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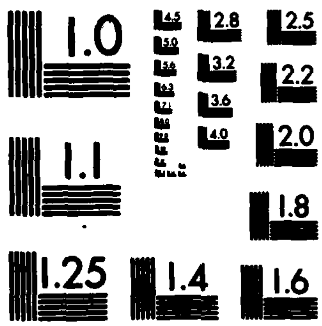

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The combined results of the first three years of the program are presented, with emphasis on the stress corrosion cracking and hydrogen embrittlement of the PM 7090 Al alloy. Additional results on 7075 are also given. In particular, the role of temper and loading mode in susceptibility were examined for three test methods---time to failure of notched round bar specimens in a brine solution; straining electrode tests

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We remain confident that we have established the basis and a good portion of the results necessary to understand, predict and model the role of hydrogen in stress corrosion cracking of high-strength aluminum alloys.

AFOSR ANNUAL TECHNICAL REPORT NO. 3

"Stress Corrosion Cracking of Wrought and P/M High Strength Aluminum Alloys"

CMU Report Number: AFOSR-AL-7
Grant Number: AFOSR 81-0041
Principal Investigators: A.W. Thompson
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1 March 1984
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1.0 ABSTRACT OF RESULTS

The combined results of the first three years of the program are presented, with emphasis on the stress corrosion cracking and hydrogen embrittlement of the PM 7090 Al alloy. Additional results on 7075 are also given. In particular, the role of temper and loading mode in susceptibility were examined for three test methods---time to failure of notched round bar specimens in a brine solution; straining electrode tests on notched round specimens under cathodic charging; and tensile tests on hydrogen pre-charged notched round specimens. These tests form the basis for an extensive, completed Ph.D. thesis which is summarized here. Stress corrosion testing has also been conducted on 7075 in aluminum chloride solutions and on HP 7075.

We remain confident that we have established the basis and a good portion of the results necessary to understand, predict and model the role of hydrogen in stress corrosion cracking of high-strength aluminum alloys.

2.0 TECHNICAL RESULTS

The intent of the overall program is to identify the role played by hydrogen in stress corrosion cracking of high strength aluminum alloys, first by establishing microstructural and fractographic correlations with cracking behavior, and then to develop specific criteria for microstructural optimization in both powder and ingot alloys. Specific emphasis has been placed on the role of such metallurgical variables as precipitate type, size, and distribution, in order to develop them as central variables for the production of wrought (ingot metallurgy or IM) and powder (powder metallurgy or PM) high-strength aluminum alloys, most notably PM 7090 and IM 7075, more resistant to hydrogen embrittlement (HE) and/or stress corrosion cracking (SCC).

Third-year results are summarized in the following sections and will cover the following specific topics: The role of hydrogen microstructure and loading on the stress corrosion cracking response of 7090 and 7075; stress corrosion studies on 7075 in aluminum chloride solutions; and studies on high-purity 7075.

2.1 The Role of Hydrogen in the Stress Corrosion Cracking of PM 7090 and IM 7075 Al

A general description of PM 7090 was provided previously (1), covering microstructural aspects as well as its preliminary response to stress corrosion cracking and hydrogen embrittlement. More detailed results summarizing both the first and second year have also been presented (2). The completion of this work is described in the Ph.D. thesis of R.E. Swanson (3).

Environmental mechanical testing included three test methods, all employing the loading mode technique, which can help discriminate between the relative contributions of hydrogen embrittlement and anodic dissolution to the overall SCC behavior of ferrous and nonferrous alloys (4). The primary test method to establish general response was the use of constant load (or constant torque) applied to a notched round bar held at the free corrosion potential in a chromate-inhibited brine solution (5). Although 3-1/2% NaCl solutions were used satisfactorily for previously conducted tests on 7075, we found extreme exfoliation-type damage on torsion 7090 specimens tested in 3-1/2% NaCl. The exfoliation plates appear to be aligned with oxide stringers in the P/M alloy. The chromate-inhibited brine solution eliminated this damage. This difference in exfoliation behavior is worthy of additional study because of its potentially deleterious impact on service life under specific loading conditions.

This second test method used is the straining electrode test where notched round specimens were cathodically charged under a constant potential of -1500 mV versus the SCE in hydrochloric acid (pH 1), while being strained to failure in either Mode I or Mode III. The value of this approach is that hydrogen can be introduced under more severe entry conditions than with constant load or other tests, allowing maximum effects to be observed and studied.

