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Report

RELATIONSHIP BETWEEN HYDROGEN CONTENT AND
LOW DUCTILITY IN ALUMINUM-LITHIUM ALLOYS

to

NAVAL AIR SYSTEMS COMMAND

October 19, 1982

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FINAL REPORT

CONTRACT No. N00019-81-C-0433

on

RELATIONSHIP BETWEEN HYDROGEN CONTENT AND
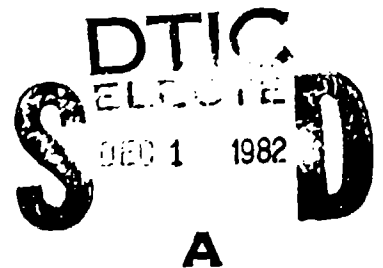LOW DUCTILITY IN ALUMINUM-LITHIUM ALLOYS

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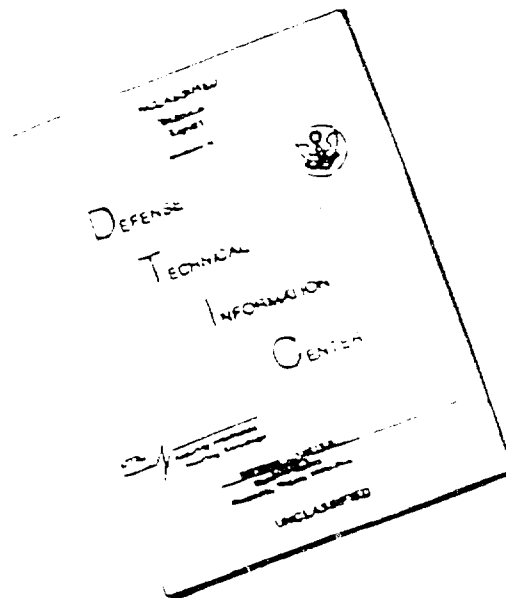
October 19, 1982

by

D. P. Hill and D. N. Williams

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Dear Sir:

Enclosed are ten copies of our Final Report on Contract N00019-81-C-0433 titled "Relationship Between Hydrogen Content and Low Ductility in Aluminum-Lithium Alloys".

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We believe that an important factor contributing to the low ductility of aluminum-lithium alloys has been identified by the research described in this report and we hope that this research will provide the impetus for alloy improvement through better control of hydrogen content during melting and fabrication. Recipients of this report who have questions regarding the research are encouraged to contact the authors for further details; D. P. Hill (614) 424-5597 or D. N. Williams (614) 424-4231.

Sincerely yours,

F. C. Holden
Manager
Physical Metallurgy Section

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An investigation was conducted to determine the relationship between hydrogen content and ductility in Al-Li alloys. A series of Al-3.4Mg-1.9Li-0.3Mn alloys was prepared in the laboratory by either arc melting or induction melting so as to produce a range of hydrogen contents. The lowest hydrogen contents were produced in alloys induction melted and cast in a rapidly flowing dry-argon atmosphere. Hydrogen is retained much more readily in Al-Mg-Li alloys than in conventional aluminum alloys, and hydrogen contents ranging from 14 to 45 ppm were measured in seven alloys that were processed to 0.040 inch sheet. (Cont'd)

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20. ABSTRACT (Continued)

Hydrogen was found to have an appreciable effect on properties; lower hydrogen contents resulted in significantly higher ductility (tensile elongation) and toughness (notched tensile strength/yield strength) and slightly lower yield strength. Hydrogen pickup occurred during processing, and the hydrogen contents of some of the alloys in sheet form were over twice as high as those measured in the ingots from which they were produced. Contamination did not appear to be limited to a surface scale; it extended some distance below the surface. Carrying out all thermal treatments in an argon atmosphere, including heating for forming and rolling, appeared to minimize contamination. Prolonged vacuum annealing was ineffective in removing hydrogen from Al-Mg-Li alloy sheet, but did cause some loss of Mg and/or Li. Efforts to determine whether hydrogen was present in the alloys in solid solution or as a hydride phase by optical metallographic examination were unsuccessful.

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RELATIONSHIP BETWEEN HYDROGEN CONTENT AND LOW
DUCTILITY IN ALUMINUM-LITHIUM ALLOYS

by

D. P. Hill and D. N. Williams

INTRODUCTION

An investigation supported by the Naval Air Systems Command
was initiated at Battelle in September, 1981, to determine whether the
ductility and fracture toughness of high-strength aluminum-lithium
alloys could be improved by better control of hydrogen content.

SUMMARY

Evaluation of laboratory-processed aluminum-magnesium-lithium alloys showed that a decrease in the hydrogen content of the alloys resulted in a significant increase in ductility and toughness. The alloys had a nominal composition of 3.4 percent magnesium, 1.9 percent lithium, and 0.3 percent manganese and contained from 14 to 45 ppm hydrogen. Commercial aluminum alloys that contain more than 4 ppm hydrogen exhibit considerable blistering, whereas the Al-Mg-Li alloys prepared in this investigation were free from blistering, indicating that much more hydrogen is retained in lithium-containing aluminum alloys than in conventional aluminum alloys.

Alloys were prepared under a controlled atmosphere using both arc-melting and induction-melting procedures. The best control of hydrogen content was achieved using induction melting in a rapidly flowing argon atmosphere. Although the resulting ingot was not processed or evaluated during this program, an induction-melted heat melted at 1300 F in flowing argon resulted in an ingot that contained only 9 ppm hydrogen.

Hydrogen contamination was found to occur during processing of the laboratory ingots to 0.040-inch-thick sheet. Hydrogen contents of some of the alloys more than doubled during such processing. Measurements of the hydrogen content of heat-treated sheet material after different amounts of surface conditioning indicated that hydrogen contamination was not restricted solely to the surface scale or to a very thin subsurface layer. Hydrogen contamination was prevented in one alloy by conducting all thermal treatments, including heating for forging and rolling, in an argon atmosphere. Vacuum annealing of 0.040-inch sheet was relatively ineffective in removing hydrogen. Moreover, some loss of magnesium and/or lithium alloy addition occurred during vacuum annealing.

The origin of the detrimental effects of hydrogen was not detected during optical metallographic studies of heat-treated sheet specimens from the series of hydrogen-containing aluminum-magnesium-lithium alloys. There was no evidence of a hydride phase in the microstructure, nor were any unusual etching effects or evidence of other structural changes observed that could be correlated with the differences in hydrogen content that were present.

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BACKGROUND

Compared with 2000- and 7000-series aluminum-base alloys, aluminum-lithium alloys that contain 1 to 3 weight percent lithium offer a 10 to 30 percent increase in specific modulus (elastic modulus ÷ density).[1-5] With comparable strength, this increase in specific modulus represents an attractive potential for substantial weight reductions in aerospace structures. However to date, neither conventional ingot processes nor powder-metallurgy processes have been successful in the production of a usable alloy. Low ductility and fracture toughness of the presently available aluminum-lithium alloys have made their use impractical.

