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(M.S. Thesis)

May 1989

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**Cryogenic Mechanical Properties of Low Density
Superplastic Aluminum Alloys.**

Stephanie Louise Verzasconi

Masters Thesis

Department of Materials Science and Mineral Engineering
University of California at Berkeley

and

Center for Advanced Materials
Materials and Chemical Sciences Division
Lawrence Berkeley Laboratory
1 Cyclotron Road
Berkeley, CA 94720

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CRYOGENIC MECHANICAL PROPERTIES OF LOW DENSITY SUPERPLASTIC ALUMINUM ALLOYS.

1 INTRODUCTION

1.1 Background

In order to reduce energy costs for transportation vehicles, materials researchers are called upon to help reduce vehicle weight by discovering lighter, stronger, stiffer materials. The need for these advanced materials is most acute in space vehicles, where the cost savings in fuel per weight reduction is so great that the use of relatively expensive aluminum alloys, such as Al-Li and Al-Sc, could be justified. Superplastically formable (SPF) low density aluminum alloys are currently being considered for aerospace applications, such as the National Aerospace Plane and the Advanced Launch System. These alloys may be used generally throughout the craft structure or, due to the promising low temperature mechanical properties of some of these alloys, in cryogenic fuel tanks. In addition to cost savings via lowering alloy density, superplastically formed structures can be more efficient than conventional milled structures because they reduce material waste, decrease forming energy needs, and can be made complicated parts that support more load for a given weight ¹.

Two alloy systems, mainly Al-Li-Cu and Al-Mg-Sc, were studied in this work. Both of these systems have been shown to be superplastically formable in the conditions chosen ^{2,3}, and both provide a significant density reduction over a currently used aluminum cryogenic fuel tankage material, 2219. For illustration, the densities of 2219, Al-Cu-Li alloy 2090, and Al-4Mg-0.5Sc (wt. %) are shown in Table 1, as is the percent savings in density of 2090 and Al-4Mg-0.5Sc compared to 2219. The Al-Mg-Sc alloy provides over 50 percent of the density reduction of 2090 over 2219. In addition to lower density, Al-Li alloys have a higher elastic modulus (stiffness) than conventional aerospace alloys ⁴;

furthermore, selected Al-Li alloys exhibit some exceptional mechanical properties at cryogenic temperatures ⁵⁻¹³.

Table 1: Density and percent density savings compared to 2219.

Alloy	Density (gm/cc)	% Savings over 2219
2219	2.72	-
Al-4Mg-5Sc	2.65	2.6
2090	2.59	4.8

Tank designs utilizing SPF aluminum alloys for the Advanced Launch System are currently being considered. Although the design criteria and property limits have not yet been agreed upon, interest was expressed at NASA Langley and Northrop Aerospace Corporation in the strength and toughness of several SPF Al-Li alloys, some of which included an indium addition to alloy 2090. Indium has previously been shown to improve the hardness of aluminum alloys ¹⁴; thus it was proposed to improve alloy strength, as strength and hardness trends are generally similar. A strength boost over the T6 condition is desired (T6 is solutionized, quenched, and peak strength aged). Furthermore, the superplastic condition does not include the stretch that improves the alloy strength-toughness combination from the T6 to the T8 condition in aluminum-lithium alloys (T8 is solutionized, quenched, stretched, and peak strength aged). Indium, like the mechanical stretch, has been shown in this and other work to improve the distribution and number of strengthening phases ¹⁵. Although the mechanism is not yet understood, improvements in strength are expected from more evenly distributed precipitation as shown by strengthening theory ¹⁶.

Cryogenic characterization of the Al-Mg-Sc alloys was stimulated both by the general interest in SPF alloys, since work at Alcoa has shown these alloys to exhibit exceptional superplastic formability and ambient temperature mechanical properties ¹⁷, and by the hypothesis that these alloys would remain exceptional at cryogenic temperatures, due to

their particle strengthening mechanisms. These materials are strengthened directly both by small, spherical, coherent, Al_3Sc precipitates, and by magnesium in solid solution. An additional component of strengthening is derived indirectly from grain structure refinement promoted by the Al_3Sc precipitate, which has been shown to pin the boundaries ¹⁷.