A third test technique, hydrogen precharging, a less severe test than the SET, was also used. Notched round specimens were cathodically precharged for 10 hours in pH 1 hydrochloric acid at -1500 mV SCE and were strained to failure at crosshead rates of 0.2

mm/min. (Mode I) or 0.001 rpm (Mode III). The results of these tests are similar to those of the SET tests, but the Mode I/ Mode III differences are expectedly smaller, since the precharge conditions are not as severe as those of the SET.

The results and conclusions of work may be summarized (3,6) as follows. The purpose of this study was to investigate the role of hydrogen in the SCC of high strength Al alloys. Microstructural features and mechanical properties used to describe SCC behavior of other 7XXX-series alloys were characterized for PM 7090 and IM 7075. A systematic test scheme was completed to aid in separating SCC mechanisms. Significant experimental contributions from this study include:

1. The use of test specimens with various geometries and loading conditions, as well as test environments can be used to separate SCC processes into initiation and propagation as well as hydrogen and dissolution mechanisms. This represents one of the most comprehensive SCC test programs to date.

2. This study has provided empirical evidence that small notch root radii (about 0.02 mm for 7090 Al) can provide stress singularities which permit the use of stress intensity analysis in both tension and torsion loading of notched cylindrical specimens.

3. The unusual Mode I/ Mode III reversal (2) found for 7090 is potentially important and deserves additional work. The difference in crack opening between the two Modes may enhance local dissolution rates for the more closed crevice produced in Mode III.

The following conclusions can be drawn for the SCC behavior of 7090 and 7075:

1. Extensive loading mode and cathodic charging testing have demonstrated an unequivocal contribution of hydrogen to the SCC of both 7090 and 7075.

2. Overaging dramatically increased the resistance of 7090 to SCC initiation, as determined by DCB testing. Since differences in microstructure and fractography were found to be small between peak aged and overaged 7090, the mechanism for initiation appears to operate on a smaller scale than that used to characterize the microstructures.

3. The contribution of hydrogen to the SCC of 7090 also decreased dramatically for overaging.

4. Increased aging of 7090 gradually increases the resistance to propagation by a hydrogen mechanism.

5. The resistance of 7075 T73 to SCC initiation in both longitudinal and short transverse loading orientations appears related to the resistance of the T73 conditions to the tunneling corrosion attack found in the UT and T6 conditions.

6. The gradual increase in the SCC propagation resistance is accompanied by a gradual decrease in the hydrogen contribution to SCC.

7. The differences in SCC behavior with aging of 7075 and 7090 may be related to differences in grain structure or chemical composition, especially Co and Cu.

2.2 Stress Corrosion Behavior of 7075 Aluminum in Aluminum Chloride Solutions

In stress corrosion cracking (SCC) research on high strength aluminum alloys (such as 7075), aluminum chloride solutions have been used to simulate aggressive chemical conditions, such as at the crack tip of a stress corrosion crack. The question then arises: What is the operating mechanism for SCC in this type of environment? The probable operating mechanisms can be grouped into

- anodic dissolution (AD), and
- hydrogen embrittlement (HE).

We have used the Mode I/ Mode III technique to investigate this question. Our results have been reported (7) and can be summarized as follows.

1. The corrosion rate of the T73 temper is considerably higher than the rates for UT and T6.

2. The aluminum chloride SCC behavior of 7075 in three different tempers is influenced by the loading mode.

3. In both the UT and T6 temper, HE and AD are likely to be the predominant mechanisms for SCC under Mode I and Mode III loading, respectively.

4. The well-known dependence of Mode I SCC properties of high-strength Al alloys on aging temperature has been reconfirmed and attributed to an increasing amount of innocuously trapped hydrogen at rather large precipitates, such as η and T-phase particles; to a decrease in the number of small, H-embrittlement-promoting precipitates; and possibly to decreasing slip coarseness and planarity, all of which occur when aging temperature is increased.

5. Due to similarities in SCC and HE response, mechanical properties and microstructure, the three tempers have been grouped in two sets: {UT,T6} and {T73}.

6. According to the results obtained on the T73 tempered material, it has been shown that highly aggressive environments, such as $AlCl_3$ can add a strong non-SCC component to cracking and so lead to possible misinterpretations of accelerated laboratory SCC tests conducted in this environment.

