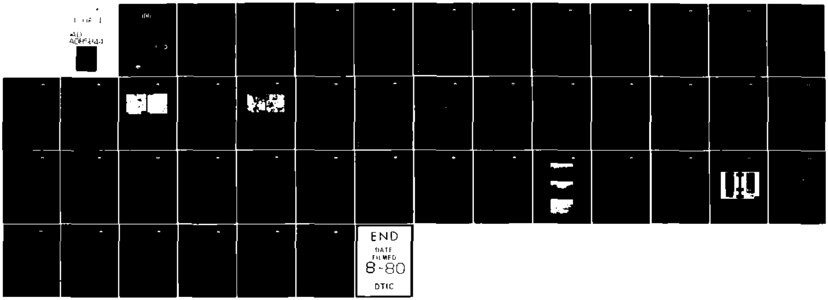
AD-A085 844 ROCKWELL INTERNATIONAL THOUSAND OAKS CA SCIENCE CENTER F/G 1/3
MECHANICAL BEHAVIOR OF AIRFRAME MATERIALS.(U)
MAR 80 J A WERT, N E PATON, J C CHESNUTT F44620-76-C-0025
UNCLASSIFIED SC5053.6FR AFOSR-TR-80-0451 NL



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(11) LEVEL II

Copy No. 6

MECHANICAL BEHAVIOR OF AIRFRAME MATERIALS

FINAL REPORT FOR THE PERIOD
January 1, 1976 through December 31, 1979

ADA085844

GENERAL ORDER NO. 5053
CONTRACT NO. F44620-76-C-0025

Prepared for

Air Force Office of Scientific Research
Building 410
Bolling Air Force Base
Washington, D.C. 20332

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MARCH 1980

Rockwell International
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9 REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER: 18 AFOSR TR- 80 - 0451
2. GOVT ACCESSION NO.: AD-A085 844
3. RECIPIENT'S CATALOG NUMBER:

4. TITLE (and Subtitle): 6 MECHANICAL BEHAVIOR OF AIRFRAME MATERIALS.
5. TYPE OF REPORT & PERIOD COVERED: Final Report 01/01/76 thru 12/31/79
6. PERFORMING ORG. REPORT NUMBER: 14 SC5053.6FR

7. AUTHOR(s): 10 J.A. Wert, N.E. Paton [redacted] J.C. Chesnutt, ~~Science Center~~ Rockwell International
8. CONTRACT OR GRANT NUMBER(s): 15 F44620-76-C-0025

9. PERFORMING ORGANIZATION NAME AND ADDRESS: Science Center, Rockwell International, 1049 Camino Dos Rios, Thousand Oaks, CA 91360
10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: Project No. 2306/A1, 61102F

11. CONTROLLING OFFICE NAME AND ADDRESS: Air Force Office of Scientific Research/NE, Bolling Air Force Base, Bldg. 410, Washington, DC 20332
12. REPORT DATE: 11 Marc[redacted]80
13. NUMBER OF PAGES: 40

14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office): 12 47
15. SECURITY CLASS. (of this report): Unclassified
15a. DECLASSIFICATION/DOWNGRADING SCHEDULE:

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

16 2306 17 A1

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

9 Final rept.

18. SUPPLEMENTARY NOTES

1 Jan 76-31 Dec 79

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Titanium, hydrogen embrittlement, fracture, aluminum alloys, grain size

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This is the final report for a program which was initiated in 1976 to systematically evaluate the influence of hydrogen on mechanical properties of alpha titanium alloys. In the second year of the program (1977), the effort was extended to include work on the properties of fine grain aluminum alloys. Both segments of the extended program have yielded promising results which are summarized in this report. Amont the more important results have been a demonstration of high sustained-load cracking rates and an acceleration

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of fatigue crack propagation rates by tensile hold periods in Ti-6Al containing modest amounts of hydrogen. The study of fine grain aluminum alloys has demonstrated a marked increase in exfoliation corrosion resistance in 7075 Al with fine equiaxed grain, as compared to standard commercial products. Both of these observations have important implications for the structural efficiency of Ti and Al alloys as airframe materials.

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1.0 ABSTRACT

This is the final report for a program which was initiated in 1976 to systematically evaluate the influence of hydrogen on mechanical properties of alpha titanium alloys. In the second year of the program (1977), the effort was extended to include work on the properties of fine grain aluminum alloys. Both segments of the extended program have yielded promising results which are summarized in this report. Among the more important results have been a demonstration of high sustained-load cracking rates and an acceleration of fatigue crack propagation rates by tensile hold periods in Ti-6Al containing modest amounts of hydrogen. The study of fine grain aluminum alloys has demonstrated a marked increase in exfoliation corrosion resistance in 7075 Al with fine equiaxed grains, as compared to standard commercial products. Both of these observations have important implications for the structural efficiency of Ti and Al alloys as airframe materials.

2.0 INTRODUCTION

This final report covers the accomplishments of the 4-year period of the subject program. Three interim reports describing the accomplishments of each successive year of the program have been submitted.[1-3] Those results will be briefly summarized in this report, providing background for presentation of the accomplishments of the final year. The program consists of two parts: Part 1 is concerned with the influence of hydrogen on mechanical behavior of titanium alloys, Part II is a study of the effect of grain size on the mechanical properties and corrosion resistance of high strength Al alloys.

All commercial titanium alloys contain residual hydrogen, although the amounts may vary over a considerable range. The current specification placed on hydrogen in the widely used Ti-6Al-4V alloy is 150 ppm by weight maximum (Mil-T-9046, revision F) with typical concentrations running from 50 to 90 ppm. Mill products of other commercial alloy compositions contain residual hydrogen in similar quantities.

Generally, discussions of the effects of hydrogen on mechanical properties are split into two categories: effects caused by hydrogen already present in the material (either in solution or as hydrides) and those related to the interaction of hydrogen in the environment with the titanium alloy. In many cases, hydrogen from the environment is gradually taken into solution in the material and subsequently behaves in the same manner as internal hydrogen already present. In such cases, the environment acts simply as a source of additional hydrogen which increases the overall hydrogen content of the alloy. In other cases, external hydrogen can produce more localized effects. Such localized effects were not addressed in this study.

The hydrogen concentration of titanium alloys can be reduced by vacuum annealing, and the distribution of hydrogen within the alloy can be altered by suitable alloying additions. However, since a basic understanding of the effects of hydrogen on the behavior of complex alloys does not yet exist, it is unclear whether such measures would be desirable. Theoretical and experimental work conducted in Part I of this program (detailed later) has shown that internal hydrogen concentrations in the range of 100 to 150 ppm can have a significant effect on the mechanical behavior of α-Ti alloys. The effects are particularly pronounced in cases where relatively long range diffusion of hydrogen can occur during the course of a test, such as sustained-load cracking or hold-time effects in fatigue. In these cases, hydride formation at stress concentrations can lead to serious embrittlement even though the average hydrogen concentration is below that which is normally thought to cause embrittlement. Since these effects involve hydrogen diffusion in the lattice and hydride nucleation and growth, they are very temperature sensitive, an area which has been investigated in this program.

Part II of the program was designed to investigate the influence of grain size on the mechanical and corrosion properties of high strength aircraft aluminum alloys. Grain refinement is frequently used to enhance mechanical properties of structural materials. However, this method of improving properties has not yet been exploited in aluminum alloys on account of the difficulties encountered in refining the grain structure beyond the as-cast grain size. Attempts to refine the grain size by conventional mechanical working and recrystallization have generally resulted in grain growth to approximately the original grain size. Recent studies at the Science Center and work under this program have shown that a suitable combination of heat treatment and mechanical working steps can be used to substantially refine the grain size of precipitation hardening aluminum alloys. The process is based on a fundamental understanding of nucleation of grains at precipitate particles and is highly successful in reducing the grain size. Grain sizes studied in this program range from the large grain sizes obtained in conventional material (~300 μm) down to very fine grains approximately 10 μm in diameter.

In the past year, extensive testing of mechanical and corrosion properties has been performed to evaluate the potential benefits of grain refinement by this process. The results show that the room temperature mechanical properties are not substantially altered by grain refinement over the range achieved by the grain refining process developed at the Science Center. Exfoliation corrosion resistance is substantially improved by grain refinement, an important achievement which allows 7075 Al to be used in the peak strength condition in cases where exfoliation corrosion resistance is important. Interestingly, stress corrosion cracking (SCC) resistance of the T6 temper appears to be slightly degraded by grain refinement. SCC is generally not a problem with sheet material[4] which the thermomechanical treatment for grain refinement produces.

3.0 PART I - HYDROGEN IN ALPHA TITANIUM

3.1 Theory of Hydride Cracking

Despite improvements in processing techniques, all commercial titanium products contain residual hydrogen which can, under certain conditions, influence their mechanical behavior. The amount of residual hydrogen is, to some extent, controllable by processing but is frequently in the vicinity of 100 ppm by weight. This study has examined the circumstances under which such levels of hydrogen can affect crack growth in titanium structures. In particular, hydrogen tends to diffuse toward and concentrate at regions of large tensile stress. Depending upon temperature, hydrogen concentration, time, magnitude of the stress and solubility of hydrogen in the alloy, hydrogen may form a hydride which subsequently cracks. Hydride formation and cracking can cause crack growth in situations where it would normally not occur.

During the second year of this program, a mathematical model was developed for two-dimensional time-dependent hydrogen diffusion near a crack tip. Hydride formation was related to crack growth rate by assuming an initial hydride nucleus in the plastic zone and equating (in an average sense) the crack growth rate to the hydride growth rate. Numerical solutions to the resulting diffusion equations were performed and the theoretical crack growth rate studied as a function of time, temperature, and stress intensity. The details of the model and the ensuing calculations will not be presented in this report since they are discussed in detail in Ref. 5. A summary of the results is presented here.

Calculations were performed for the case where the stress intensity in 50 $MPa \cdot m^{1/2}$, and the hydrogen content 100 ppm in the Ti-6Al alloy. Numerical results for crack growth rate as a function of temperature are shown in Fig. 1, and the form of the curve showing a peak crack growth rate at around 0°C is significant. The physical explanation for the peak is clear. Crack growth rate is limited by the hydrogen diffusion rate at low temperatures and limited by the thermodynamic stability of the hydride at high temperatures.

