had been observed at the fracture surfaces of hydrogen-embrittled specimens (6,8,11).

The fact that hydrogen can induce brittle intergranular cracking makes it attractive to speculate that intergranular SCC, a major practical problem in these high-strength alloys, is also due to hydrogen embrittlement. However, there is no direct evidence for a causal relationship at present, and this question is also addressed in this paper.

Experimental

The experiments were carried out on a single high-purity ternary alloy, kindly provided by Mr. D. O. Sprowls, Alcoa. The alloy contained 5.63 wt pct zinc and 2.59 wt pct magnesium, and the concentration of each of the elements silicon, iron, copper, manganese, chromium, nickel, and titanium was < 0.01 wt pct. The cast ingot was rolled to sheet of thicknesses 0.305, 0.076, and 0.038 cm, respectively, with intermediate and final anneals at 470 °C in air. The air-formed film produced at 470 °C is known to play an important role in the embrittlement phenomenon (5,6). Two types of specimens were cut from the sheet:

- (i) Tensile specimens of thickness 0.076 and 0.038 cm with a gauge length 0.635 cm long and 0.318 cm wide. No machining was carried out on the rolled surfaces.
- (ii) Bend specimens of cross section 0.25 cm x 0.25 cm.

The specimens were solution treated for 1 h at 470 °C in dry argon and water quenched to room temperature. The 0.076 cm-thick tensile specimens were then aged for 60 d at room temperature in either dry or moist air. Based on previous work (4,6,8), the latter treatment was expected to introduce significant amounts of hydrogen into the specimens. After natural aging, the specimens were tested to failure at room temperature in dry argon at a strain rate of $1.3 \times 10^{-4} \text{ s}^{-1}$.

After quenching, the 0.038 cm-thick tensile specimens and the bend testpieces were aged to peak hardness (24 h) at 130 °C in a silicone-oil bath. The tensile specimens were cleaned with acetone and, using the method described by Scamans and Tuck (9), they were then hydrogenated by exposure to water vapor saturated air (WVSA) at 70 °C for various times up to 7 d. The hydrogen content of certain specimens was measured by means of a LECO Hydrogen Determinator. Other tensile specimens were tested to failure as in the case of the 0.076 cm-thick specimens. The bend specimens were electropolished and stressed under constant deflection in a four-point bend jig in either aqueous 3 pct sodium chloride solution at room temperature, or in 70 °C WVSA.

DISCLAIMER

This book was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

Electron-diffraction studies were carried out to identify the fragmented layer found on certain fracture surfaces. These were performed using a JEOL 200 TEM operated at 200 kV. Diffraction patterns were obtained either by transmission through pieces of the layer extracted by replica techniques, or directly from the fracture surface, either by glancing-incidence diffraction or by transmission through fragments partially detached from the surface. Diffraction was carried out at the eucentric position of the microscope to minimize error in the use of the camera constant. A densitometer was used to scan the diffraction patterns. One series of diffraction experiments was carried out on the naturally aged tensile specimens, 0.076 cm thick, fractured at a strain rate of $1.3 \times 10^{-4} \text{ s}^{-1}$ in argon and subsequently stored in laboratory air before being placed in the TEM. A second series was carried out on the peak-aged tensile specimens, 0.038 cm-thick, which were exposed to 70 °C WWSA for 7 d. These specimens were etched with Tuckers etch to remove any surface films, rinsed with acetone, and dried. They were then placed in a small tensile machine enclosed in a glove bag which was also attached to the port of the TEM. The glove bag was purged of air by means of flowing argon and the specimens fractured at a strain rate of $1.3 \times 10^{-4} \text{ s}^{-1}$. One half of the fracture surface was transferred to the TEM within 5 min of fracture without exposure to air.

Results and Discussion

Tensile Tests

Data from tensile tests on 0.076 cm-thick specimens naturally aged for 60 d in dry or moist air are given in Table I.