2.3 Stress Corrosion Cracking of a High Purity 7075 Al Alloy

The stress corrosion response of commercially produced high strength Al alloys, such as 7075, depends strongly on the anisotropy of the grain structure. As previous work on stress corrosion cracking and hydrogen embrittlement showed, the SCC life time and the ductility of hydrogen charged specimens are the lowest in the short transverse direction. Moreover, secondary cracking parallel to the rolling plane is also promoted by the "pancake" grain structure of commercially produced alloys. However, it has not been established how the morphology of the grain structure influences the SCC response and eventually also the SCC mechanism. Therefore stress corrosion testing of a high purity 7075 Al-alloy (low in Fe, Si and Cr), having equiaxed grains, under tension (Mode I) and torsion (Mode III) loading in a 1N aluminum chloride solution has been performed. Comparison of results obtained recently on a commercially produced 7075 Al alloy (7) with the results of this investigation are being used to study the influence of grain morphology and microstructure on the stress corrosion properties and mechanisms under either tension or torsion loading. Results of the work are being prepared for publication (8).

2.4 Needs for Additional Work

Earlier work in this program has followed the conventional view of the role of microstructure in SCC of 7XXX alloys, namely that slip planarity effects are central to behavior. It is now obvious from the 7090 results presented in this report, as well as from ealier results on commercial grain structures of 7050, 7075 and 2124, that slip planarity effects in these alloys are sufficiently modest that they cannot be the dominant variable in SCC behavior. In the concluding years ot this five-year program, we propose both to evaluate quantitatively how important is the role of slip planarity, and also to identify the critical microstructural variable in hydrogen embrittlement and in SCC of high-strength aluminum alloys. For this work we will use the high-purity (HP) 7075 alloy.

While the fractography seems to be dominated by consituent particles such as oxides, the trends in failure times in the SCC tests indicate a role that must be played by the aging precipitates, but not apparent from fractography. For that reason, we believe it useful to characterize carefully the aging sequence of the microstructure via TEM. If the fine grain size impedes precipitate identification, the material can be treated to give a larger grain size prior to solution treating and aging. This larger grained material may also be used as part of a study to characterize the slip mode as a function of age condition. Previous work in our group on HP 7075 has established the processing conditions necessary to achieve the desired grain growth.

The behavior of grain boundary precipitates has long been implicated in SCC behavior, but were only regarded as sites for preferential anodic dissolution. The concept we propose to test is distinctly different: that these precipitates act as hydrogen traps of an effectiveness which varies with heat treatment. Detailed trapping models already exist, and trapping effects may well be viable explanation for hydrogen effects in aluminum alloys. Any rationale based on grain boundary precipitates, however, suffers from the complication that the character and size of these precipitates change with heat treatment at the same time as do the grain interior precipitates which affect slip planarity and coarseness. However, we have confirmed in recent work that it is possible to vary these precipitate populations independently with appropriate heat treatments. We thus are using such heat treatments in our work. By varying both slip coarseness, and also (separately) grain boundary precipitates, we can test the response of 7075 to hydrogen charging, SET testing, and (to the extent that the program scope permits) SCC conditions.

As part of the foregoing studies, it will be important to identify the operating nuclei for fracture events, with and without hydrogen. One means of study for these phenomena is the use of systematic variations in stress state, through use of varying notch geometries and of plane strain tensile specimens. (These techniques are already in use in separately-funded programs in our laboratory on fracture fundamentals in steels.) This will enable us to identify whether fracture is stress-controlled or strain-controlled; whether grain boundary precipitates act as preferential fracture nuclei when hydrogen is present; and to measure the microvoid or intergranular fracture initiation conditions.

The end product of this work should be a further increase in understanding of microstructural effects on hydrogen and SCC fracture of high-strength aluminum alloys. The issue of slip planarity vs. coarseness should be resolved, and an answer obtained as to how important is the role of grain boundary precipitates as traps. These issues must be clarified before alloy design recommendations for resistance to environmental fracture in these alloys can be formulated.

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4.0 AFOSR-SPONSORED PUBLICATIONS (1983)

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2. I.M. Bernstein and A.W. Thompson, "The Importance of Transient Effects Resulting from Dislocation Transport of Hydrogen", in Atomistics of Fracture (R.M. Latanision, ed.), Plenum Press, N.Y., pp. 813-20 (1983).
3. M. Mueller, A.W. Thompson and I.M. Bernstein, "Recovery Behavior of Hydrogen-Charged 7075-T6 Aluminum", Scripta Met., 17 (1983) 1039-42.
4. I.M. and A.W. Thompson, "The Role of Microstructure in Hydrogen Embrittlement", A.R. Troiano Honarary Volume (R. Gibala, ed.), Academic Press, N.Y., in press.
5. D.A. Hardwick, A.W. Thompson and I.M. Bernstein, "The Effect of Copper Content and Heat Treatment on the Hydrogen Embrittlement of 7050-type

Alloys", in Control and Exploitation of the Corrosion of Aluminum Alloys, in press.