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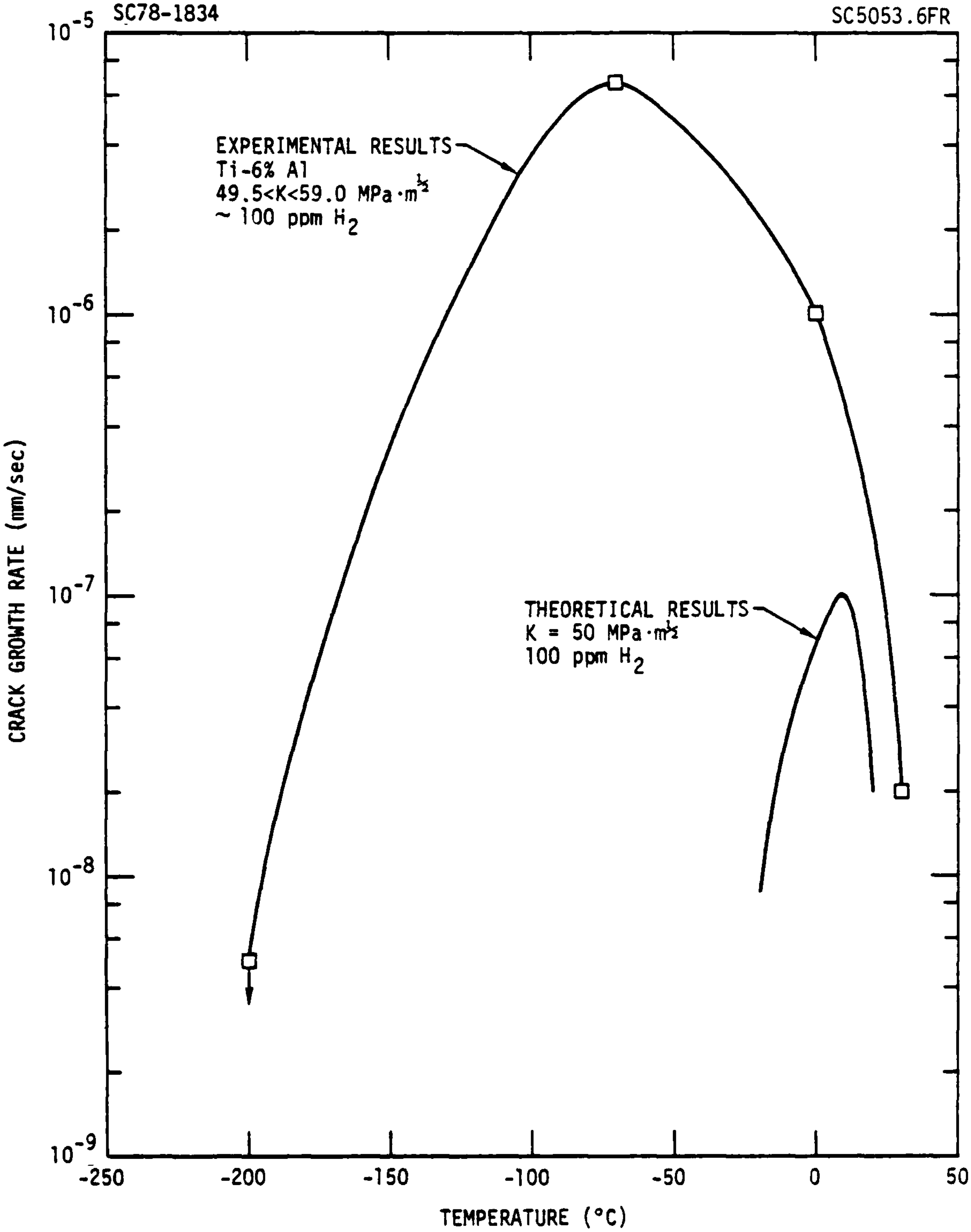

Fig. 1 Crack growth rate (da/dt) as a function of temperature for Ti-6Al.

Therefore, a maximum sustained load crack growth rate occurs at an intermediate temperature where the hydrogen diffusion rate is high enough to permit significant quantities of hydrogen to arrive at the region near the crack tip but where hydride precipitation is thermodynamically permitted.

Experimental results presented in the next section indicate that the theoretically predicted temperature of maximum crack growth rate is too high for a Ti-6Al alloy. That is, experiments seem to indicate a barrier to hydride growth not included in this model. It seems likely that this is related to nucleation. The model has assumed an irreversible but quasi-equilibrium transition rate from hydrogen in solution to hydrogen in hydride. It is clear that a complete theory must include the role of nucleation kinetics in addition to the effects of the elasto-plastic strain field on diffusion and solubility. Furthermore, a precise knowledge of the diffusivity of hydrogen in alpha titanium is lacking at present.

3.2 Sustained Load Cracking Results - Hydrogen in Alpha Titanium

The influence of hydrogen content, temperature, and specimen orientation on sustained-load cracking (SLC) in a Ti-6Al alloy has been investigated.[5] Compact tension specimens were used for the tests, oriented so that the crack path was either in the longitudinal or transverse direction. Hydrogen contents chosen were 4 and 100 ppm.

Experimental results included in Fig. 1 are for the crack growing in the longitudinal direction (TL orientation) in a specimen containing 100 ppm hydrogen. Cracking was not observed in transverse (LT) specimens, nor was it observed in specimens containing 4 ppm hydrogen for either orientation. It was reasoned that this resulted from a moderate basal transverse texture in the 12.7 mm plate used to make the samples. This type of texture places a large number of basal planes parallel to the crack plane in the longitudinal specimens. Since the basal plane is also the hydride habit plane in the Ti-6Al alloy,[6] this crack orientation is most susceptible to SLC.

The similarity between the experimental results and the theory in Fig. 1 is striking; both show a maximum in crack growth rate at a temperature below room temperature. As discussed previously, however, the temperature at which the maximum is observed is somewhat lower than that predicted by the theory.

3.3 Hold Time Effects in Fatigue - Hydrogen in Titanium

In a study of fatigue crack propagation in titanium alloys,[7,8] the combined effect of hold time (a 5 min hold at K_{max}) and internal hydrogen was found to produce small increases in fatigue crack growth rate (FCGR) at room temperature for Ti-6Al-4V containing 300 ppm hydrogen. Subsequent studies under the Rockwell IR&D program for Ti-6Al-4V containing 100 ppm showed that the FCGR for the Ti-6Al-4V containing 100 ppm hydrogen were further increased by lowering the temperature to -70°C, the peak growth rate temperature in the sustained load cracking study.

In an effort to improve our understanding of the hold-time effects in titanium alloys, a study of hold time-temperature effects in Ti-6Al containing 100 ppm hydrogen was conducted as part of the present program. Compact tension specimens were machined from the same 12.7 mm thick plate used for the SLC specimens; a majority of the testing reported here is for TL specimens, that is specimens in which the crack is propagating parallel to the longitudinal direction in the plate, the orientation found most susceptible to SLC in this alloy. Specimens were tested at both -40°C and -70°C at R = 0.3 using sinusoidal loading at 10 Hz. At several times during each test, a block of cycles with a 5 min hold at K_{max} was included.

The results for Ti-6Al in the TL orientation are shown in Fig. 2 and are tabulated in Table 1. In addition to the increase in da/dN caused by the 5 min hold, the corresponding average crack growth rate (da/dt) for the crack growth increment is given in Table 1 for comparison with da/dt data from sustained load cracking (SLC) tests.[5] Significant increases in crack growth rate (da/dN) was observed at both -40 and -70°C with the largest effect occurring

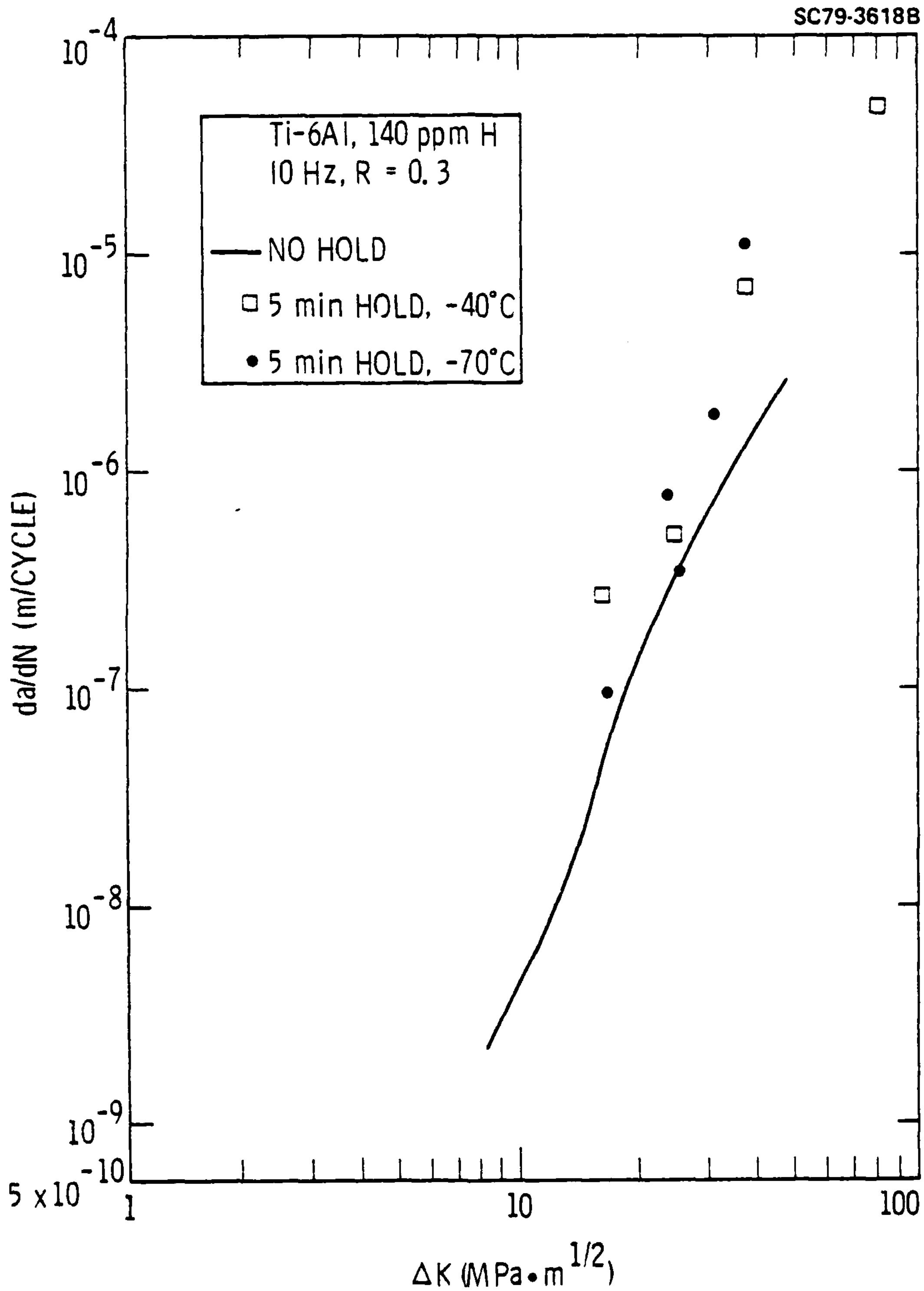

Fig. 2 Fatigue crack propagation results for Ti-6Al containing 140 ppm hydrogen tested at -40 and -70°C.