The main purpose of this work is to characterize the cryogenic strength and toughness of several Al-Cu-Li and Al-Mg-Sc alloys. In addition, the microstructures and fracture surfaces are characterized and related to these properties where possible.

1.2 Approach

The basic approach was to test the strength and toughness of several candidate alloys in the unformed condition. The properties of formed parts are the subject of near term future research in the ongoing project; however, it was felt that in the absence of formed material, the properties of unformed material would present valuable information.

The Al-Li alloys that were tested at room (300 K) and liquid nitrogen temperatures (77 K), included 2090, 8090, 2090+In, and 2219 (compositions in Table 2). Liquid helium (4 K) tests are relatively expensive and are therefore usually postponed pending promising properties at 77 K. The Al-Mg-Sc materials tested had a constant weight percent scandium while the magnesium content was varied. The identical lots of these materials were originally tested in ambient temperatures at Alcoa. Because of this and limited material, tests for this work started at 77 K, and later, due to the exceptional 77 K properties, selected materials were tested at 4 K. Alloy 6061, readily available at the Lawrence Berkeley Laboratory (LBL), was used to standardize methods prior to testing other materials, which were in short supply.

Toughness characterization was done using an indicator test, the Kahn tear, for various reasons. First, wide specimens, such as the center cracked panel, are problematic since the cryogenic test facility available for 4 K testing is only three inches wide and since material

was limited in most cases. Second, the choice of the Kahn tear over a notched tensile test was made both because of the large existing cryogenic data base on aluminum alloys tested with this method and because the Kahn tear test provides three toughness measurements, while the notch tensile test has only one.

During the tear test, load versus displacement data are collected, as shown schematically in Figure 1. The three measures of toughness are as follows: 1). the unit initiation energy (UIE), or area under the load-displacement curve before maximum load, P , divided by the sample ligament area, A , 2). the unit propagation energy (UPE), or area under the curve after peak load, divided by A , and 3). the tear-yield ratio, which is the tear strength, $T=4P/A$, divided by the 0.2 % offset yield strength. It is recognized that the standard labels, UPE and UIE, are misleading, since maximum load does not necessarily correspond to crack initiation. The tear-yield ratio is similar to the notched tensile strength in that it measures the ratio of the strength to fracture a material with and without a controlled stress concentration.

Figure 1: Schematic of Kahn tear test load versus displacement data.

In addition to mechanical properties, elements of the microstructures, e.g. grain and particulate morphology, and fracture surfaces were characterized and related to the properties where possible. Tension and toughness test results for the Al-Li materials used suggest that although the peak strength indeed improves when indium is added, the Kahn toughness at peak strength decreases. This behavior is in contrast to the case of T8-type treatments, where peak strength increases compared to a T6 treatment, and toughness remains constant or improves, depending on the test method. In order to optimize use of agents like indium we must better understand the specific changes in precipitate type and distribution that are responsible for these observed changes in mechanical behavior. Towards this end, transmission electron microscopy (TEM) has been used here and elsewhere. TEM was used for this work to observe qualitatively the difference between the T_1 (Al_2CuLi) phase amount and distribution in alloys 2090-T6 and 2090+In-T6, where T_1 has been shown to be one of the main strengthening phases in these alloys ¹⁸. Work by L. Blackburn at NASA Langley is being done to discover the mechanisms of this change in precipitation, which could hopefully be used later for materials design ¹⁵. While design prediction is an important long term goal, it is beyond the scope of this work, which focuses on the material properties of the existing materials.

The Al-Mg-Sc materials tested appeared promising in their current conditions, as their strength-toughness combinations at cryogenic temperatures were superior to those of the SPF Al-Li alloys and as good as or better than the 2219-T87 reference material.

2 EXPERIMENTAL PROCEDURE

2.1 Materials

2.1.1 Al-Li Alloys

Composition:

The main alloy of interest was basically 2090+In (Al-Cu-Li-Zr-In), while other alloys were studied for comparison, including 2090, 8090, 2219, and 6061. Material donations included the SPF alloys, from Northrop, 2090-T81, from Alcoa, and 2219-T87, from Martin Marietta.

The compositions of the aluminum-lithium alloys are listed in Table 2. In weight percent, the nominal compositions of 2219 and 6061 are 6.3Cu-0.3Mn-0.2Zr-0.1V-0.1Ti and 1.0Mg-0.6Si-0.3Cu-0.2Cr, respectively.