Considerable research has been directed toward improving the ductility of these alloys, but little success has been achieved. Most of this research has been directed toward alloy or process modification specifically designed to inhibit planar slip, the facto generally considered responsible for low ductility in these alloys. A Battelle-funded literature survey suggested that the poor ductility of aluminum-lithium alloys might be caused by the formation of an extremely stable lithium hydride phase (LiH) during ingot-casting and powder-production processes, not planar slip.[6] Observations that led to that suggestion were:

(1) Lithium hydride is the most stable of the alkali-metal hydrides. The reaction of pure lithium with hydrogen at 1 atmosphere is spontaneous and exothermic at all temperatures up to 1560 F.[7] Lithium hydride seems likely to be present in aluminum-lithium alloys processed such that contact with moisture could occur.

(2) Aluminum-lithium alloys are reported to contain over 10 times the amount of hydrogen normally observed in high-strength aluminum-base alloys, but usually they are not porous.[4]* An explanation

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* Although ingots of aluminum-lithium alloys usually exhibit considerable porosity, analysis of ingots after removal of the porous portions shows the remainder to have an extremely high hydrogen content, even though it is acceptable by normal aluminum-porosity standards.

for this observation could be that hydrogen is
present as lithium hydride. Hydrides at grain
boundaries could easily have escaped detection
by researchers who did not apply special techniques
for their identification.

(3) Vacuum heat treatments have been used by Soviet re-
searchers to reduce the amount of porosity in welds
made in thin-gage aluminum-magnesium-lithium alloy
sheet (Soviet Alloy 01420).[8] Long-time high-
temperature, high-vacuum treatments were used for
this purpose, treatments not likely to be practical
for treating either powder compacts or large ingots.*

(4) The fatigue properties of the aluminum-copper-lithium
alloy 2020 are reported to show no evidence of the
environmental sensitivity common to other high-
strength aluminum-base alloys.[4] Environmental
sensitivity usually is attributed to the absorption
of hydrogen released by reaction of moisture with
freshly exposed slip surfaces generated during
fatigue.[9] This observation suggests that lithium
inhibits absorption by reacting with the hydrogen
to produce a stable, nondamaging hydride layer on
the slip surface.

The presence of a hydride phase is known to cause low ductility
and toughness in other alloys, such as, for example, certain titanium
alloys.[10] If LiH is responsible for the embrittlement of aluminum-
lithium alloys, controlling the amount of hydrogen in aluminum-lithium
alloys, thus by inference controlling the amount of LiH in the alloys, should
improve ductility and toughness. The present investigation was undertaken
to confirm this conclusion. The following sections of this report describe
in detail the preparation and evaluation of a series of alloys designed to
determine the effect of hydrogen content on the properties of an aluminum-
lithium alloy.

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* The low permeation rate of hydrogen through solid aluminum, even just below
  its melting temperature, would inhibit removal of hydrides from ingots.
  Removal of hydrides from powder compacts would be inhibited by the diffi-
  culty of achieving a high vacuum within the internal voids of a powder
  compact.

OBJECTIVES AND APPROACH

The objective of this research program was to determine whether the ductility and fracture toughness of aluminum-lithium alloys could be improved by reducing the hydrogen content of the alloys. The approach selected for achieving this objective was to determine the effects of hydrogen content on the ductility and fracture toughness of a series of laboratory-produced alloys having a range of hydrogen contents*. The alloy base selected for use in the investigation was Al-3.5Mg-2.5Li-0.3Mn, an alloy that had been extensively investigated in prior work described in the literature[4,5,8,12].

Methods selected for controlling the hydrogen content in the aluminum-lithium alloy included:

(1) Arc melting in a dry-argon atmosphere. Arc melting was expected to be particularly effective in providing low hydrogen contents because of the multiple melting and freezing cycles applied, the high temperature anticipated at the point of arcing, and the ability to both melt and freeze under a precisely controlled atmosphere. It was planned to prepare alloys with higher hydrogen contents either by the addition of lithium hydride to the charge or by melting under a partial pressure of hydrogen.

(2) Induction melting and casting in a rapidly flowing dry-argon atmosphere.

(3) Vacuum annealing of sheet material fabricated from arc-melted buttons.

Following forging and rolling of the laboratory-melted aluminum-lithium alloys to 0.040-inch-thick sheet and heat treating to the T6 temper, specimens were prepared to permit the measurement of unnotched and notched tensile properties. The ratio of notched tensile strength to unnotched yield strength was used to assess fracture toughness. Hydrogen analyses of the alloys were performed to permit the relationship between hydrogen content and ductility to be determined.

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* Original plans were to vary the hydrogen content from less than 0.04 ppm, the amount of hydrogen soluble in solid unalloyed aluminum at its melting point, to at least 0.77 ppm, an amount reported to be present in a low-ductility alloy described in the literature.[11] This plan was altered as the work progressed when it was found that the hydrogen content of aluminum-lithium alloys was much higher than reported in the literature.

PROCEDURES

Melting

The 3.5Mg-2.5Li-0.3Mn alloys were prepared from commercial-purity aluminum (99-plus percent aluminum), to provide impurity contents typical of those in commercial high-strength alloys, and high-purity alloy additions.

Lithium was added in two forms, either as high-purity ingot material from Foote Mineral Company or as an Al-19.5Li master alloy from the Lithium Corporation of America. The high-purity ingot material was handled with special precautions so as to minimize opportunities for contamination with hydrogen. The ingot was stored in oil prior to use. Sections cut from the ingot for use in preparing the alloys were stored under oil until immediately before melting, at which time they were cleaned in alcohol and acetone to remove the oil film and any decomposition products and charged within 1 minute into the melting equipment. The master alloy, supplied in the form of pea-sized splat particles, was received stored in argon and was handled in an argon-filled dry box. The master-alloy particles were weighed in the dry box and sealed in the melting unit before being removed from the dry box.

Magnesium was introduced as chips of high-purity ingot. Manganese was introduced as chips of an Al-10Mn master alloy prepared by arc melting at Battelle from commercial-purity aluminum and high-purity manganese (Alloy 1).

Lithium hydride powder was added to three of the charges -- 0.16 percent to Alloys 8 and 10, and 0.32 percent to Alloy 11 -- in an ineffective attempt to alter the hydrogen content of these alloys. When this was done, the lithium content of the hydride was included in computing the lithium content of the charge.

Charges were made up with slight excesses of magnesium and lithium to compensate for losses during melting. A series of preliminary trial melts was prepared to determine approximate melt losses during arc melting prior to preparing the alloys intended for evaluation (Alloys 2 through 5).

Arc-melted buttons that weighed 50 grams were melted in a tungsten-electrode electric-arc furnace with a water-cooled copper crucible, under one-third atmosphere of argon. All charges for arc melting were made up using lithium-ingot material. To insure good homogeneity in the resulting arc-melted button and to facilitate hydrogen removal, each button was melted six times,

flipping the button in the furnace between each melt. A static atmosphere was used during the majority of the arc melts, but two of the buttons (Alloys 6 and 9) were melted under a slowly flowing atmosphere in an effort to facilitate removal of hydrogen.