Table 1

Increase in Fatigue Crack Growth (da/dN) and Corresponding da/dt for Dwell Loading of Ti-6Al Containing 140 ppm Hydrogen

K_{max} ($MPa.m^{1/2}$)	da/dN Increase	da/dt (mm/s)
	-40°C	
22.5	6.5x	$9x10^{-7}$
33.8	2.0x	$2x10^{-6}$
52.4	7.2x	$2x10^{-3}$
64.0	>100x	$2x10^{-3}$
	-70°C	
23.5	2.7x	$3x10^{-7}$
32.7	3.3x	$3x10^{-6}$
34.0	1.2x	$1x10^{-6}$
42.7	3.6x	$6x10^{-6}$
51.8	13.7x	$4x10^{-5}$

during high K_{max} loading. It should be noted that the specimens used are the increasing ΔK, compact tension type and for long crack lengths, ΔK and K_{max} may be increasing quite rapidly resulting in non-conservative average ΔK and K_{max} values. At intermediate and high, no-hold, growth rates, that is growth rates at which striation formation is the major mode of crack propagation, the increase in crack growth rate with the hold at K_{max} is accompanied by a distinct fracture mode transition from striations to transgranular cleavage as shown in Fig. 3. No change in crack growth rate with hold was observed for specimens tested in the TL orientation a result consistent with SLC data for this material.[5]

These results show that, in the Ti-6Al alloy, increases in fatigue crack growth rate occur with a 5-minute hold at both -40 or -70°C. The conditions required for the observation of an increased growth rate are: (1) more than the base level hydrogen content, i.e. 100-300 ppm hydrogen (2) a

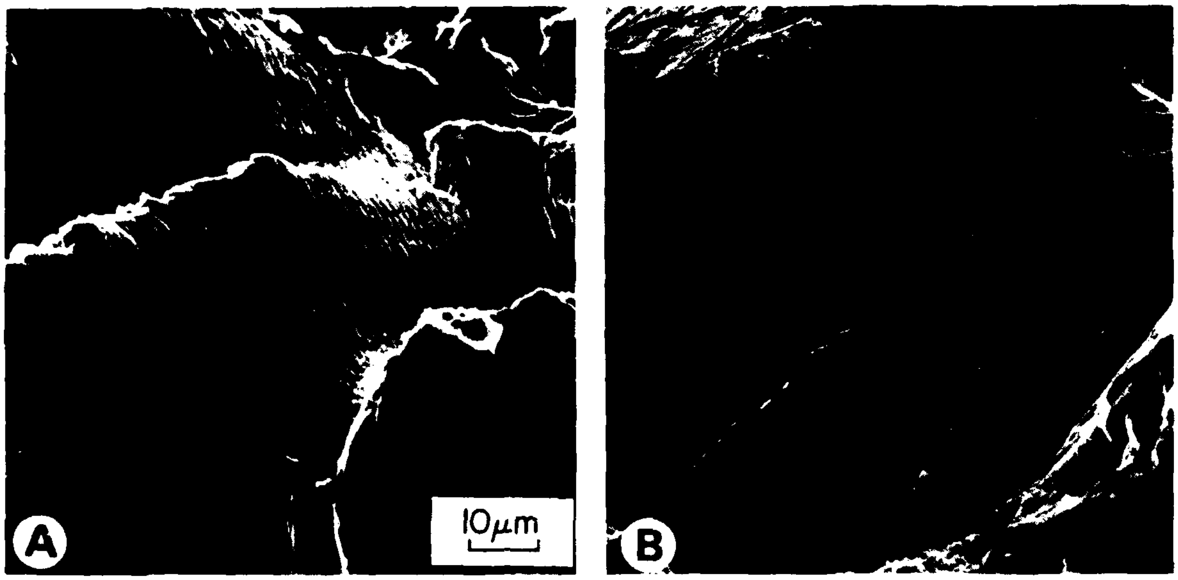

Fig. 3 Scanning electron micrograph of Ti-6Al containing 140 ppm hydrogen tested at -70°C. (a) no hold, (b) 5 minute hold at K_{max}.

temperature below room temperature, and (3) a significant hold-time in the tensile portion of the loading cycle. Without these three elements present simultaneously, increases in fatigue crack growth rate were not observed. For example, a sample containing 100 ppm hydrogen with a hold time at room temperature did not exhibit any acceleration in fatigue crack growth rate. An additional important observation is that of the fracture mode transition which occurs upon going from standard fatigue cycling to cycling with a hold time, as shown in Fig. 3. The transition from ductile striation formation in standard fatigue cycling to a quasi-cleavage brittle fracture with hold time appears to be consistent with a model suggesting hydride formation at the crack tip during the hold cycle. Calculations made recently by Pardee and Paton[5] suggest that a hold time of the order of one minute or more is sufficient on account of the high triaxiality, to drive a significant quantity of hydrogen to the crack tip, increasing the local hydrogen concentration. The high tensile stress and local deformation occurring at the crack tip aids in the nucleation of strain induced hydrides, and thus conditions required for hydride nucleation may exist at the crack tip even though the average hydrogen concentration is insufficient for hydride formation. This process was known to be greatly favored at temperatures somewhat below room temperature in the calculations presented in Ref. 5; temperatures of the order of -70°C were found to provide conditions for maximum crack growth rate in sustained load cracking experiments.

Crack growth rates shown in Table 1 are comparable to those calculated in Ref. 5, suggesting that sustained load cracking during the tensile hold is the dominant mechanism. This idea is supported by similarities in the fracture surfaces produced by tensile hold periods in the fatigue experiments and by SLC experiments. The fracture mode produced during a tensile hold period in FCP tests is shown in Fig. 4a and can be compared with the fracture produced by SLC in Fig. 4b. The similarity of fracture mode in these two cases is striking and contrasts sharply with the more ductile fracture produced by standard fatigue cycling.

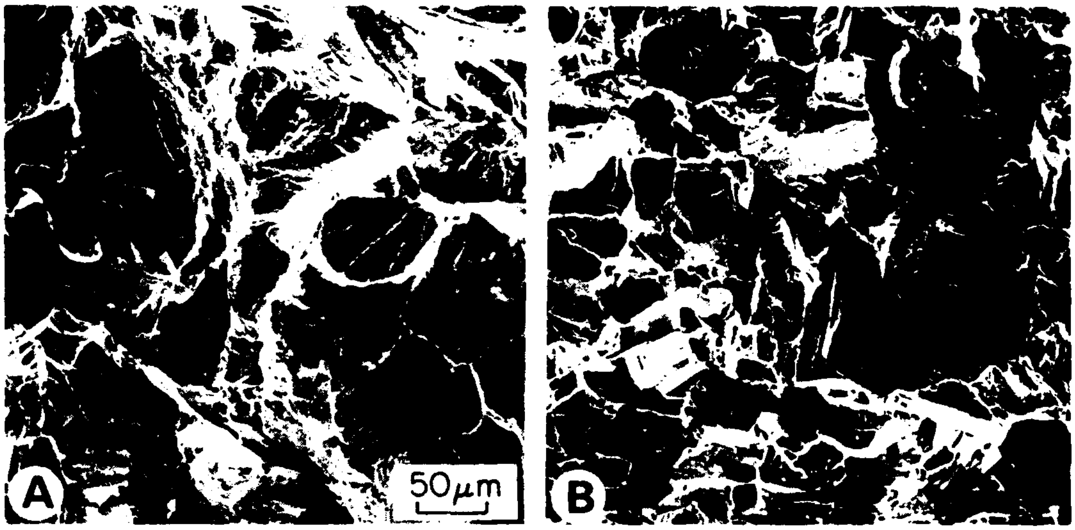

Fig. 4 Fracture surface of Ti-6Al containing approximately 100 ppm hydrogen tested at -70°C. (a) 5 min hold at Kmax (da/dt = 6×10^{-6} mm/s, Kmax = 42.7 mPa.$m^{\frac{1}{2}}$), (b) SLC (da/dt = 7×10^{-6} mm/s, Kmax = 49.4 mPa.$m^{\frac{1}{2}}$).

It is suggested, that the hold time during the tensile part of the fatigue cycle provides the right conditions for diffusion of hydrogen to the crack tip and subsequent hydride nucleation, thus causing the quasi-cleavage type fracture as a result of the hydride formation. This process is aided by high hydrogen contents, long tensile hold times and temperatures below room temperature, as found experimentally in the present results.

These results are important in that under conditions where significant accelerations were observed, titanium alloys can sometimes be found in critical structural components. The -40° to -70°C temperature regime is that which can be encountered by aircraft flying at moderately high altitudes. Hydrogen contents of the order of 100 to 150 ppm are frequently encountered in titanium alloys after conventional processing, and structures which are subjected to a tensile hold in their normal loading sequence are also frequently encountered. It is worthwhile noting, however, that if any of these factors are removed, no fatigue crack acceleration was observed in the materials investigated here. Therefore, if structures are suspected to be operating under the conditions which encourage a hold time effect, vacuum annealing to remove the hydrogen to levels of the order of 10 to 20 ppm should be successful in alleviating any of the harmful effects discussed here.

In conclusion, it has been found that accelerations in fatigue crack growth rate were observed in Ti-6Al containing 100 to 300 ppm hydrogen with a 5-minute tensile hold conditions. Acceleration was increased at -40° and -70°C, but was less than a factor of 2 at room temperature.

4.0 PART II - FINE GRAIN ALUMINUM

4.1 Background

Potential benefits of grain refinement include improved mechanical properties and corrosion resistance. Furthermore, if the grain size is sufficiently small, materials may become superplastic during high temperature deformation at slow strain rates. A thermomechanical processing technique for producing small grain sizes in 7075 Al has been developed at the Science Center, as discussed in the Interim Reports[2,3] for this program. Interpretation of the results of the present study requires understanding the thermomechanical process used to refine the grain size. The processing technique is briefly described in the following paragraphs.