Table I

Results of tensile tests on specimens naturally aged for
60 d in dry or moist air. Strain rate $1.3 \times 10^{-4} \text{ s}^{-1}$

Aging Environment (dry or moist air)	Ultimate Tensile Strength (MN/m ²)	Total Strain to Failure (pct)
Dry	285	42.4
	274	39.4
	261	40.5
	287	41.4
Moist	238	22.1
	205	15.0
	222	19.0

Exposure to moist air can be seen to have caused significant reductions in tensile properties compared to those for specimens aged in dry air. The latter underwent ductile transgranular fracture. Specimens aged in moist air also failed predominantly by the ductile mode, but, in addition, the fracture surfaces exhibited up to 30 pct intergranular fracture, Figure 1(a). At higher magnifications, several of these intergranular

facets were found to exhibit the fragmented layer, reported earlier (6,8), Figures 1(b)-(d). Examination of the opposing fracture faces indicated that part of the layer was frequently attached to one side and the remainder to the other, Figures 1(c) and (d), indicating clearly that the layer was not produced after fracture. The maximum thickness of the layer was $\sim 1 \mu\text{m}$. Electron-diffraction studies on extracted pieces yielded ring patterns which were indexed as FCC aluminum, see below. In no cases were patterns corresponding to aluminum hydroxides or oxides observed. It should be pointed out that the fragmented layers had been exposed to laboratory air for at least 2 to 3 d before the diffraction experiments were carried out.

Figure 1. Scanning electron micrographs of fracture surfaces of a tensile specimen naturally aged for 60 d in moist air. The intergranular facets in (a) are shown at high magnifications in (b)-(d), revealing the fragmented layer. The opposing fracture faces are illustrated in (c) and (d), indicating the matching and interlocking nature of the layer.

As heat-treated specimens naturally aged in dry and moist air were also tested at a higher strain rate, viz. 10^{-2} s^{-1} . The results, presented in detail elsewhere (6), demonstrated that pre-exposure embrittlement was completely suppressed at this strain rate, no significant differences in mechanical properties being observed, and ductile fracture occurring in all cases with no evidence of the fragmented layer. These results indicated that the layer observed in tests at $1.3 \times 10^{-4} \text{ s}^{-1}$ is not formed by penetration of grain boundaries by a corrosion process during aging,

AMERICAN
SOCIETY

THIS PROCEEDINGS TEMPLATE
For final size: 6" x 9" book

but that it is produced during straining. The necessity for slow strain rates also suggests that formation of the layer requires diffusion in the lattice, presumably of hydrogen.

To investigate the nature of the film, further tests were carried out on 0.038 cm-thick tensile specimens, aged to peak hardness at 130 °C and hydrogenated by exposure to WWSA at 70 °C. The hydrogen content of these specimens was found to increase progressively with exposure time, attaining ~ 300 ppm (~ 1 at pct) after 7 d (12). The presence of the hydrogen led to increasing embrittlement, manifested by reductions in ultimate tensile strength and plastic strain, ϵ_{pl} , defined as

$$\epsilon_{pl} = \frac{\text{crosshead displacement after yielding}}{\text{gauge length}} \times 100 \text{ pct.}$$

It can be seen in Figure 2 that embrittlement for exposure times up to approximately 4 d was characterized by large reductions in ϵ_{pl} but little decrease in UTS, whereas there were large reductions in UTS for longer exposure times. These two regimes are subsequently referred to as Stage I and Stage II. Increasing exposure times were associated with increasing amounts of brittle intergranular fracture, and the intergranular facets exhibited the fragmented layer of the type illustrated in Figure 1. The amount and thickness of the layer increased with increasing exposure time. The maximum thickness of the layer was ~ 2.5 μm .

Figure 2. Effect of exposure to 70 °C WWSA on the mechanical properties of 0.038-cm thick tensile specimens tested in dry argon at a strain rate of $1.3 \times 10^{-4} \text{ s}^{-1}$.

Studies were made of the effects of strain rate and thermal treatments designed to outgas the hydrogen. These results, described in detail elsewhere (12), indicated that Stage I embrittlement could be completely suppressed by testing at a strain rate of $1.3 \times 10^{-2} \text{ s}^{-1}$, whereas testing

at this increased rate did not influence the degree of embrittlement in Stage II. In fact, brittle intergranular facets and the fragmented layer were produced in Stage II specimens fractured under impact conditions. It should be recalled that embrittlement was suppressed in specimens hydrogenated by exposure to moist air at room temperature, suggesting that the hydrogen contents of those specimens corresponded to Stage I of those exposed to 70 °C WWSA. Outgassing hydrogen by a complete re-heat treatment, i.e., solution treatment at 470 °C in argon and aging at 130 °C, completely recovered the mechanical properties of both Stage I and II specimens, and demonstrated the reversibility of the phenomenon. TEM studies of samples produced by thinning specimens exposed for 7 d at 70 °C WWSA revealed no evidence for the presence of any layer or other unusual structures at the grain boundaries, suggesting that the fragmented layer is not formed prior to stressing the specimens.