6. A.W. Thompson and I.M. Bernstein, "Loading Mode (Mode I–Mode III) Testing for Stress Corrosion Cracking", in Environment-Sensitive Fracture: Evaluation and Comparison (E.N. Pugh and G.M. Ugiansky, eds.) ASTM, in press.

7. M.P. Mueller, A.W. Thompson and I.M. Bernstein, "Stress Corrosion Behavior of 7075 Aluminum in 1N Aluminum Chloride Solutions", Corrosion, in press.

8. W.Y. Choo and I.M. Bernstein, "The Effective Diffusivity of Hydrogen in Vapor-deposited Aluminum", Met. Trans. A, in press.

5.0 PRESENTATIONS

1. A.W. Thompson, "Recent Progress in Understanding Environmental Fracture of High-Strength Aluminum Alloys" (invited), Banbury Laboratories, Alcan International, Banbury, UK, 8 February 1983.

2. M.P. Mueller, I.M. Bernstein and A.W. Thompson, "The Controlling Mechanism for Stress Corrosion Cracking of 7075 Al Alloy in an $AlCl_3$ Solution", Annual Meeting, AIME, Atlanta, 8 March 1983.

3. M.P. Mueller, I.M. Bernstein and A.W. Thompson, "Recovery Effects in Hydrogen-charged 7075 Al Alloy", ibid, 8 March 1983.

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5. A.W. Thompson, "Micromechanisms of Environmental Fracture", Laboratoire de Metallurgie Physique Seminar, Universite de Paris-Sud, Orsay, France, 11 April 1983.

6. A.W. Thompson, "The Interaction of Microstructure with Micromechanisms of Hydrogen-assisted Fracture" (invited), Research Center, Brown, Boveri and Co., Baden, Switzerland, 13 April 1983.

7. A.W. Thompson, "Modeling of Hydrogen Effects on Ductile Fracture", (invited) Conf. on Crack Tip Structure and Processes, Nat'l Bureau of Standards, Gaithersburg, MD, 7 June 1983.

8. R.E. Swanson, I.M. Bernstein and A.W. Thompson, "The Role of Hydrogen in the Stress Corrosion Cracking of P/M X-7090 Aluminum", Fall Meeting, TMS-AIME, Philadelphia, 5 Oct. 1983.

9. M. Mueller, I.M. Bernstein and A.W. Thompson, "Stress Corrosion Cracking of a High Purity 7075 Al Alloy", ibid., 6 Oct. 1983.

10. I.M. Bernstein, "Microstructural Control of Environmental Embrittlement" (invited), C.S. Barrett Silver Medal Lecture, Rocky Mountain Chapter, ASM, Denver, 6 December 1983.

5.1 Technical Contacts with Other Investigators

The principal investigators continue to have significant and often extensive contacts with other AFOSR investigators in related fields, as briefly summarized below.

Professor J.L. Swedlow (solid mechanics) – Carnegie-Mellon University: Discussions and guidance for the development of finite element methods to study and model stress and strain localization.

Professor J.C. Williams – Carnegie-Mellon University: A continuing dialogue on the role of microstructures on strengthening mechanisms in aluminum alloys.

Professor R.P. Wei – Lehigh University: Discussions on the role of loading mode on discriminating fracture mechanisms.

Professor E.J. Starke – University of Virginia: Discussions of the role of Cu additions to Al-Zn-Mg alloys on microstructure and SCC resistance.

5.2 Personnel

A.W. Thompson – Professor and Co-Principal Investigator
(10% AY, 30% summer).

I.M. Bernstein – Professor and Co-Principal Investigator
(10% AY, 10% summer).

R.E. Swanson – Graduate Student (completed Ph.D., Dec. 1983).

M. Mueller – Post-doctoral Associate (completed work, Sept. 1983).

W.Y. Choo – Post-doctoral Associate, no cost (completed work, Aug. 1983).

D. Nguyen – Post-doctoral Associate, from National Research Council, Canada (began work, October 1983).

J. Bray – Graduate Student (began Sept. 1983)

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