To obtain small grain sizes, solution treated 7075Al is processed through the following three-step procedure:

1. An aging treatment to develop a uniformly distributed precipitate phase intended to produce uniform nucleation.

2. Warm reduction by rolling performed for the purpose of introducing a large number of recrystallization nuclei.

3. Recrystallization to produce a stable, small grain size.

Heat treatments may be performed subsequently to attain the standard T-6 or T-73 heat treatment conditions. This procedure has resulted in grain sizes on the order of 10 μm, (Fig. 5b), in contrast to commercial material with longitudinal grain dimensions typically around 300 μm and short transverse grain dimensions of approximately 25 μm (Fig. 5a). The heavy reduction required for the production of fine grain 7075 substantially alters the inclusion distribution, in addition to changing the grain size. Therefore, changes in properties between fine grain and commercial aluminum are not necessarily

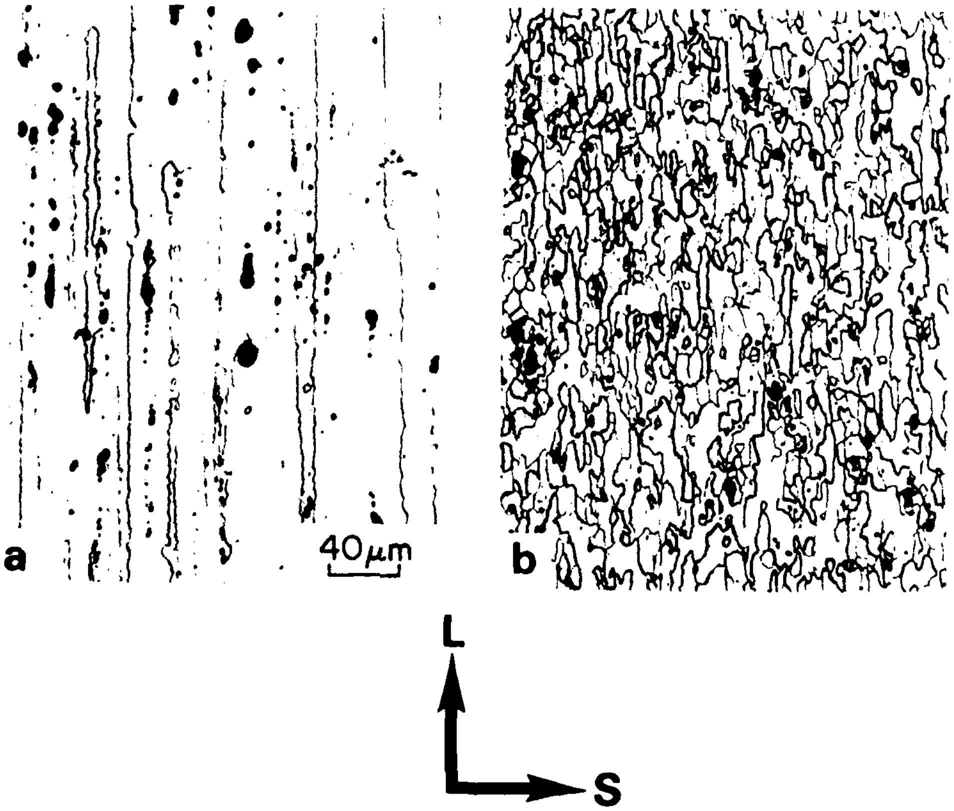

Fig. 5 Commercial 7075 Al plate (a) and fine grain material (b). L indicates longitudinal direction and S indicates short transverse direction.

entirely caused by the grain size difference. For a proper study of the effect of grain size on the mechanical and corrosion properties of aluminum alloys, specimens with different grain sizes but the same inclusion distribution (equivalent reduction) were required.

A method of producing such specimens has been developed under a concurrent IR&D program at the Science Center.[9] In this procedure, all specimens are reduced approximately 90% following overaging. Therefore, all specimens have equivalent inclusion distribuions. Different grain sizes are produced by subsequent heat treatments. Fine grain sizes (~10 μm) are produced by the normal recrystallization procedure outlined above. To produce coarser grain sizes, samples are isothermally annealed at various temperatures for 1 hour prior to recrystallization. The purpose of the annealing (or recovery) treatment is to deactivate some of the potential nucleation sites for new grains which were created by the earlier reduction. The final grain size following the recovery plus recrystallization experiments depends on the number of nucleation sites removed by the recovery treatment since all specimens initially contained the same density of nucleation sites. Note that any changes in precipitate distribution which occur during recovery are removed by the subsequent recrystallization (solution treatment) step. Therefore, specimens of various grain sizes but equivalent in all other respects can be produced by choosing the proper annealing temperature. Slow heating from 300°C to 400°C at a rate of around 6°C/hr was substituted for the isothermal anneal as a method for producing the coarsest grain size.

Following the recrystallization step, specimens were water quenched. During recrystallization, particles precipitated during previous aging and annealing steps were dissolved since the alloy was recrystallized at the solution treatment temperature. Thus, after the thermomechanical processing sequence to produce fine grain sizes, any of the standard aging conditions used for 7075 could be produced. The times and temperatures used in this study are listed in Table 2.

Table 2
Aging Conditions for 7075 Aluminum

Temper	Time	Temperature
T6, T651*	24 hr	121°C
T73, T7351*	4 hr	121°C
	+ 28 hr	163°C

*TX51 tempers were plastically strained 1.5% in tension prior to aging

Several grain sizes have been selected for studies of the effect of grain size on the mechanical and corrosion properties of 7075 Al. They are shown in Figs. 6 to 8. The data for these microstructures are shown in Table 3. A number of mechanical and corrosion properties have been evaluated as a function of grain size in otherwise metallurgically equivalent specimens. The majority of properties have been evaluated over the past year so that the results reported in the following sections constitute progress during the final year of the contract period.

Table 3
Grain Size Data for 7075 Al

Grain Size Designation	Average Longitudinal Intercept Length μm	Average Short Transverse Intercept Length μm
Fine	10	7
Intermediate	40	11
Coarse	300	30

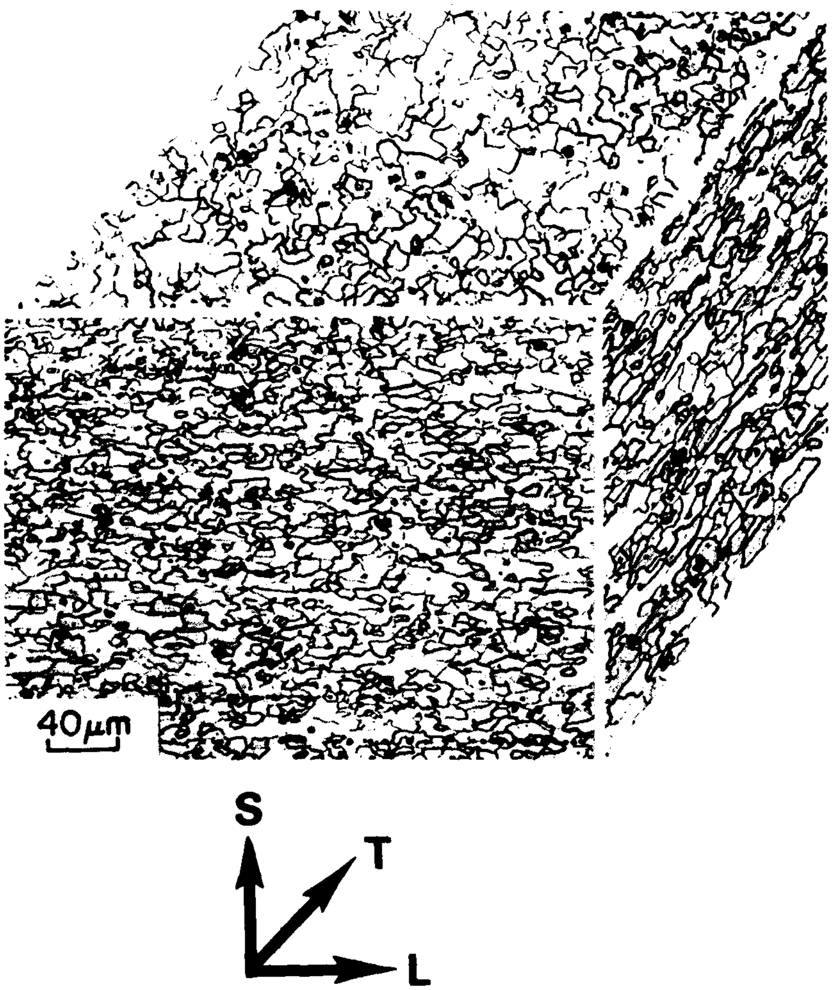

Fig. 7 Fine grain size. L indicates longitudinal direction. T indicates long transverse direction and S indicates short transverse direction.

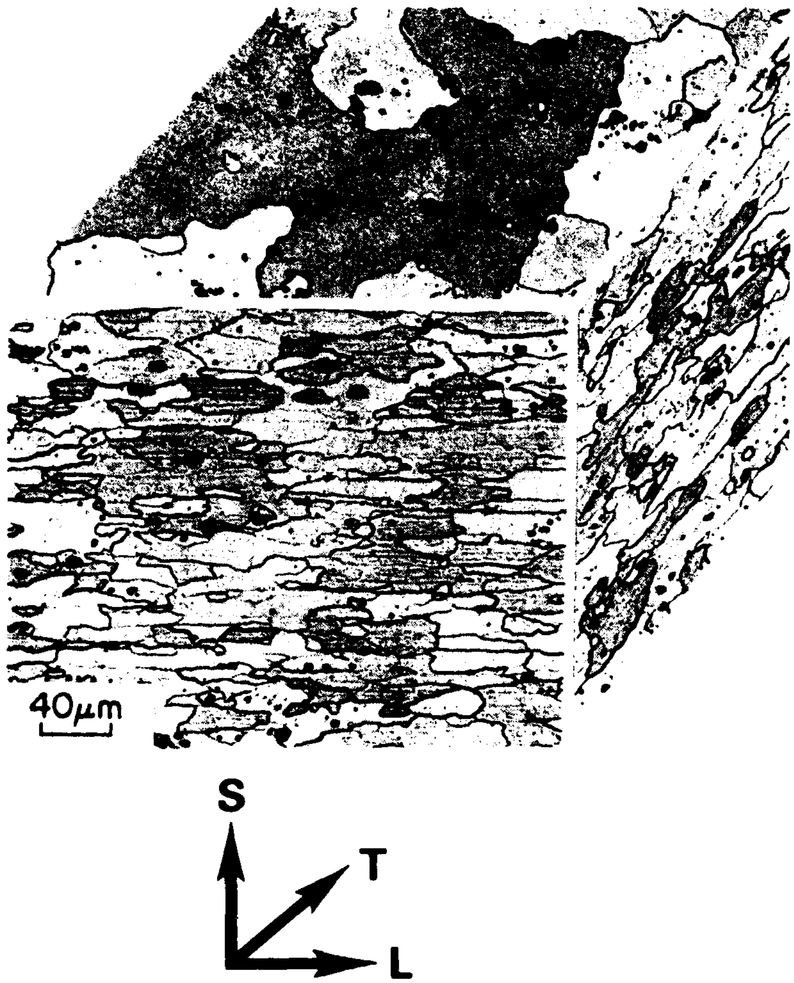

Fig. 8 Intermediate grain size. L. Indicates longitudinal direction. T indicates long transvere direction and S indicates short transverse direction.