Electron-diffraction patterns were taken directly from the fragmented layer on intergranular facets in specimens which had been exposed for 7 d to 70 °C WWSA. As described above, these specimens were fractured in dry argon and transferred to the TEM quickly and without exposure to laboratory air. This procedure was adopted because all forms of aluminum hydride have been reported to be unstable, particularly in moist environments (13). Under these conditions, the patterns obtained were quite different from those obtained for the specimens embrittled by room-temperature exposure, in which the fracture surfaces had been exposed to laboratory air for long periods before the diffraction patterns were obtained. The patterns, Figure 3(a), were characterized by continuous rings, indicating a very fine grain size, and the plane spacings obtained from these rings were found to be consistent with those for a hexagonal aluminum hydride, AlH_3 , with $a = 2.90 \text{ \AA}$ and $c = 4.55 \text{ \AA}$ (14). Table II lists the predicted plane spacings for this structure and the spacings obtained experimentally for several specimens. It can be seen that good correlation exists, although several lines are missing. The detailed structure of AlH_3 is not known, so that it is not possible to calculate structure factors and to determine which reflections would have low intensities. The spacings were also compared to those of various forms of alumina and aluminum hydroxide but no correspondence was found.

Figure 3. Electron-diffraction patterns obtained from a fragmented layer on the fracture surface of a peak-aged specimen exposed to 70 °C WWSA for 7 d and tested in argon at a strain rate of $1.3 \times 10^{-4} \text{ s}^{-1}$. Pattern (a) was obtained approximately 0.5 h after fracture without exposure to air and (b) was obtained from the same fragment after subsequent exposure to laboratory air for 150 h.

Table II

Comparison of plane spacings for AlH_3 with typical values obtained from electron-diffraction patterns such as that shown in Figure 3(a).

Reflection	Plane Spacing for AlH_3 (a) ($\text{\AA}$)	Plane Spacings Obtained from Typical Diffraction Patterns		
		1	2	3
0001	4.55			
01 $\bar{1}$ 0	2.51	2.53	2.54	2.53
0002	2.28			
01 $\bar{1}$ 1	2.20	2.19	2.16	2.21
01 $\bar{1}$ 2	1.69	1.70		
0003	1.52	1.49	1.52	1.51
11 $\bar{2}$ 0	1.45			
11 $\bar{2}$ 1	1.38	1.41	1.42	
01 $\bar{1}$ 3	1.30	1.33		1.33
02 $\bar{2}$ 0	1.26			
11 $\bar{2}$ 2	1.22			
02 $\bar{2}$ 1	1.21	1.20		1.21

(a) Calculated for hexagonal structure with $a = 2.90 \text{ \AA}$ and $c = 4.55 \text{ \AA}$ (14).

Several fractured specimens for which the AlH_3 structure had been observed were subsequently exposed to laboratory air for approximately 150 h, and further electron diffraction patterns were obtained. In no cases were patterns consistent with AlH_3 observed, all the patterns being indexed as polycrystalline aluminum. For example, Figure 3(b) shows a pattern obtained from the fragmented layer from which the pattern shown in Figure 3(a) had been obtained previously. The polycrystalline nature of the aluminum pattern is of particular interest, since patterns taken from the fracture surfaces of non-embrittled specimens corresponded to single-crystal aluminum. Such single-crystal patterns would be expected since the grain size of the alloy was large ($\sim 300 \mu\text{m}$) and since the beam diameter was small (200 to 300 $\text{\AA}$).

Tests on Bend Specimens

Stress-corrosion tests were carried out on peak-aged bend specimens tested under constant deflection in 3 pct aqueous sodium chloride (6). Crack-growth rates in these tests varied between $4.2 \times 10^{-6} \text{ cm s}^{-1}$ and $2.0 \times 10^{-5} \text{ cm s}^{-1}$, depending on deflection. The resulting fracture surfaces consisted entirely of brittle intergranular facets except for several

grains on the compression side which exhibited ductile transgranular fracture. SEM examination of the intergranular SCC facets revealed no evidence of the fragmented layer.