There were two lots of 2090+In studied. The second lot received has a lower iron and silicon content (these elements are detrimental to both the mechanical properties and the superplastic formability of aluminum-lithium alloys ¹⁹⁻²⁰); furthermore, it has a higher amount of indium than the first lot.

Table 2: Compositions, in weight percent, of aluminum-lithium alloys.

Alloy	Cu	Li	Mg	Zr	In	Fe	Zn
2090-T6	2.61	2.29	<0.01	0.15	--	0.08	0.03
2090+In-T6	2.64	2.22	<0.01	0.20	0.13	0.11	0.03
2090+In#1,2	2.54	2.30	<0.01	0.18	0.21	0.05	<0.01
8090-T6	1.19	2.46	0.72	0.19	--	0.08	0.02
2090-T81	2.86	2.05	<0.01	0.12	--	0.02	--

Processing:

The SPF alloys were cast and processed for superplastic formability at Reynolds Aluminum Company, received from Northrop, and further processed at the Lawrence Berkeley Laboratory.

Effects of the SPF pre-treatment steps have been described by others and will not be repeated here ²¹. The processing began with hot roll homogenization of the ingot to 0.625 in. The plate was then solution heat treated for 30 min. between 538 and 543°C (1000-1010°F) and cold water quenched. Next the plate was overaged for 16 hrs. at 413°C (775°F). Finally, the plate is rolled to approximately 0.125 in. sheet at 0.125 in. per pass. Actual average sheet thicknesses, in inches, are 0.120 for 2090, 0.126 for 8090, 0.122 for 2090+In-T6, and 0.132 for 2090+In #1,2.

After the materials were received, additional thermal treatment was conducted at LBL, including SPF thermal treatment, solutionizing, quenching, and aging to peak or near peak strength. The following paragraphs relate the details of these processes.

The SPF thermal treatment was given only to the second lot of 2090+In (alloys designated #1 and #2), which were held at the forming temperature, 502°C (935°F), for 2 hrs. and then air cooled. This step simulates a common forming thermal treatment. Alloy 2090+In #1 received no further treatment, representing the most economical process examined, which would of course be favored if the properties were acceptable. The remaining materials were solution treated, quenched, and aged as explained in the following paragraphs. Alloy 2090+In #2 was solutionized for 30 minutes at 555°C, 2090+In-T6, for 15 minutes at 555°C, 2090 and 8090, for 15 minutes at 560°C. As described below, all solution temperatures used in this study were determined using differential scanning calorimetry (DSC) and times were determined via metallographic observations and energy dispersive X-ray spectrometry (EDX) of the intermetallic particles.

In DSC, two samples, the material to be studied and a reference, are heated (or cooled) simultaneously, while keeping them at the same temperature. The energy input difference between the sample and reference is monitored. In this way, endotherms and exotherms associated with phase transitions, such as dissolution or melting, can be located. The

solutionizing temperature range, above the precipitate dissolution temperature and below the melting temperature, can thus be determined. Since diffusion is an exponential function of temperature, the solutionizing temperature was chosen close to that of melting, allowing a safety margin of about 15°C for furnace overshooting.

To determine the appropriate solutionizing time and check the temperature chosen from DSC results, particles observed in metallography of the as-received and heat treated samples are compared. For illustration, the details of this experiment follow for 2090. As-received and 15, 30, and 60 min. solutionized samples were mounted and polished to observe intermetallic particles. Metallography of the as-received sample shows two general particle sizes. The smaller particles had vanished after 15 min. of solutionizing; however, the larger particles remained even after 60 minutes. Metallography of the as-received and 15 min. solution treated samples are shown in Figure 1. Since longer solution time results in more lithium loss, solutionizing times were kept between 15 and 30 min.

To further assure that the remaining particles were insoluble in aluminum, EDX was conducted on the different particles and the base material using an Ortec attached to an ISI scanning electron microscope. Observable peaks for the larger particles were those of aluminum, copper, iron, vanadium, and titanium. These are present in aluminum alloys as impurities, all of which form various intermetallic phases ²².