Three 300-gram induction-melted ingots were prepared in a rapidly-flowing argon atmosphere in which the flow rate was controlled so as to completely change the atmosphere in the melting unit four times per minute. These ingots were melted either in zirconia or graphite crucibles and were solidified in the crucibles upon completion of the melting period. Both the total time that the charge was held in the molten state and the maximum temperature achieved during melting were varied in an effort to control hydrogen content and, in addition, one of the alloys (Alloy 15) was solidified, and then remelted a second time. The form of the lithium used to prepare the charges also was varied in an effort to achieve better control of the hydrogen content.

A summary of the melting practices used in the preparation of the alloys is given in Table 1.

Fabrication

The arc-melted buttons were sectioned to provide a rectangular block approximately 0.6 inch thick that was surface ground to remove all of the original ingot surface and any visible cold-shut surface defects. Large near-surface voids were observed in several of the buttons during the conditioning operations; most of these were removed during conditioning. The blocks were homogenized for 48 hours at 900 F in a sealed Vycor capsule containing dry argon.

The conditioned-and-homogenized blocks were press forged to a thickness of 0.25 inch at 850 F, with the workpiece being reheated after each forging reduction. Relatively light reductions were used, particularly at the start of the forging process, and frequent surface conditioning was carried out during the forging process so as to remove cracks that developed during forging. Alloys 6 through 8 were heated in an air atmosphere for forging, but the atmosphere was changed to a flowing-argon atmosphere for the other alloys in order to minimize surface contamination during forging. Material losses between the button and completion of the forging process were relatively high because of the extensive amount of conditioning required throughout the process.

TABLE 1. DESCRIPTION OF THE LABORATORY-PRODUCED ALUMINUM-LITHIUM ALLOYS

Alloy No. [a]	Melting Method	Ingot Weight, grams	Type of Argon Atmosphere	Form of Lithium Addition	Other Melting Details
6	Arc melting	50	Slowly flowing	Ingot	Melted 6 tim
7	Arc melting	50	Static	Ingot	Melted 6 times
8	Arc melting	50	Static	Ingot + hydride	Melted 6 times
9	Arc melting	50	Slowly flowing	Ingot	Melted 6 times
10	Arc melting	50	Static	Ingot + hydride	Melted 6 times
11	Arc melting	50	Static	Ingot + hydride	Melted 6 times
12	Induction melting	300	Rapidly flowing	Ingot	Zirconia crucible, molten for 12 minutes at 1200 F maximum
13	Induction melting	300	Rapidly flowing	Master alloy	Zirconia crucible, molten for 12 minutes at 1300 F maximum
15	Induction melting	300	Rapidly flowing	Master alloy	Graphite crucible, melted twice, molten for 7 minutes at 1600 F maximum

(a) Other alloys prepared during this investigation, but not included in this table were:

Alloy 1: Arc-melted Al-10Mn master alloy.
Alloys 2 through 5: Series of arc-melted alloys prepared to ascertain magnesium and lithium losses during arc melting and to check fabrication procedures.
Alloy 14: Induction-melted alloy that was lost when the zirconium crucible cracked during melting.

The forged billets were rolled from 0.25 inch thick to 0.040 inch thick at 850 F in a two-high rolling mill with 3.5-inch-diameter work rolls, using reductions of up to 15 percent per pass. Alloys 6 through 8 were heated in an air atmosphere for rolling, whereas the other alloys were heated in a flowing-argon atmosphere in an effort to reduce surface contamination. Some grinding was necessary between passes during the hot-rolling process to remove surface and edge cracks, but material loss during the rolling process was not excessive. Alloys 6 through 8 were ground using a wet-belt grinder; the other alloys were ground dry to reduce opportunities for surface contamination.

Only one of the induction-melted ingots, Alloy 15, was fabricated to sheet. A rectangular block approximately 0.8 inch thick was cut from this ingot and homogenized in an argon-filled Vycor capsule for 12 hours at 900 F, 4 hours at 950 F, and 48 hours at 1000 F. The longer time, higher temperature homogenizing treatment was used because of the low freezing rate that the induction-melted ingots encountered upon solidification in the melting crucible and the opportunity it provided for considerable coring to occur. The homogenized block from the induction-melt ingot was fabricated to 0.040-inch-thick sheet by forging and rolling using techniques similar to those described above for the arc-melted buttons. All heating for forging and rolling was carried out in a flowing-argon atmosphere, and all conditioning was done by dry grinding.

Sheet from Alloys 6 through 8 was solution heat treated for 10 minutes at 950 F, water quenched, and aged for 36 hours at 375 F. This treatment was selected, on the basis of a review of published information on alloys similar to the Al-3.5Mg-2.5Li-0.3Mn alloy, to produce the fully aged T6 temper. All heat treatments of Alloys 6 through 8 were performed in air. Heat treatments were modified slightly for the remainder of the alloys following a brief laboratory study undertaken to determine whether the hardness could be increased by an alternate heat treatment. Although this study suggested that increases were only marginal, a revised treatment was applied consisting of solution heat treatment for 20 minutes at 1000 F, water quenching, and aging for 48 hours at 375 F. This treatment was conducted in a flowing-argon atmosphere.

Sheet from one of the alloys, Alloy 10, was vacuum annealed prior to solution heat treatment and aging, in an attempt to produce a very low hydrogen content. This material was heated in an evacuated chamber at a pressure less than 10^{-3} mm Hg and annealed, with continuous evacuation, for 48 hours at 1000 F. The sheet was cooled to room temperature in the vacuum chamber.

Evaluation

Chemical analyses were performed to determine both the metallic-element content and the hydrogen content.

Metallic elements were determined by emission-spectrographic analysis at a commercial testing laboratory. Analyses of as-cast material were performed for both the major alloying elements and for the common impurities in aluminum.

Preliminary information suggested that reliable determination of hydrogen in aluminum-lithium alloys was likely to prove difficult, and a telephone survey was made to determine what procedures were available and the probable reliability of each. On the basis of this survey, the Boeing Aerospace Company was selected to perform the hydrogen analyses. The sensitivity of the Boeing equipment was reported to be 5 ppb by weight[13], and the reproducibility and accuracy of the equipment had been proven with NBS standards[14]. Also, it appeared that this equipment was not likely to be adversely affected by the presence of lithium in the alloy to be analyzed. Hydrogen analysis in the Boeing equipment consists of the following steps:

- (1) Hydrogen is liberated from the specimen by induction melting in a zirconia boat.

- (2) The liberated hydrogen is carried in a flowing high-purity-argon gas stream to a semipermeable palladium membrane (the membrane is permeable to hydrogen, but not argon).

- (3) Hydrogen passes through the membrane into a high-vacuum chamber (10^{-9} mm Hg).

- (4) The quantity of hydrogen introduced into the chamber is determined from measurement of the vacuum-pump current required to maintain the vacuum.

The results of the initial analyses performed with the Boeing equipment indicated that the hydrogen content of the alloys was much higher than had been anticipated so, as a check of the Boeing procedures, duplicate analyses were carried out using Battelle's ITHAC-01 Hydrogen Determinator. This equipment has a sensitivity of 10 ppb and a reproducibility of $\pm$ 0.1 ppm, but is not well suited to the hydrogen analysis of materials that contain metallic elements that volitilize readily during melting. Although the Battelle equipment required extensive cleanup after use, analyses of several of the samples previously analyzed with the Boeing equipment confirmed the accuracy of the

Boeing results. Hydrogen analyses were performed using samples from both as-cast ingots and heat-treated sheet.