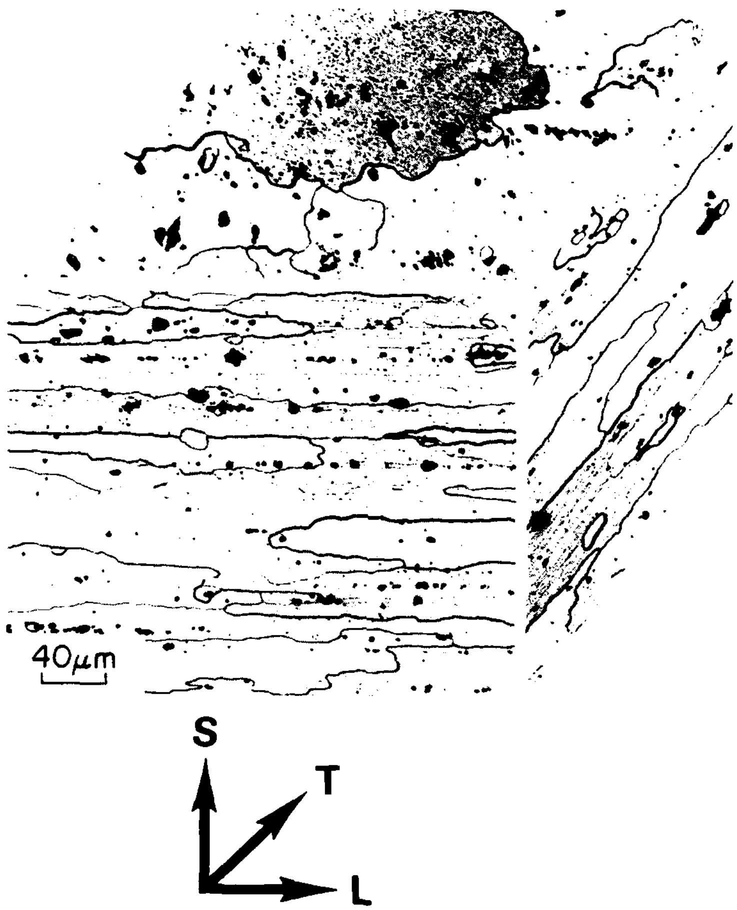

Fig. 9 Coarse grain size. L indicates longitudinal direction. T. indicates long transverse direction and S indicates short transverse direction.

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4.2 Results

Tensile Properties

Tensile properties of 7075 Al have been measured for coarse, intermediate and fine grain sizes listed in Table 3. Two tensile specimens of rectangular cross section were prepared and tested for each grain size in the T6, T651, T73 and T7351 aging conditions. The stress axis was parallel to the rolling direction for all specimens. The results of these tests are shown in Figs. 9 and 10 for the T6 and T651 aging conditions. For the material aged to peak hardness (T6), there is a slight increase in yield strength, ultimate strength and true fracture strength as the grain size is reduced. The trend toward increased strength is accompanied by a small increase in reduction of area (RA) or, equivalently, fracture ductility for finer grain sizes.

For the T6 aging condition, the weak trends for strength and ductility as functions of grain size are only slightly greater than the scatter in experimental data. For the T73 and T7351 aging conditions, no trends could be distinguished outside of the normal experimental scatter. In the overaged condition, the tensile properties appeared to be independent of grain size over the range of grain sizes studied in this program.

The results obtained in this study of tensile properties as a function of grain size are consistent with currently accepted ideas about the influence grain size on tensile properties.[11,12] The increase in strength resulting from grain size reduction has been well documented for a wide variety of materials. It is probable that the larger effects in the T6 (peak hardness) condition are a result of the difference in slip character between the peak hardness and overaged conditions.[10,12] Grain size is generally believed to affect strength by influencing the length of dislocation pile-ups which occur before slip propagates across grain boundaries. Formation of intense discrete slip bands during deformation of the T6 specimens occurs because planar slip is encouraged by the coherent precipitates present after aging to the peak strength condition. This favors formation of long dislocation pile-ups which then propagate across the boundaries as described above.

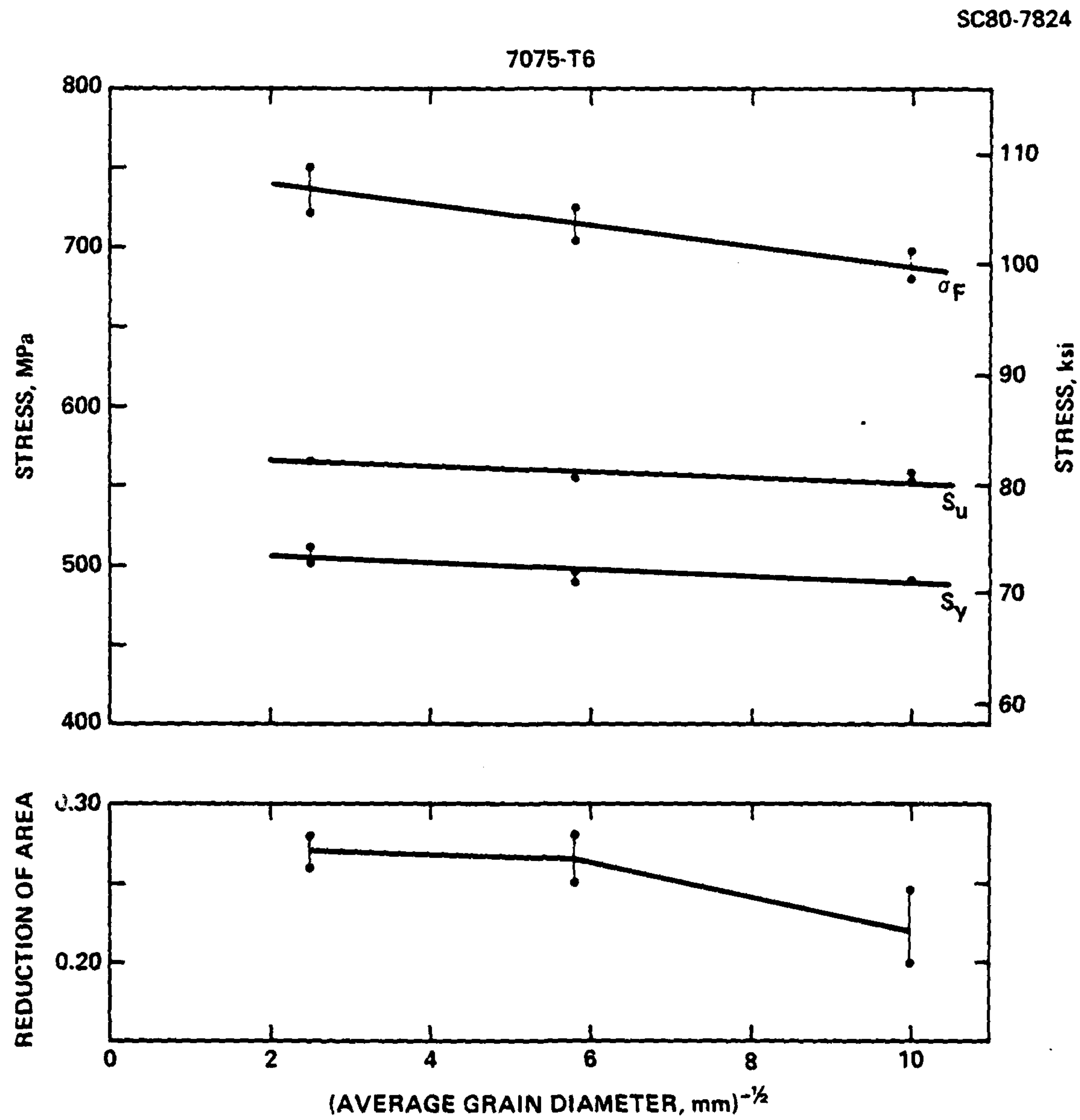

Fig. 9 Tensile properties for 7075-T6 as a function of grain size. Sy and Su are engineering yield and ultimate strengths and σ_F is the true fracture strength.

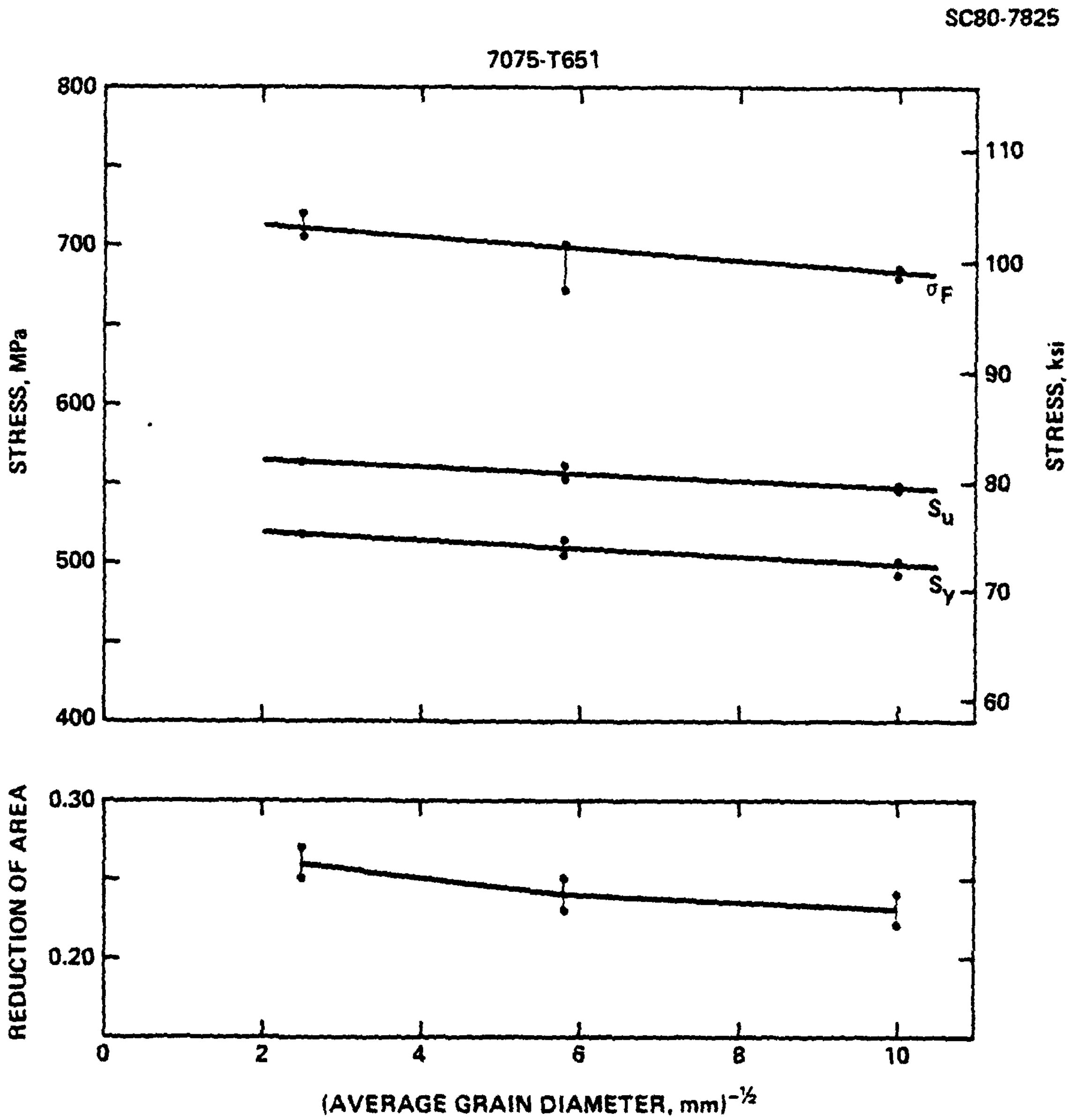

Fig. 10 Tensile properties for 7075-T651 as a function of grain size. Sy and Su are engineering yield and ultimate strengths and σ_F is the true fracture strength.