In one test, a specimen was unloaded prior to complete fracture, dried with acetone, and then immediately strained to failure in tension at a strain rate of $1.1 \times 10^{-5} \text{ s}^{-1}$ in dry air. Fractographic examination of the transition region from I-SCC to ductile overload indicated an intervening intergranular region, 200 to 300 μm wide, which exhibited the fragmented layer, Figure 4(a). No electron-diffraction studies were carried out on this layer, but its characteristics were the same as the layer observed in the preceding section. It is considered that this region was also produced by internal hydrogen embrittlement, the hydrogen in this case being introduced from the aqueous solution at the tip of the advancing stress-corrosion crack.

Figure 4. Scanning electron micrographs of fracture surfaces of peak-aged bend specimens. (a) A region in the transition zone between I-SCC and ductile overload for a specimen partially fractured in aqueous sodium chloride and then strained to failure in tension in dry air. (b) Taken from a specimen tested under constant deflection in 70 °C WWSA.

Constant-deflection bend tests were also conducted in 70 °C WWSA (12): Slow crack growth again occurred, with complete failure taking place within 24 h; the crack velocity was not determined in these tests. The primary fracture mode was again brittle intergranular but, in contrast to the tests in aqueous sodium chloride, the fragmented layer was detected on many of the intergranular facets, Figure 4(b). No attempts were made to carry out electron-diffraction studies on these layers.

General Discussion and Conclusions

The major finding of these studies is the occurrence of a layer of AlH_3 at the intergranular fracture surfaces produced in tensile tests on hydrogenated specimens. The results established that the hydride is not produced by a corrosion process prior to testing, and strongly suggest that it is formed during stressing. Thus it is concluded that the hydride is stress-induced, and that its formation is responsible for internal hydrogen embrittlement (IHE) in this alloy, by a mechanism similar to that proposed for zirconium (15) and niobium (16).

The occurrence of two stages of embrittlement for specimens hydrogenated by exposure to 70 °C WWSA, Figure 2, can be rationalized in terms of the several different roles of stress in the embrittlement process.

When the hydrogen concentration is low, Stage I, it is considered that diffusion of hydrogen is necessary into the region ahead of the advancing crack tip to provide a sufficient concentration to produce AlH_3 . Thus the large fracture stresses in Stage I are considered to correspond to the stresses necessary to concentrate sufficient hydrogen by the Troiano-Oriani mechanism. This view is consistent with the observed strain-rate dependence of embrittlement in Stage I.

In Stage II, it is considered that sufficient hydrogen is already present at the grain boundaries to form AlH_3 , so that diffusion of hydrogen over long distances is not required during stressing--hence the absence of a strain-rate dependence. Given the measured bulk hydrogen concentrations, this view requires that virtually all the hydrogen in the alloy is at grain boundaries for stoichiometric AlH_3 to be formed. Under these conditions, the formation of AlH_3 requires only the stress necessary to induce the phase transformation. It is assumed here that the transformation is martensitic in nature, requiring only local rearrangement of atoms, and that it can be stress induced. It should be emphasized that the details of the phase transformation are not known, but, by analogy with other hydrides, one can expect ordering of hydrogen and correlated shears of the aluminum lattice to be involved; stress might also be important in accommodating the volume change associated with the transformation (16). The third stress to be considered is that to fracture the hydride, or, more probably, the hydride-matrix interface. Thus the low fracture stresses in Stage II correspond to either the stress required to induce the transformation or the fracture stress, whichever is larger.

Detection of the fragmented layer on the fracture surfaces of all specimens which underwent IHE suggests that the phenomenon can be attributed to a hydride-rupture mechanism (15,16), and that there is no necessity to seek other embrittling mechanisms in this system. Conversely, the absence of the layer on the fracture surfaces of specimens which underwent SCC in aqueous sodium chloride questions whether hydrogen plays a role in the stress-corrosion process. The experiment in which a stress-corrosion crack was halted and the specimen then fractured in dry air, Figure 4(a), clearly demonstrates that sufficient hydrogen is transported ahead of the advancing stress-corrosion crack to produce IHE; a similar effect has been observed by other workers (17,18). Further, the occurrence of slow crack growth in specimens stressed in 70 °C WWSA and the presence of the fragmented layer on the resulting fracture surfaces indicates that the hydride-rupture mechanism can operate when the source of hydrogen is external. Thus the possibility must be recognized that stress-corrosion cracks in this alloy may propagate by a competing and basically different mechanism. For example, it has been argued that the film-rupture mechanism can operate in this system--that is, localized plastic deformation within the soft PFZ at grain boundaries can lead to rupture of the passive film and thus to crack propagation by preferential anodic dissolution (19).