Microstructural studies were performed on metallographic specimens prepared from as-cast material, homogenized material, and heat-treated sheet. These studies included examination of grain size, precipitate distribution, the type and distribution of secondary constituents, and fracture characteristics. Efforts also were made to determine in what form hydrogen was present in the alloys, whether in solid solution or as a precipitated phase, but these efforts were unsuccessful.

Specimen configurations used to measure unnotched and notched tensile properties are shown in Figure 1. Because of material limitations, properties of the arc-melted alloys were determined by single tests. However, for Alloy 15, the induction-melted alloy, sufficient material was available for the preparation of duplicate specimens, and the results of those duplicate tests were in reasonable agreement. Unnotched tensile specimens were tested at a crosshead speed of 0.01 inch per minute; notched tensile specimens were tested at a speed of 0.0025 inch per minute.

a. Tensile Specimen
Elongation Measured in the 1.000-Inch Gage Length
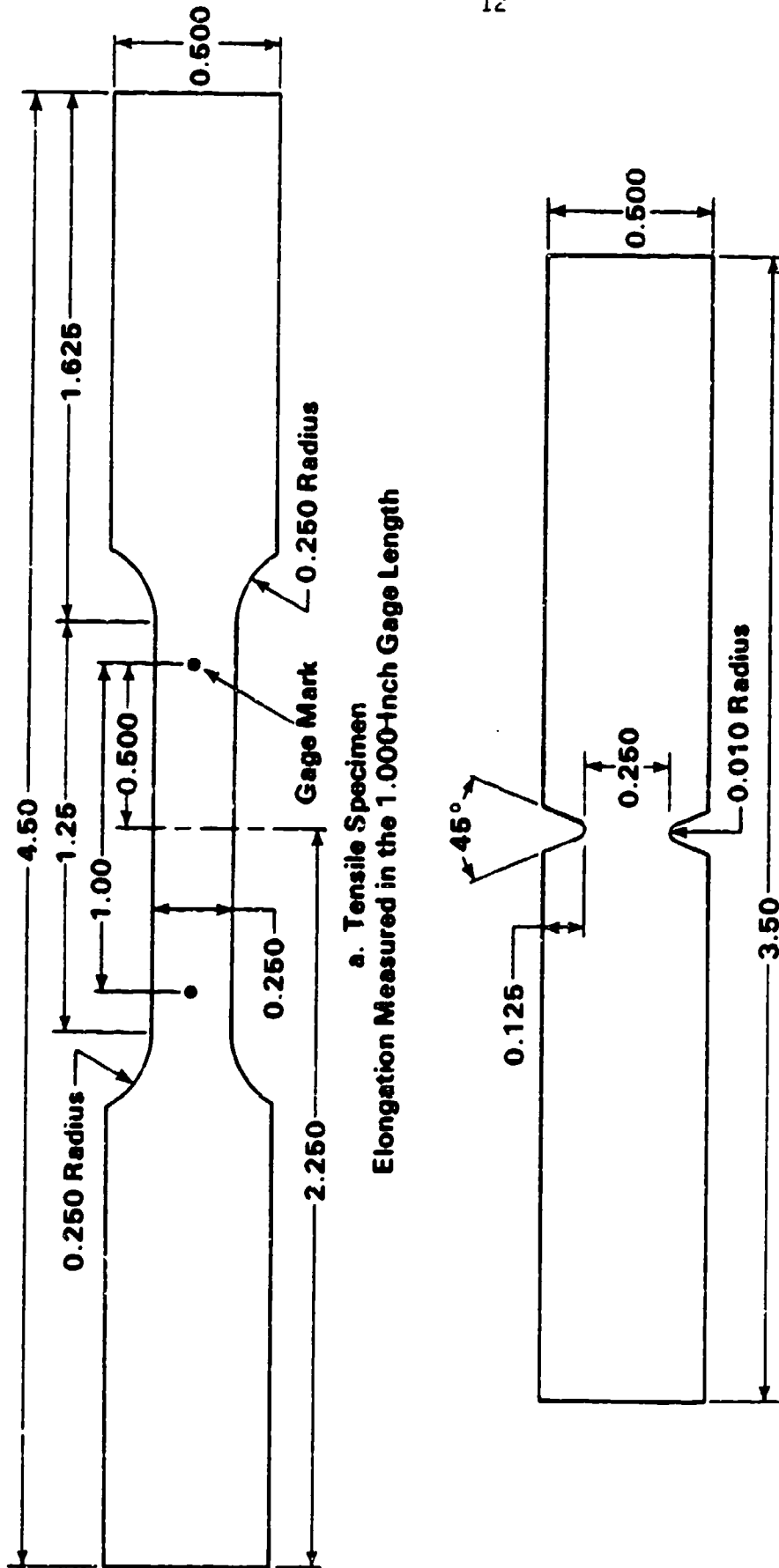
b. Notched Tensile Specimen
$K_T \cong 3.5$

FIGURE 1. SPECIMEN CONFIGURATION

All specimens were machined from solution-heat-treated-and-aged 0.040-inch-thick sheet with the axis of the specimen parallel to the rolling direction of the sheet. All dimensions are in inches.

RESULTS

The chemical compositions of the seven alloys that were fabri-
cated to sheet are given in Table 2. The average composition of these
alloys was Al-3.4Mg-1.9Li-0.3Mn, which was somewhat different from the
desired composition, Al-3.5Mg-2.5Li-0.3Mn. The lithium contents of the
seven alloys were nearly identical, although appreciably less than was
desired. This condition resulted in lower strength in the alloys, but
did not adversely affect the assessment of the relationship between hydro-
gen content and ductility. The magnesium contents of the seven alloys
showed appreciable variation, ranging from 2.9 to 3.8 percent. However,
the variation in magnesium content did not interfere with the analysis of
the effects of the variation in hydrogen content.

TABLE 2. CHEMICAL COMPOSITION

Alloy	Alloy Content, weight percent									
	Mg	Li	Mn	Si	Fe	Cu	Zn	Ni	Cr	Ti
6	3.52	2.05	0.31	0.14	0.43	0.13	0.04	<0.01	<0.01	<0.01
7	3.23	1.88	0.31	0.12	0.41	0.12	0.02	<0.01	<0.01	<0.01
8	2.92	1.90	0.31	0.13	0.41	0.12	0.02	<0.01	<0.01	<0.01
9	3.46	1.98	0.28	0.20	0.44	0.08	0.07	0.07	0.06	<0.01
10	3.24	1.95	0.28	0.24	0.56	0.12	0.03	0.07	0.04	<0.01
11	3.18	1.95	0.29	0.40	0.54	0.14	0.05	0.07	0.05	<0.01
15	3.80	1.90	0.28	0.16	0.43	0.19	0.02	<0.01	<0.01	<0.01
Target Composition	3.50	2.50	0.30							

The results of hydrogen analyses performed at Boeing are given in
Table 3. Analyses of the first three arc-melted alloys were performed using
samples cut from both the as-cast buttons and heat-treated sheet (T6 temper).