The more homogeneous slip which occurs in the overaged specimens with semi-coherent or incoherent strengthening precipitates will discourage dislocation pile-up formation and grain size should have a smaller effect than in the peak hardness condition. The experimental results are consistent with these ideas.

The conclusions of the study of tensile properties as a function of grain size in 7075 Al are:

1. In the T6 aging condition, there are small increases in yield, ultimate and fracture strengths and the reduction of area for small grain sizes compared with commercial grain size.

2. In the T73 aging condition, there are no measurable changes in the tensile properties over the range of grain sizes investigated in this study.

Fatigue Life

The fatigue life properties of several materials have been shown to depend on grain size.[13] Furthermore, models developed at the Science center predict that crack initiation during fatigue at low strain amplitudes is grain size dependent.[14] Low cycle fatigue life, where significant plastic strains occur on each cycle, is known to be increased by increasing fracture ductility.[15] Since the tensile results showed that fracture ductility increased for the fine grain sizes, low cycle fatigue life is expected to be slightly increased for the fine grain size.

The fatigue life of 7075 Al has been measured in this study for coarse and fine grain sizes in the T6 aging condition. The T73 aging condition was not evaluated because the tensile results showed the peak strength condition to be most sensitive to grain size.

Cantilever beam fatigue specimens were tested at constant strain amplitude with fully reversed loading. The stress axis was parallel to the rolling direction. The specimens were enclosed in an environmental chamber and tests were carried out in dry N_2. This eliminated effects due to changes in relative humidity, which are known to affect fatigue properties of aluminum alloys.[16]

Results of these tests are shown in Fig. 11. The test results demonstrate that grain size does not have an appreciable effect on fatigue life over the range of grain sizes investigated in this study. There is a substantial scatter in the data which is typical of fatigue life results. A large amount of additional testing would have been required to increase confidence in the fatigue life at particular strain amplitudes. However, it is clear from the limited test results shown in Fig. 11 that grain size does not have an appreciable effect on fatigue life and further testing would not change that conclusion.

The absence of a substantial grain size effect on fatigue life is surprizing in view of the slightly increased fracture ductility for small grain sizes. Increased fracture ductility would be expected to increase fatigue life at high strain amplitudes.[15] It seems likely that a grain size effect was not observed in the fatigue life studies because the effect was too small to be revealed in the scattered results. The lack of a significant grain size effect in the present investigation conflicts with results obtained for 7075-T7351 in smooth-bar axial fatigue tests.[9] Grain refinement was found to significantly increase fatigue life at low strain amplitudes (life $>10^4$ cycles) in the axial fatigue tests. The only difference was the aging condition: T6 in the present study vs T7351 in the axial tests.

Fatigue Crack Propagation

The effect of grain size on fatigue crack propagation rates has been found to be small in many materials at intermediate growth rates.[13,17] However, in ferritic steel, grain refinement has been found to decrease threshold crack propagation rates.[18] Fatigue crack propagation (fcp) rates were measured for coarse and fine grain sizes in the T6 aging condition to assess the effect of grain size on fcp in 7075 aluminum.

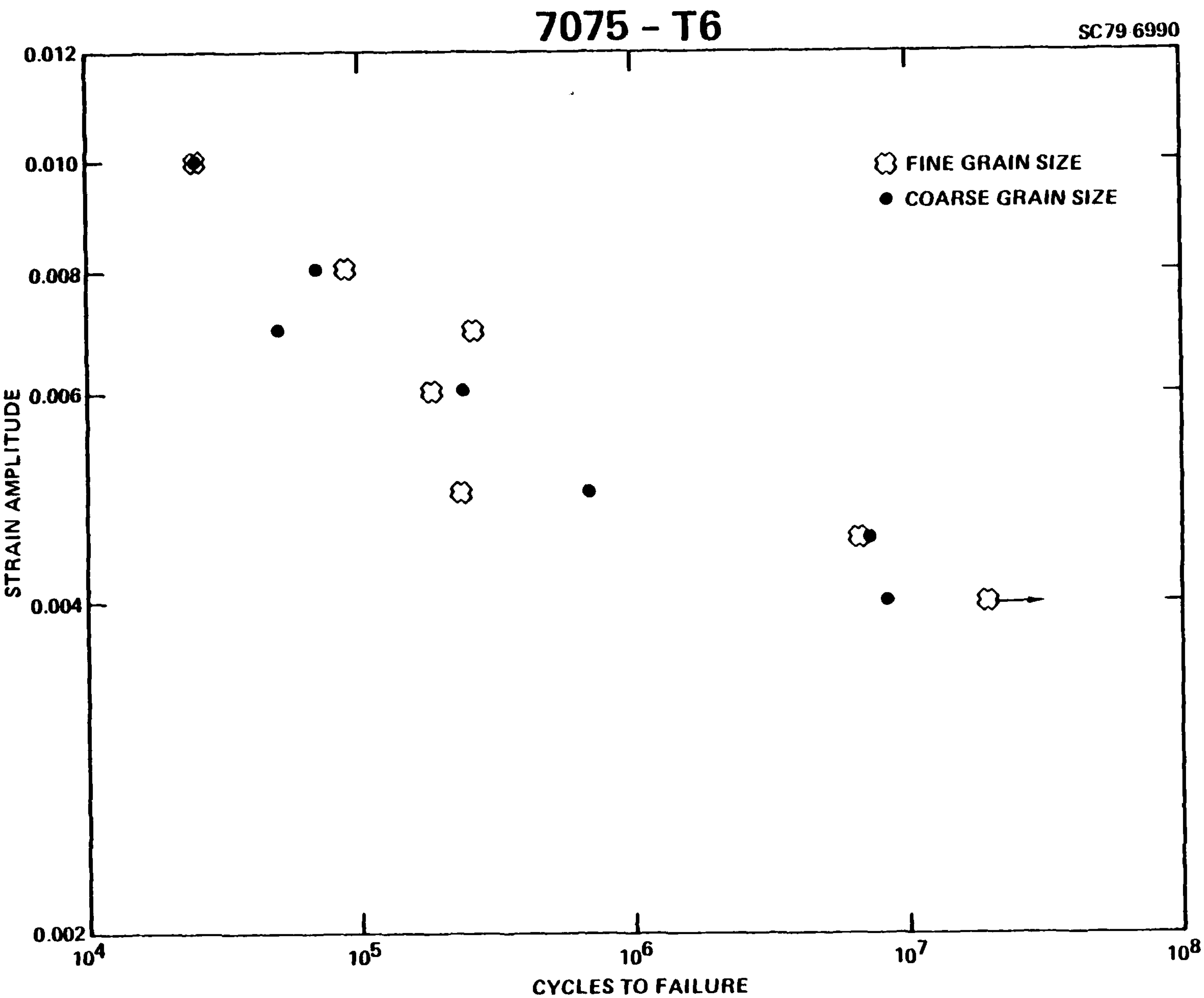

Fig. 11 Fatigue life of 7075-T6 for fine and coarse grain sizes.

Compact tension specimens 0.25 inches thick were used to measure fcp rates as a function of grain size, the specimen orientation was LT in all cases. Results of these tests are shown in Fig. 12. Fatigue crack growth rates were found to be slightly lowered by grain refinement. The threshold $\Delta K(\Delta K_{th})$ was found to be about 1 $MPa \cdot m^{1/2}$ lower for the coarse grain material. Near threshold, the steep slope of the curves converts the 1 $MPa \cdot m^{1/2}$ difference between the curves into nearly an order of magnitude difference in crack growth rate at the same ΔK. In the intermediate growth rate regime, the difference in crack growth rate is less than a factor of two for the same ΔK value. It should be emphasized that these results were obtained from one specimen each of coarse and fine grain material. The scatter bands obtained by testing more specimens for each grain size would probably overlap to some extent.

This result is surprising in view of the large grain size effect on ΔK_{th} found in ferritic steels.[18] Reducing the grain size of steel over the same range used in this study would have decreased ΔK_{th} by approximately 10 $ksi \cdot in^{1/2}$, according to the analysis of Masonnaur and Bailon.[18] The origin of this effect is unknown. It should be noted that the effect of grain size on yield strength of the ferritic steel was also much larger than the effect of grain size on yield strength of 7075 Al. It appears that mechanical properties of aluminum alloys may be much less sensitive to grain size changes than steels. Possible explanations for this difference may originate from the different slip systems for fcc and bcc crystals.

Exfoliation Corrosion Resistance of Fine Grain 7075 Al

Exfoliation corrosion tests have been carried out over the range of grain sizes. Exfoliation corrosion occurs in the absence of applied stress by the uplifting of surface layers owing to intergranular attack parallel to and just below the surface. The EXCO accelerated exfoliation corrosion test has been developed by ASTM for testing resistance to exfoliation corrosion.[19] The EXCO test involves total immersion of specimen coupons in a solution containing NaCl, KNO_3 and HNO_3. Following a 48 hour immersion, the corrosion products

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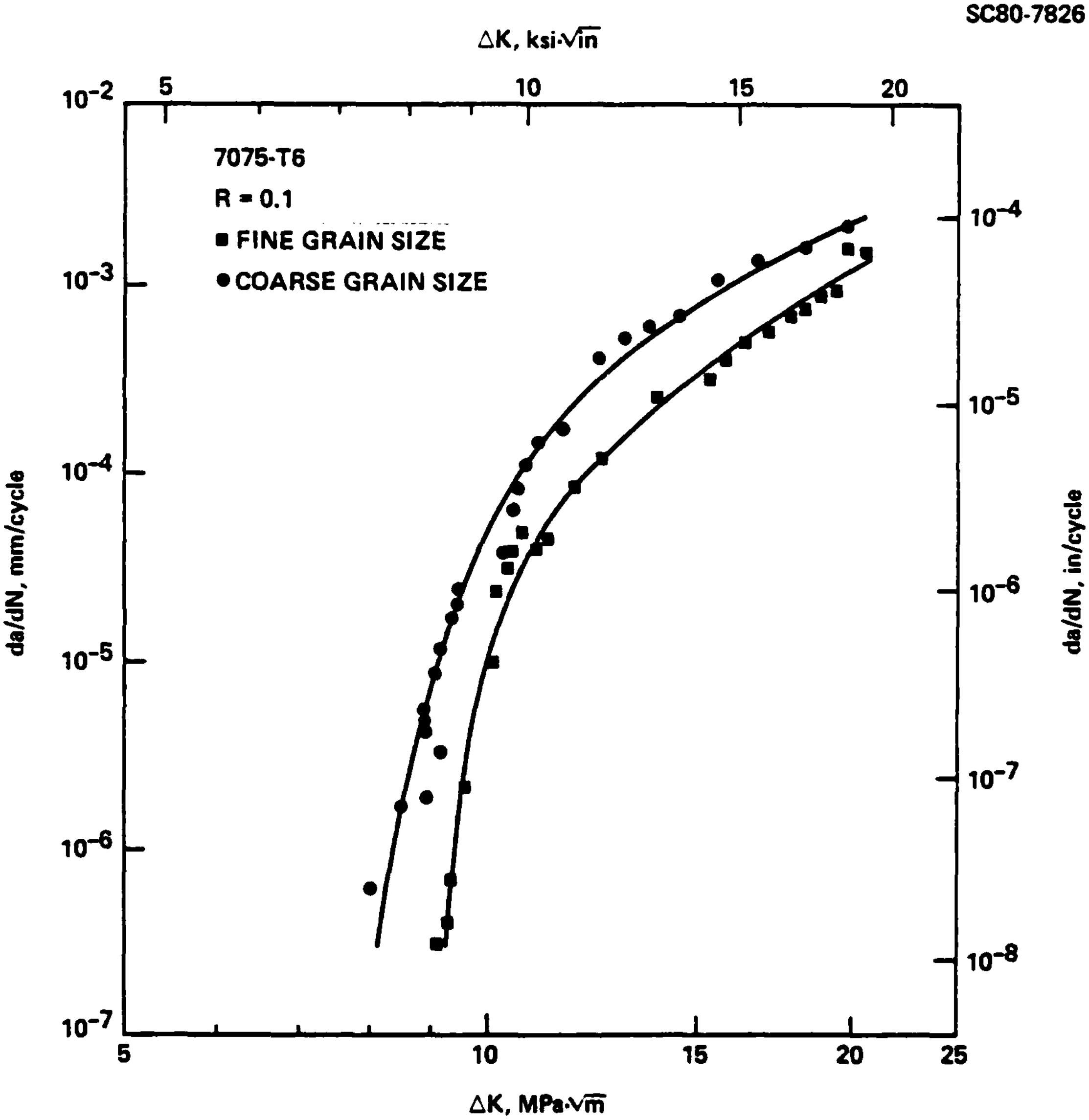