Solution treatment was followed, within a few hours, by aging. Alloys 2090, 8090, and the first 2090+In lot were peak strength aged at 190°C for 16 hrs., a time determined with the Meyer hardness technique. In this method, one measures hardness via a standard technique, such as Rockwell hardness, for a range of d/D (indenter diameter divided by indentation diameter) by varying indenter size and applied load. The method has been used to correlate hardness to strength for aluminum alloys ²³. Alloy 2090+In #2 was aged just

short of peak strength for 72 hours at 160°C, an aging treatment determined by J. Wagner at NASA using Rockwell hardness data ²⁴.

Figure 2: Metallography of intermetallics in the as received versus solutionized conditions.

Materials tested for reference included 6061-T6, 2090-T81, and 2219-T87. Alloy 6061-T6 is a solution treated, quenched, and peak aged material. Alloys 2090-T81 and 2219-T87 are both solution treated, quenched, cold worked, and peak strength aged.

2.1.2 Al-Sc Alloys

Composition:

The investigation of the Al-(Mg)-Sc alloys focused on the variation in the cryogenic properties with temperature and composition for materials already mechanically tested in ambient temperature at Alcoa ¹⁷. Table 3 shows the compositions of the Al-Sc alloys. These were received from Alcoa, and range in composition from 0 to 6 weight percent

magnesium, while the scandium content is approximately constant at 0.5 percent. One material also has about 0.4 percent manganese.

Table 3: Compositions, in weight percent, of aluminum-scandium alloys.

Lot (S) #	Mg	Sc	Mn
504957	--	0.54	--
504952	2.0	0.54	--
504954	4.0	0.56	--
504956	4.0	0.55	0.36
504959	6.0	0.54	--

Processing:

The Al-(Mg)-Sc alloys were processed at Alcoa for superplastic forming. As with the Al-Cu-Li alloys, alloys were tested in the unformed condition. The alloys were cast as 1 in. thick ingots using semi-continuous DC (direct chill) techniques. Ingots were then trimmed to remove solidification defects, warm rolled to 0.25 in. at 288°C (550°F), and then sections were removed from the warm rolled plates and cold rolled to 0.1 in. Aging was then conducted for 4 hrs. at 288°C (550°F). These steps were followed for all but the Al-4Mg-0.5Sc alloy, which received all but the cold rolling step.

2.2 Mechanical Testing

2.2.1 Tensile

Al-Li Alloys:

Each alloy was tension tested twice at 300 and 77 K in the longitudinal orientation. Subsize flat tensile specimens were machined and tested at LBL. Figure 2 details the specimen dimensions used for most tests. The specimens have a one inch gage and samples were taken from the center 0.1 in. of the 0.125 in. sheets. The 2219 and 2090+In #1,2 samples were slightly larger due to a change in the fillet machining method, which was originally done with a mill and later, with a Tensicut (essentially a router with guide blanks made to the specimen size). The larger specimen is 3.55 in. long, 0.75 in. wide at full

width and 0.2 in. at the gage, and just under 1/8 inch thick, while the hole, fillet radius and gage were the same as for the smaller specimens. Alloy 2090-T81 tensile data were taken from previous work at LBL on the T/4 tensile properties of 0.5 in. plate, since the same plate was used for toughness tests ⁷.

Figure 3: Subsized tensile specimen drawing.

All tests were done using a stroke rate of 2×10^{-4} in/s on a hydraulic testing machine, shown schematically in Figure 4. Strain was measured using a clip gage, which is spring loaded to hook onto two pins which are tightened onto the specimen. The testing machine is controlled and output is obtained via an IBM personal computer AT model 5170, and data can be viewed while the test is in progress. For tension, the loading configuration, as shown in the figure is such that the inner 'pull rod' moves upward while the outer 'compression tube' is held still. The specimen is connected to the pull rod and an extension grip which is connected to the compression tube. The tube design allows the specimen to be immersed in a cryostat which holds the cryogen, such as liquid nitrogen or helium.

Properties calculated from the tensile tests included 0.2% offset yield strength, tensile strength, area reduction, and total elongation. Digital data were collected during the test every 0.2 second in the form of load, stroke, and displacement. A Fortran program was written by the author which converts the data to stress and strain, calculates the strengths, elongation, and strain hardening rate, and plots the data at various stages of manipulation, such as the plot of true stress and strain hardening rate versus true strain. Additional

information about the program, along with the Fortran code, is given in Appendix 1. Finally, area reduction was measured with calipers.