TABLE 3 HYDROGEN CONTENT[a]

| | Hydrogen Content (ppm by weight) for Indicated Condition | | | | |
| | Ingot With All Original Surface Material Removed | Sheet in T_4 Temper, Lightly Abraded | Sheet in T_6 Temper, No Abrasion | Sheet in T_6 Temper, Lightly Abraded | Sheet in T_6 Temper, Heavily Abraded |
Alloy					
			Arc-Melted Alloys		
6	16	--	41	41, 43[b]	23
7	23	--	--	43[b]	26
8	29	--	44	44, 45[b]	29
9	--	28	--	--	--
10	--	20[c]	--	--	--
11	--	27	--	--	--
			Induction-Melted Alloys		
12	9	--	--	--	--
13	29	--	--	--	--
15	15	14	--	--	--

(a) Determined by Boeing unless otherwise noted.

(b) Determined at Battelle.

(c) Vacuum annealed before heat treatment.

The latter samples were examined after three different types of surface preparation; none, light abrasion, and heavy abrasion. It is estimated that as much as 0.005 inch of metal was removed from each surface during heavy abrasion (mea.:urements of the amount of metal removed were not made.) As is shown in Table 3, it appeared that appreciable hydrogen had been absorbed by these three alloys during processing, and that the absorbed hydrogen had diffused a considerable distance below the surface. Analyses of the second group of arc-melted alloys, Alloys 9, 10, and 11, were limited to samples of lightly abraded sheet in the T4 condition.* Both as-cast and sheet samples (T4 temper) of induction-melted Alloy 15 were analyzed. Interestingly, Alloy 15, which was forged, rolled, and heat treated in a flowing-argon atmosphere, did not appear to have absorbed significant hydrogen during processing. The hydrogen content of two induction-melted alloys that were not fabricated to sheet also are included in Table 3. Comparison of the hydrogen contents of the three induction-melted alloys provided two tentative conclusions about the effectiveness of the changes in induction-melting practice used to control the hydrogen content. Alloy 12 was prepared with lithium from the lithium ingot, whereas the other two alloys were prepared using the aluminum-lithium master alloy. Even though the melting practices for Alloy 12 were less well controlled than for the other two, the resulting ingot had a lower hydrogen content, presumably as a consequence of the difference in the form in which the lithium addition was made. The differences in hydrogen content between Alloys 13 and 15 are considered likely to have resulted from double melting and the difference in the melting temperature (see Table 1).

Because of the significant absorption of hydrogen that occurred in the alloys during processing, and the observation that this hydrogen was not located at the surface but had diffused an appreciable distance into the material, it was concluded that comparisons of the relationship between hydrogen content and ductility should be based on hydrogen contents measured in lightly abraded heat-treated sheet (T4 or T6 temper).

The vacuum-annealing treatment applied to Alloy 10 resulted in only a moderate reduction in hydrogen content. This alloy was processed

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* Surface reactions during aging at 375 F were not considered likely to result in subsurface hydrogen contamination. Therefore it was assumed that the hydrogen content of an alloy could be determined using material in either the T4 or T6 temper.

in the same manner and at the same time as Alloys 9 and 11, and it is presumed that the hydrogen content in this alloy prior to vacuum annealing was about 28 ppm. If so, only about 8 ppm hydrogen was removed by the lengthy vacuum-annealing treatment applied to sheet material prior to heat treatment. However, as is indicated below, vacuum annealing had a significant effect on properties as a consequence of removal of alloying elements.

The results of unnotched and notched tensile tests of the Al-Li alloys in the T6 temper are given in Table 4. With the exception of Alloy 6, which had a rolling lamination in the gage length of the unnotched tensile specimen, and Alloy 10, which was vacuum annealed prior to heat treatment, the alloys exhibited very similar tensile strengths, approximately 56 ksi. The differences in strength that were observed did not correlate with differences in magnesium content (see Table 2). Differences also were observed in yield strength among the alloys. As is shown in Figure 2, the yield strength decreased slightly with decreasing hydrogen content. (Alloy 6 was included in this figure on the assumption that the rolling lamination that led to premature failure was not likely to have affected the yield strength significantly.) The tensile-elongation values of the six arc-melted alloys were quite similar, with the exception of Alloy 6 that failed prematurely as a result of a rolling lamination in the gage section. Alloy 15 exhibited appreciably higher elongation. This is attributed to its lower hydrogen content, not the fact that it was induction melted. All seven of the alloys showed a flat, intergranular tensile fracture with relatively little evidence of necking prior to fracture. The most necking was observed in the tensile specimen from Alloy 10, the alloy that was vacuum annealed. That alloy also exhibited much lower tensile strength and yield strength than did the others. Microstructural examination of the vacuum-annealed-and-heat-treated sheet suggested that the difference in properties was related to removal of magnesium and/or lithium during vacuum annealing; also, considerable porosity was evident in the material.

The notched tensile strength appeared to increase as the hydrogen content decreased, although there was some variation among alloys of similar hydrogen content (this variation did not appear to be related to

TABLE 4. MECHANICAL PROPERTIES OF ALLOY SHEET IN THE T6 TEMPER

Alloy	Tensile Strength, ksi	Yield Strength, ksi[a]	Elongation in 1.0 Inch, percent	Notched Tensile Strength	NTS/YS
6	44.7[b]	39.6	1.5[b]	43.6	1.10
7	55.9	40.3	3.0	46.6	1.16
8	59.2	39.6	4.0	48.5	1.22
9	54.1	38.3	3.5	48.7	1.27
10[c]	35.2	23.9	4.0	40.8	1.71
11	55.7	38.8	3.0	45.8	1.18
15[d]	55.8	35.8	7.5	50.8	1.42

(a) Determined at 0.2 percent offset.

(b) This value is low because the specimen failed along a lamination.