Fig. 12 Fatigue crack propagation rate vs ΔK in 7075-T6 for fine and coarse grain sizes.

are cleaned from the specimen surfaces and the corrosion resistance of each specimen is determined by comparison with photographs of standard specimens. The results of this experiment appear in Table 4. Although not part of the standard test procedure, the thickness of some of the specimens was determined (in an average sense) before and after the test. These results are shown in Fig. 13. Clearly, from these results, the exfoliation resistance of the fine grain material is superior to that of the coarse grain material. Not only was the surface appearance smoother in the fine grain specimens but the loss of material from the surface by complete exfoliation was substantially reduced for all aging conditions.

Table 4
Grain Size Dependence of Exfoliation Corrosion Resistance

Grain Size	Temper		
	T6	T6 + 4 hr 160°C	T6 + 8 hr 160°C
Fine	P	P	P
Intermediate	ED	EC	P
Coarse	ED	EC	EA

Designation: P = pitting only
E = exfoliation
A to D = increasing severity of exfoliation corrosion
(A - most resistant, D - least resistant)

The cross-sectional appearance of the specimens aged to the T6 condition is shown in Fig. 14. Note the total absence of the undermining and uplifting attack in the fine grain specimen, A, where as the coarse grain specimen, C, was extremely susceptible to this type of attack. This is a direct consequence of the grain size difference since all specimens were aged to the same extent.

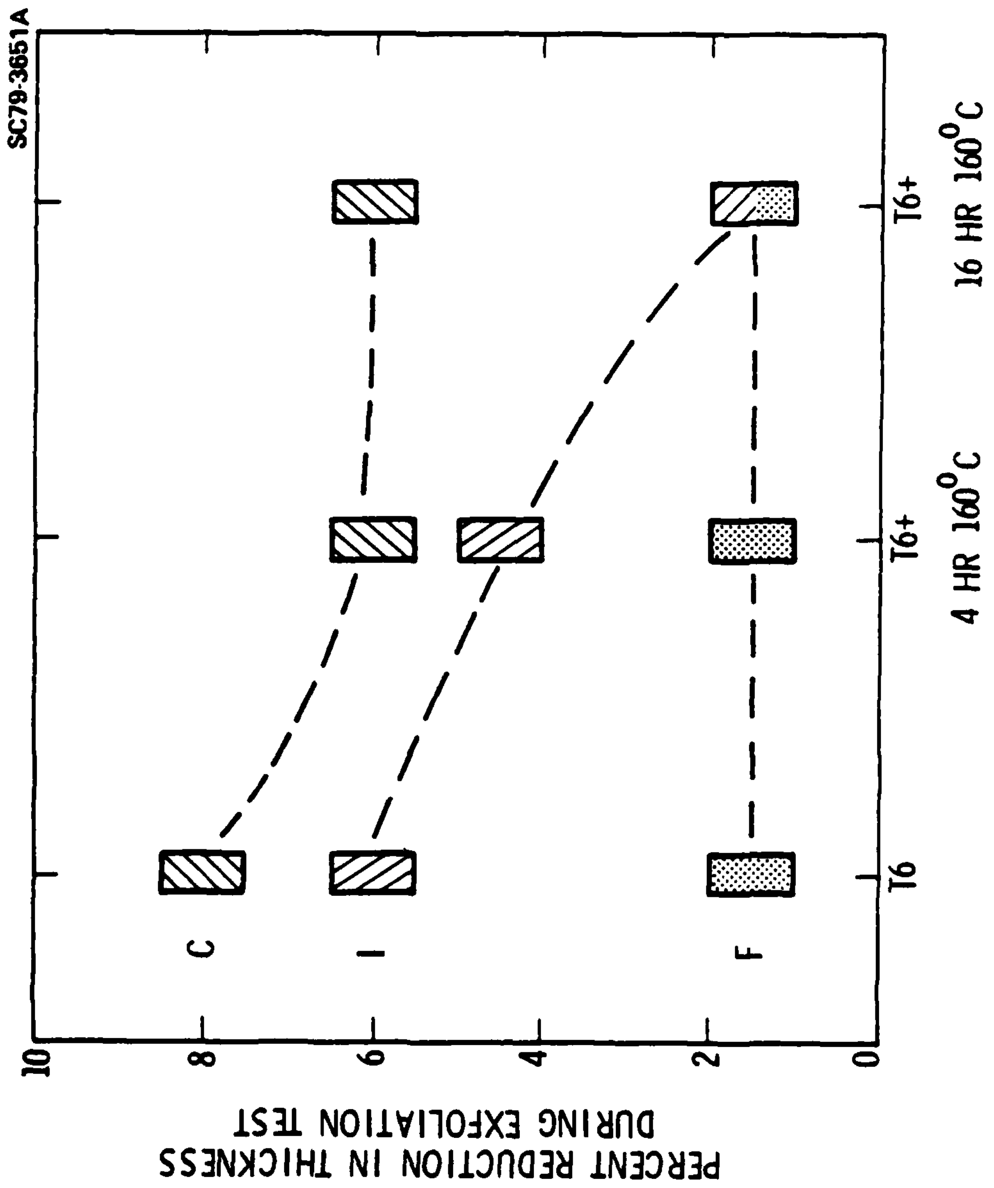

Fig. 13 Thickness change during EXCO test for fine (F), intermediate (I) and coarse (C) grain size material.

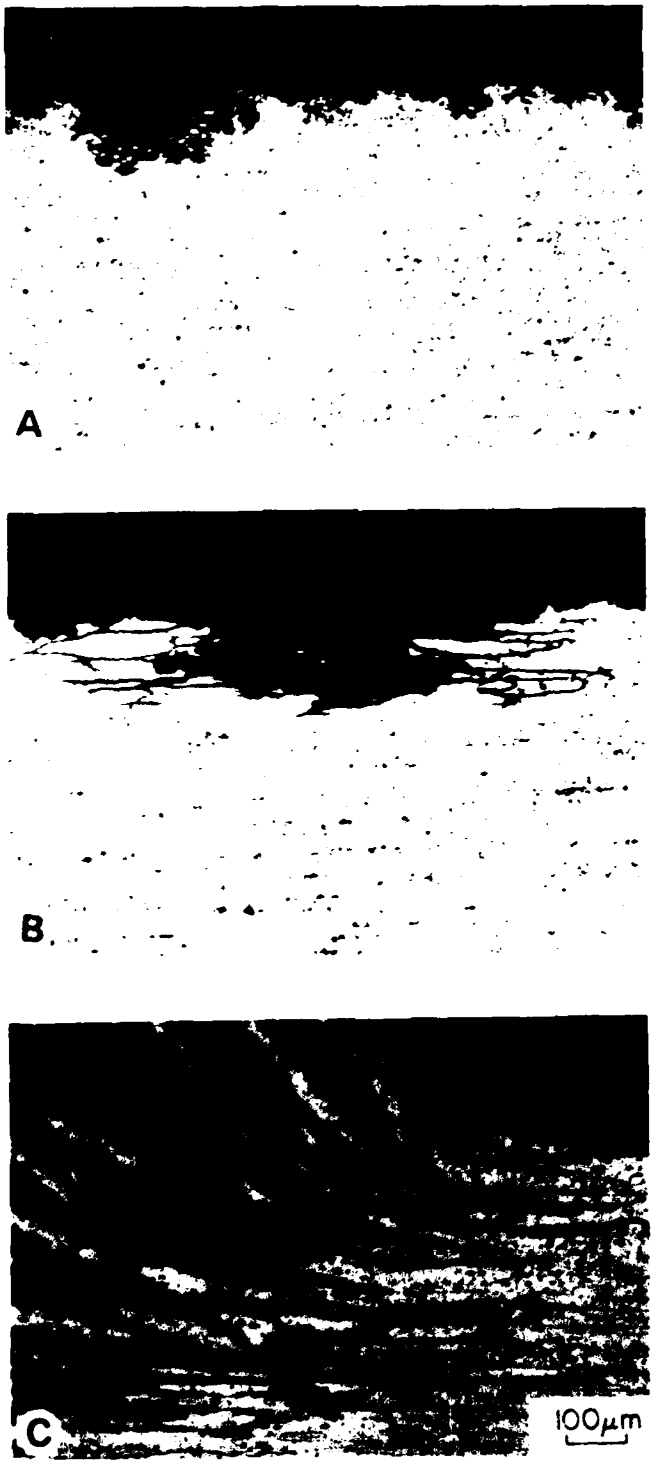

Fig. 9 Cross-section of exfoliation corrosion specimens aged to T6 + 4 hours 160°C condition.

The implications of this finding extend beyond the high exfoliation corrosion resistance as such. In applications requiring high resistance to exfoliation corrosion, commercial 7075 is overaged to the T76 or T73 aging conditions, which causes a decrease in the yield strength of 5 to 10 ksi. Thus, in current commercial practice, exfoliation resistance is acquired at the expense of yield strength. For the fine grain material, however, no overaging is required to achieve high exfoliation corrosion resistance. Therefore, material requiring resistance to exfoliation corrosion can be used in the peak strength T6 condition. Although the fine grain processing technique does not directly result in a substantial gain of yield strength, exfoliation corrosion resistant material can be used in a higher strength condition when the grain size is fine.