Pursuing the possibility that a second mechanism of slow crack growth can operate in this alloy in aqueous solutions in addition to hydride rupture, it is interesting to note that double plateaus have been reported in the V-K curves for aluminum-zinc-magnesium alloys in some cases (20-22). It may be speculated that this results from the overlapping of two different V-K curves, one corresponding to HE and the other to the second mechanism. In the majority of cases, a single plateau is observed for these alloys, suggesting that one of the V-K curves becomes completely

submerged by the other, so that the corresponding mechanism plays no role in the fracture process. The present observations could be interpreted to indicate that HE is the "submerged" mechanism in this specific case. Further work is clearly necessary to pursue these possibilities. Moreover, it should be recognized that any conclusions for this high-purity alloy cannot necessarily be extended to other high-strength aluminum alloys, particularly to the commercial alloys.

It is concluded that HE of the high-purity ternary alloy investigated in the present work occurs by the formation and rupture of a stress-induced hydride, AlH_3 . Experiments in 70 °C WWSA indicate that the hydride-rupture mechanism can also be responsible for slow crack growth in this alloy. However, failure to detect the hydride phase in specimens failed by SCC in aqueous sodium chloride questions that hydrogen plays a role in the stress-corrosion process.

References

1. W. Gruhl and D. Brungs: Metall., 1969, vol. 23, p. 1020.
2. R. Gest and A. R. Troiano: Corrosion, 1974, vol. 30, p. 274.
3. R. M. Pelloux and J. A. Van den Avyle: Hydrogen in Metals, p. 275, ASM, Metals Park, OH, 1974.
4. L. Montgrain and P. R. Swann: ibid., p. 575.
5. C. M. Scamans, R. Alani, and P. R. Swann: Corros. Sci., 1976, vol. 16, p. 443.
6. J. L. Nelson: Ph.D. Thesis, University of Illinois at Champaign-Urbana, 1976.
7. J. Albrecht, B. J. McTiernan, I. M. Bernstein, and A. W. Thompson: Scripta Met., 1977, vol. 11, p. 893.
8. J. L. Nelson (reported by E. N. Pugh): Mechanisms of Environment Sensitive Cracking of Materials, p. 493, The Metals Society, London, 1977.
9. G. M. Scamans and C. D. S. Tuck: Environment-Sensitive Fracture of Engineering Materials, p. 464, TMS-AIME, Warrendale, PA, 1979.
10. J. Albrecht, A. W. Thompson, and I. M. Bernstein: Met. Trans. A, 1979, vol. 10A, p. 1759.
11. G. H. Koch: Corrosion, 1979, vol. 35, p. 73.
12. S. W. Ciaraldi: Ph.D. Thesis, University of Illinois at Champaign-Urbana, 1980.
13. Metal Hydrides, p. 573, Academic Press, New York, NY, 1968.
14. M. Appel and J. P. Frankel: J. Chem. Phys., 1965, vol. 42, p. 3984.
15. D. G. Westlake: Trans. ASM, 1969, vol. 62, p. 1000.
16. H. K. Birnbaum: Environment-Sensitive Fracture of Engineering Materials, p. 326, TMS-AIME, Warrendale, PA, 1979.

17. B. F. Brown: Private communication, The American University, Washington, DC, 1980.
18. J. E. Morral: University of Connecticut, Storrs, CT, 1980.
19. A. J. Sedriks, P. W. Slattery, and E. N. Pugh: Trans. ASM, 1969, vol. 62, p. 238.
20. M. O. Speidel: The Theory of Stress Corrosion Cracking in Alloys, p. 289, NATO, Brussels, Belgium, 1971.
21. P. K. Poulouse, J. E. Morral, and A. J. McEvily: Met. Trans., 1974, vol. 5, p. 1393.
22. R. Haag, J. E. Morral, and A. J. McEvily: Environmental Degradation of Engineering Materials, p. 19, VPI Press, Blacksburg, VA, 1977.

*Reprints
Reviewed*