(c) Vacuum-annealed alloy.
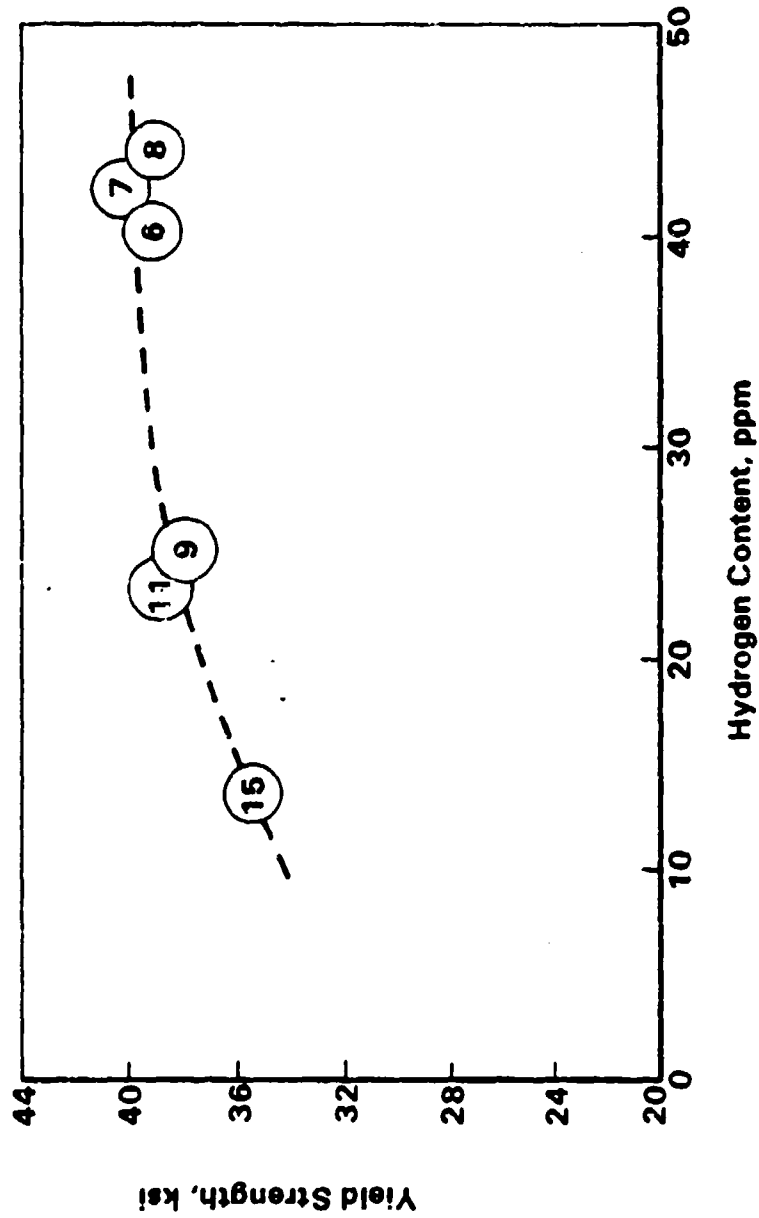
(d) Average of duplicate tests.

FIGURE 2. EFFECT OF HYDROGEN CONTENT ON THE YIELD STRENGTH OF
ALLOY SHEET IN THE T6 TEMPER

The numbers in the data-point circles denote the
alloy number.

differences in magnesium content). The average notched tensile strength
for the three alloys that contained about 43 ppm hydrogen (Alloys 6, 7,
and 8) was 46.2 ksi. The average notched tensile strength for the two
alloys that contained about 28 ppm hydrogen (Alloys 9 and 11) was 47.2 ksi,
and the notched tensile strength of Alloy 15 that contained only 14 ppm
hydrogen was 50.8 ksi. Even greater differences were apparent in the
ratio of notched tensile strength to yield strength, the value most fre-
quently used to assess the fracture toughness of aluminum-lithium alloys,
as a function of hydrogen content. As is shown in Figure 3, as the hydro-
gen content decreased, the ratio of notched tensile strength to yield
strength increased.

All of the alloys exhibited a coarse, equiaxed grain structure
as fully heat treated (T6 temper), indicating that the manganese addition
was not effective in inhibiting recrystallization during processing. The
grain size is shown in Table 5. All of the alloys except Alloy 10
exhibited about the same grain size. Alloy 10, which was vacuum annealed,

TABLE 5. GRAIN SIZE OF THE ALLOY
SHEET IN THE T6 TEMPER

Alloy	Average Grain Diameter[a], inches
6	0.0017
7	0.0020
8	0.0018
9	0.0015
10	0.0053
11	0.0016
15	0.0013

(a) Line-intercept method.

Hydrogen Content, ppm
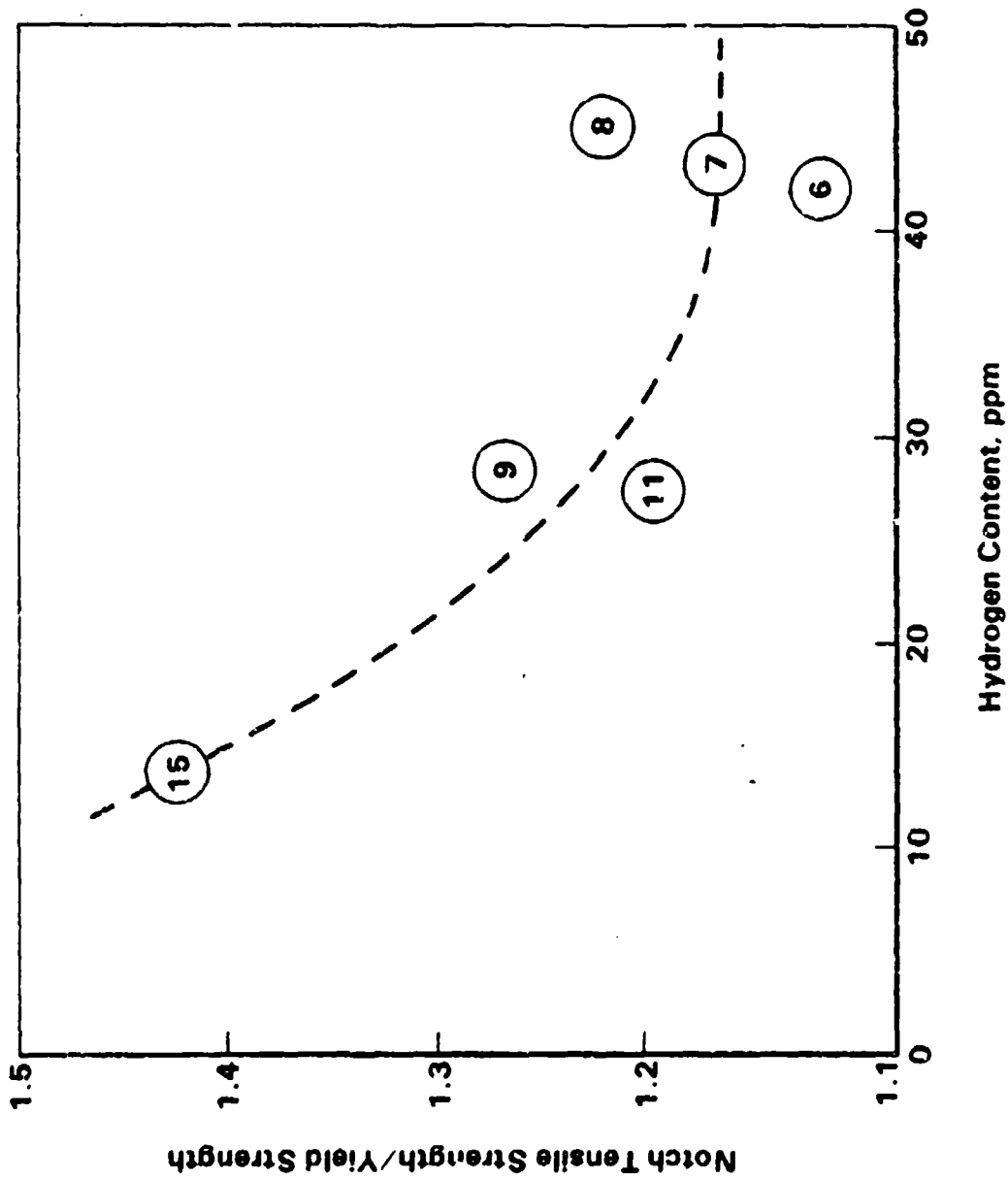
Notch Tensile Strength/Yield Strength

FIGURE 3. EFFECT OF HYDROGEN CONTENT ON TOUGHNESS (NOTCHED TENSILE STRENGTH/YIELD STRENGTH) OF ALLOY SHEET IN THE T6 TEMPER

had an appreciably coarser grain size. All of the alloys also exhibited at least two primary intermetallic phases. One was an Al-Fe-Si phase; the other was tentatively identified as the Al₂LiMg-phase. This phase was extremely reactive and corroded rapidly when exposed to the atmosphere. Also, it was easily removed during metallographic preparation. Relatively coarse grain-boundary precipitates were also apparent in the heat-treated material. There appeared to be either fewer or finer grain-boundary precipitates in Alloy 15 than in the others. This was surprising in view of the higher magnesium content of this alloy and may indicate that decreasing the hydrogen content of Al-Mg-Li alloys reduced the tendency for grain-boundary precipitation.