Stress Corrosion Cracking

Stress corrosion cracking (SCC) of high strength aluminum alloys occurs principally in commercial plate material.[4] The grain refinement process developed at the Science Center is well suited to production of sheet material but is not particularly suitable for thick plate manufacture; it is difficult to achieve the required 90% warm reduction in plate material. SCC of the fine grain 7075 is unlikely to occur in most applications simply because of the product form. In order to measure SCC resistance as a function of grain size, a special plate of grain refined material was prepared. The reduction was limited to 70% so that the finest grain size which could be produced was approximately 20 µm in the longitudinal direction and 10 µm in the short transverse direction. For the SCC experiments, this was termed "fine grain size" and was compared to material with the coarse grain size listed in Table 2.

Bolt loaded double cantilever beam (DCB) specimens of SL orientation were employed to determine SCC resistance. The DCB specimens were 0.6 inches square by 4 inches long. The first specimens tested were not side grooved and attempts were made to establish the relation between K_I and crack growth rate, using the method of Finnegan and Hartt.[20] After loading to pop-in, specimens were immersed in a 3.5% NaCl solution and crack length was measured periodically. These experiments were unsuccessful because the cracks in the fine grain specimens grew out of the side of the specimens. Subsequently, specimens were side grooved, which made crack length measurements unreliable. Companion side grooved specimens were immersed for equal lengths of time in the 3.5% NaCl solution and broken open immediately after removal. This technique at least provided means for comparing SCC resistance of coarse and fine grain 7075, although the more useful K_I vs crack growth rate curves could not be established by this technique.

Results on one pair of companion specimens are shown in Fig. 15. The pre-crack, SCC and overload regions are labelled on each photograph. The average growth rate of the stress corrosion crack in the fine grain specimen was 6.0×10^{-5} mm/s as compared with 3.9×10^{-5} mm/s in the coarse grain material. Duplicate specimen pairs have yielded very similar results. These crack growth rates are somewhat higher than those reported by Sprowls, Coursen and Walsh[21] for 7075-T6. However, that is not suprising since the duration of the tests performed by Sprowls, Coursen and Walsh was 360 hours compared to 120 hours in the present investigation. The crack growth rate decreases with time during testing of bolt-loaded DCB specimens so the longer tests give slower average growth rates.

In an effort to determine whether the SCC mechanism was the same in the coarse and fine grain material, companion specimens were sectioned for examination of the crack path. The results are shown in Fig. 16. The fine grain specimen was one in which the crack was growing out of the rolling plane which explains the average crack direction in that micrograph. In both cases, the crack is clearly intergranular in nature, as found by many investigators for SCC in 7075.[22]

The conclusion of these tests is that the SCC resistance of the fine grain 7075 is slightly lower than the coarse, commercial-type grain structure in the T6 aging condition. No data were obtained for the overaged conditions

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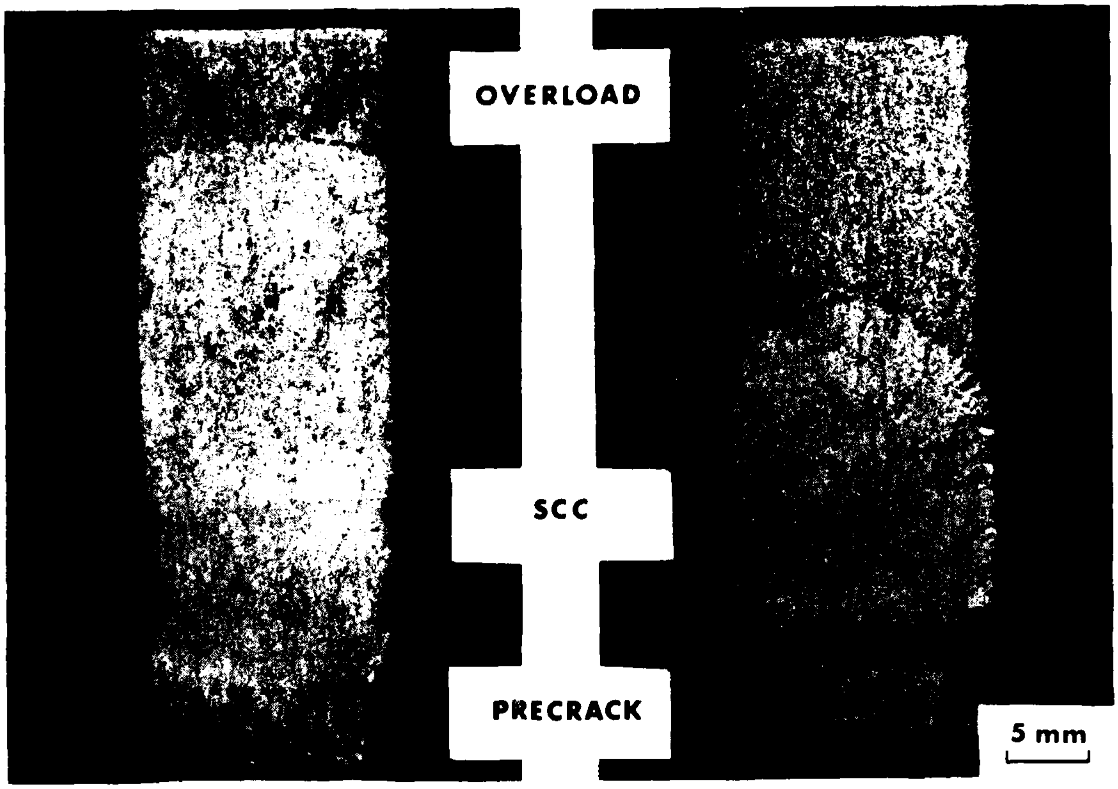
7075 - T6



FINE COARSE

3.5% NaCl SOLUTION
120 HOUR IMMERSION

Fig. 15 Stress corrosion cracking specimens after 120 hour immersion in 3.5% NaCl solution. Precrack, SCC and overload regions are indicated.

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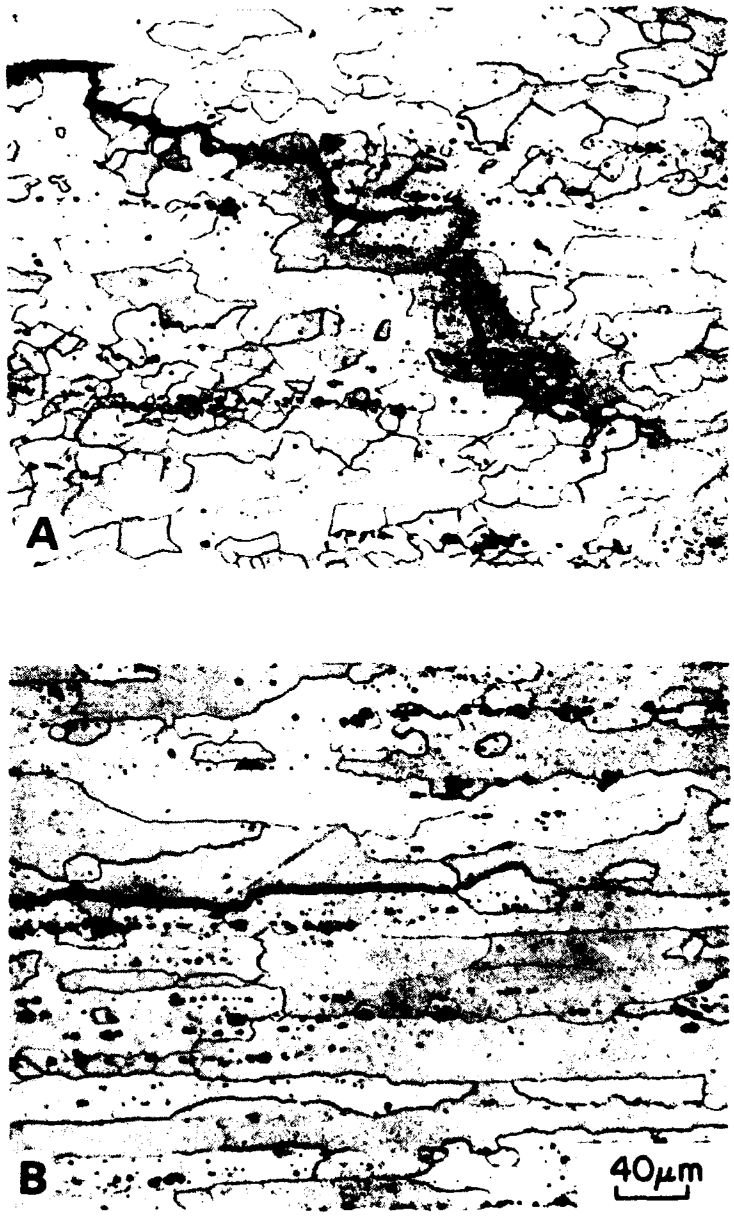

Fig. 16 Cross-section of crack tip region in (a) fine grain and (b) coarse grain SCC specimens. Cracking is intergranular in both cases.

which markedly improve SCC resistance in 7000 series alloys.[4,22,23] It is important to consider this result in the proper context: the stress corrosion crack growth rate in the fine grain material is less than a factor of two times the crack growth rate in the coarse grain microstructure. This variation is considerably smaller than the heat-to-heat variations found by Hyatt[23] for a variety of alloys and aging conditions. Furthermore, the less-than-two-times increase in SCC growth rate attributed to grain refinement is small compared to the order of magnitude changes caused by changing the aging conditions. Small changes in the aging kinetics of the grain refined 7075 which could not be detected by mechanical property measurements may be responsible for the slight difference in stress corrosion cracking rates.

The slightly more rapid crack growth rate in the grain refined material can be compared with the results obtained by Waldman, Sulinski and Markus[24] (WSM), who studied the influence of intermediate thermomechanical treatments (ITMT) in properties of 7075. The maximum grain refinement produced by the ITMT processes resulted in a grain size approximately equal to the intermediate grain size shown in Fig. 7. WSM evaluated SCC resistance using C-ring specimens. Their test results showed that some processing sequences improved SCC resistance while others produced lower SCC resistance. In addition to grain size, however, different ITMTs produced different dispersoid particle distributions which may have affected SCC resistance. Therefore, it is difficult to interpret the test results of WSM on the basis of grain size alone, which can be done in the present investigation.

Conclusion of Mechanical and Corrosion Tests

The conclusions of this study of the effects of grain refinement on the properties of 7075 Al are as follows.

Mechanical Properties. Tensile and fatigue properties are not appreciably affected by grain refinement over the range of grain sizes studied in this investigation.

Corrosion Properties. Exfoliation corrosion resistance is greatly improved by grain refinement, although it is not clear whether grain size or shape causes this improvement. Stress corrosion crack propagation rates are slightly more rapid in the grain refined material.

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6.0 PERSONNEL

Program lanager on the subject program is Dr. Neil Paton, Dr. John Wert and Mr. J. C. Chesnutt were Principal Investigators. Supporting them on the theoretical side of the program was Dr. Bill Pardee. Mr. Murray Mahoney was responsible for some of the experimental work.

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