No differences in microstructure were detected as a function of the differences in hydrogen content other than the difference in the nature of the grain-boundary precipitate noted above. There was no apparent difference in general staining behavior or in the rate of grain-boundary etching that might be attributed to the presence of a sub-microscopic hydride phase.

The results of studies directed toward determining the optimum heat treatment for the Al-3.4Mg-1.9Li-0.3Mn alloy are shown in Tables 6 and 7. There was little change in microstructure among specimens solution heat treated at 900 F, 950 F, and 1000 F, as is shown in Table 6. However, solution heat treatment of 1050 F caused growth in the size of the primary Al₂LiMg intermetallic particles. The depth below the surface that evidence of dealloying could be detected increased with both increased solution-heat-treatment temperature and increased time. The hardness of the alloy, measured after aging for 36 hours at 375 F, increased slightly when the solution-heat-treatment temperature was increased from 900 F to 950 F, but remained essentially the same with further increases in the solution-heat-treatment temperature. The hardness of the alloy also increased when the solution-heat-treatment time at 950 F was increased from 10 to 60 minutes, but was unaffected by a further increase of the solution-heat-treatment time to 240 minutes. It was also observed that variation in the solution-heat-treatment conditions affected the susceptibility of the metallographic specimens to staining upon exposure to the environment.

TABLE 6. EXAMINATION OF SOLUTION-HEAT-TREATMENT CONDITIONS

(Studies were conducted using specimens from Alloy 11)

Solution Heat Treatment(a) Temperature, F	Time, min.	Depth of Visible Dealloying, inch per side	Microstructure	Knoop Hardness (500-Gram Load)
As rolled		0.0005	Homogeneous precipitate + primary phase	121
900	60	0.0013	Recrystallized grains out-lined by ppte. + primary phase	143
950	10	0.0008	Recrystallized grains out-lined by ppte. + primary phase	134
	60	0.0013		148
	240	0.0030		147
1000	60	0.0024	Recrystallized grains out-lined by ppte. + primary phase	149
1050	60	0.0034	Recrystallized grains out-lined by ppte. + primary phase. Growth of Al$_2$LiMg primary-phase particles was evident	147

(a) The specimens were water quenched and aged for 36 hours at 375 F prior to evaluation.

The longer the time or the higher the temperature of solution heat treatment, the less staining was observed upon exposure. On the basis of the above investigation, solution heat treatment for 20 minutes at 1000 F was selected for use in heat treating Alloys 9 through 15.

The effects of aging time at 375 F on the hardness of specimens solution heat treated 20 minutes at 1000 F is shown in Table 7. Maximum hardness appeared to be achieved after aging for approximately 48 hours.

TABLE 7. EXAMINATION OF THE EFFECTS OF AGING TIME ON HARDNESS

Alloy	Aging Time, hours[a]	Knoop Hardness (500-Gram Load)
9	10	144
	24	145
	48	150
	72	148
10[b]	10	144
	24	146
	48	147
	72	146

(a) The specimens were solution heat treated for 20 minutes at 1000 F.

(b) Vacuum annealed prior to solution heat treatment and aging.

DISCUSSION OF RESULTS

For the Al-3.4Mg-1.9Li-0.3Mn alloy selected for investigation in this work, reducing the hydrogen content from 43 to 14 ppm reduced the yield strength from 39.8 to 35.8 ksi and increased the ratio of notched tensile strength to yield strength from 1.10 to 1.42. The relationship between the yield strength and the toughness parameter for six of the alloys evaluated* is plotted in Figure 4 on a graph reproduced from an earlier investigation in which yield strength/toughness relationships were investigated for a series of Al-Mg-Li alloys. It is evident from the trend line for the data from the present work that reducing the hydrogen content of the Al-3.4Mg-1.9Li-0.3Mn alloy resulted in a significant increase in toughness; the slope of the trend line was appreciably steeper than that of the trend line for commercial high strength aluminum-alloy extrusions. The position of the data on the graph relative to the series of Al-Mg-Li alloys evaluated in the prior work is not considered too significant. The alloys evaluated in prior work were extruded at 700 F from 6-inch-diameter direct-chill ingots and thus, would be expected to exhibit appreciable improvements in properties as a consequence of superior melting practice, greater breakdown of the cast structure, and deformation strengthening.

Interestingly, as is apparent in Figure 4, the slope of the trend line representing the alloys evaluated in the present work is very similar to that shown for several of the lower strength alloys evaluated in the prior work. It was suggested in that work that the trend line for low-strength Al-Mg-Li alloys was intrinsically higher than that for high-strength alloys, and that a transition occurred in the slope of the trend line, when evaluated over a series of alloys of different composition, at a yield strength of about 45 ksi. Higher strength alloys were reported to exhibit a much flatter trend line, such as that shown for Alloy 1 in Figure 4. However, examination of the data in Figure 4 suggests that that was not generally true, and that most of the alloys, regardless of yield strength, exhibited a steep trend line; see, for example, the dashed line representing a redrawing of the trend line for Alloy 9, which exhibited a yield strength (average) of 56.9 ksi. Only two of the alloys, 1 and 2, clearly exhibit a trend line with a slope similar to that of the commercial extrusion.

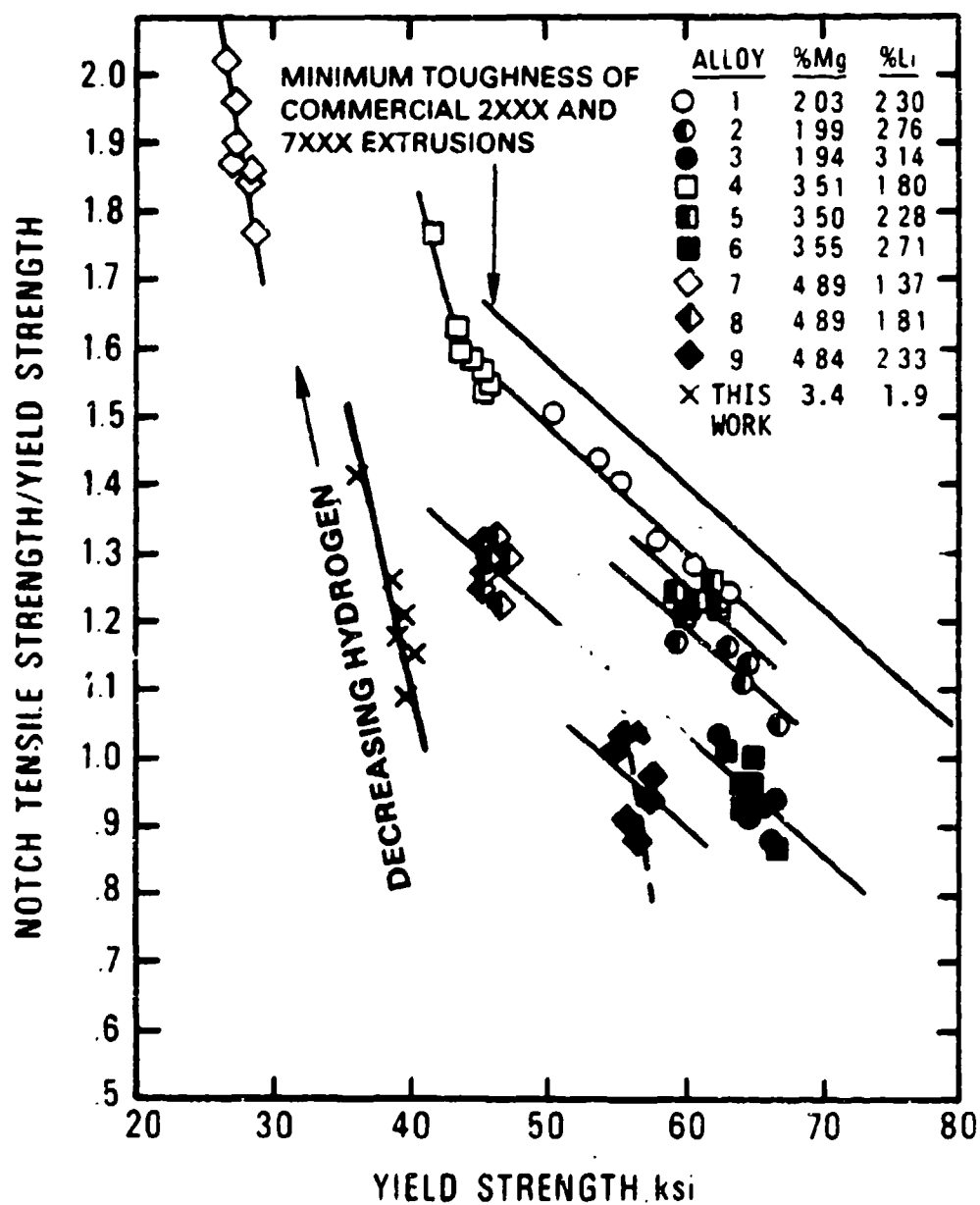* Data for Alloy 10, which was vacuum annealed, was not included.

FIGURE 4. COMPARISON OF THE VARIATION OF FRACTURE
TOUGHNESS WITH YIELD STRENGTH OBSERVED
IN THIS STUDY WITH THAT REPORTED IN THE
LITERATURE FOR ALUMINUM-MAGNESIUM-
LITHIUM ALLOYS (SEE REFERENCE 5)

The similarity of the slope of the trend line for the data from the present work and that observed for the majority of the alloys evaluated in the prior work suggests that the variation in toughness and strength observed in the prior work may also have been due to variations in the amount of hydrogen present in the alloys. The seven data points per alloy presented in Figure 4 for most of the alloys from the prior work represent tests of specimens from each alloy solution heat treated over a range of temperatures between 800 F and 950 F. In most of the cases in which a steep trend line was observed, assuming reasonable scatter in test data, lower toughness and higher yield strength were observed in specimens heat treated at the higher temperatures. This behavior suggests three possible explanations for the variation in properties observed in the alloys. First, hydrogen may have been absorbed during solution heat treatment, the amount absorbed increasing with temperature. Second, heat treatment may have resulted in a more uniform distribution of near-surface hydrogen contamination developed during processing, the amount of redistribution increasing with temperature. Third, the hydrogen content may have varied along the length of the 2-inch-diameter extruded rods from which specimens were obtained. Such variations may have resulted from variations present in the 6-inch diameter direct-chill ingot from which the rods were extruded or may have been developed as a result of hydrogen contamination during processing. Presumably, specimen location in the rod was not randomized. If not, all specimens heat treated at a given temperature may have come from the same portion of the extrusion (for example, the front end of the extruded rod).

If, as seems likely based on the present work, the toughness/yield strength variation observed in the prior work as a function of solution heat treatment temperature is related to hydrogen content, it is clear from Figure 4 that reductions in hydrogen content could readily result in combinations of toughness and yield strength for Al-Mg-Li alloys of optimum composition and processing history that equal or exceed values presently available in commercial high-strength-alloy extrusions.

CONCLUSIONS

(1) Hydrogen contents in laboratory-processed Al-Mg-Li alloys were much higher than in commercial aluminum alloys. (Measured by the same analytical technique as that used in this work, hydrogen contents of commercial alloys are less than 4 ppm.[15])

(2) A reduction in the hydrogen content of Al-Mg-Li alloys resulted in a small decrease in yield strength and a significant increase in ductility and toughness. Further reductions in hydrogen content, greater than those achieved in this work, would be expected to result in further increases in ductility and toughness.

(3) The hydrogen content of Al-Mg-Li alloy ingots was reduced by improvements in melting and casting procedures, but control of hydrogen content in these alloys is much more difficult than in conventional alloys.

(4) Hydrogen contamination occurred during processing of Al-Mg-Li alloys. Although hydrogen contamination was more severe near the surface, hydrogen was not present solely in a thin surface layer, but significant penetration into the alloys occurred.

(5) Vacuum annealing was a relatively ineffective process for removing hydrogen from Al-Mg-Li alloys. In addition, some magnesium and/or lithium was removed from the alloys during vacuum annealing, an undesirable effect.

RECOMMENDATIONS

Development of melting procedures to produce low-hydrogen aluminum-lithium alloys should continue. An induction-melted Al-Mg-Li alloy ingot that contained only 9 ppm hydrogen was cast successfully in the present work, although not evaluated, and further improvements in induction-melting practices would be expected to result in even lower hydrogen contents. Efforts should be made to develop optimum induction-melting practices for the Al-3.4Mg-1.9Li-0.3Mn alloy with the objective of reducing the hydrogen content to less than 4 ppm. The effectiveness of the optimized practice for the production of low-hydrogen Al-Cu-Li or Al-Cu-Li-Mg alloys should also be investigated.

Additional work is needed to define more exactly the source of hydrogen contamination during processing, the kinetics of absorption, and ways in which contamination can be prevented. The present work has suggested that contamination can be reduced significantly by relatively simple changes in fabrication procedures, such as carrying out all thermal treatments in an inert atmosphere. The effectiveness of this process change should be confirmed.

Finally, it is recommended that an intensive effort be made to determine how hydrogen affects the ductility and toughness of Al-Li alloys, whether through solid solubility, hydride formation, or interference with precipitation processes. These studies could most readily be carried out using a binary Al-Li alloy* to reduce the complexity of the microstructure and to facilitate the identification of the behavior of hydrogen in the alloy.

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* Modified with Zr or Mn to inhibit recrystallization during processing.

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