Figure 4: Schematic of cryogenic testing facility.

Al-Sc Alloys:

Test procedures for the Al-Sc alloys were identical to those used for Al-Li alloys except for sample thickness, test temperatures, and the number of specimens tested. Sample thickness differed because most of the sheets started at 0.1 in. thick (the thinnest of the Al-Li samples). These specimens were made 0.063 in. thick, taken from T/2. Samples from the Al-4Mg-0.5Sc warm rolled, 0.3 in. plate were taken two from each thickness centered at T/4.

The test temperatures differed since these exact material lots had already been tested at Alcoa at ambient temperature, and because material was in limited supply. Tests were done originally only at liquid nitrogen, and later at liquid helium temperatures. Finally, due to

limited material, only two tensiles could be obtained from each material. At first, each material was tested at 77 K and then after the results were analyzed, three materials were tested at 4 K (Al-2Mg-0.5Sc, Al-4Mg-0.5Sc-0.4Mn, and Al-6Mg-0.5Sc in weight percent). The remaining two tensiles of Al-0.5Sc and Al-4Mg-0.5Sc were tested at 77 K.

2.2.2 Toughness

The Kahn tear toughness tests were conducted to rate the materials studied in this investigation. Alloy 6061-T6 was tested initially to standardize test methods, which were chosen in accordance with information obtained from an internal Alcoa report ²⁵. The procedures used for sample preparation, test setup, testing, and data analysis are outlined in the following paragraphs.

Samples were machined at LBL in the L-T orientation. The specimen, Figure 5, is identical to those in the literature except for the size of the holes, which were altered slightly for the existing test setup. Since tear properties are sample thickness dependent, tear specimens were all 0.063 in. thick, taken from T/2 from the sheet materials, and T/4 of the two plate materials (2090-T81 and Al-4Mg-0.5Sc). The final preparation step was to polish samples to 600 grit prior to testing.

Figure 5: Kahn tear toughness specimen drawing.

As in tension tests, the Al-Li alloys were each tested twice at 300 and 77 K, while the Al-Sc alloys were first tested once each at 77 K. While it is recognized that one sample is not sufficient for reliable tear measurements, lack of material prevented duplication; furthermore, it was felt that the general trend of the Al-Sc materials would indicate whether further investigation would be desirable. As with the tensiles, helium tests were planned for the second specimens of Al-2Mg-0.5Sc, Al-4Mg-0.5Sc-0.4Mn, and Al-6Mg-0.5Sc. Unfortunately, a sample mix-up occurred in the machining process and subsequent hardness tests showed that the Al-0.5Sc rather than the Al-6Mg-0.5Sc specimen had been tested at 4 K. The remaining tears samples were tested at 77 K.

Spacers and careful observation during preloading insured proper sample alignment. Lubrication was not used, as the internal Alcoa report claims that lubrication has no significant effect on properties.

Tests were conducted at a stroke rate of 2×10^{-4} in/s on the same hydraulic testing machine as used for tension tests, Figure 4. The stroke rate, also used for tension tests, was chosen to emulate that used at Alcoa during the initial tear test period; however, tests at Alcoa were conducted in load control while those at LBL were stroke rate controlled, thus this was an approximation. Samples were preloaded to approximately 50 lbs. and the test was stopped when the load dropped below 50 lbs. Digital load and stroke data were collected every 0.2 seconds via the IBM.

A Fortran program, listed in Appendix 2, was used to calculate tear strength and unit initiation and propagation energies. Results were obtained by removing data with loads less than or equal to 50 lbs. No account was taken for the angle of transverse propagation in calculations and data are not discarded if the crack propagates at angles of greater than 10 degrees, as was done at Alcoa. Instead, it is recognized that the UPE of some specimens are elevated and emphasis for comparisons is based on the tear-yield ratio, which is

unaffected by the crack path. The angle of crack propagation off load normal is included in the discussion section.

2.3 Microscopic Analysis

2.3.1 Optical Metallography

Optical microscopy was used to show qualitatively the grain and intermetallic size and distributions of the SPF alloys. Samples of each material were taken from L-S, S-T, and L-T orientations and mounted in Koldmount. They were then polished to 0.06 μm . After 600 grit grinding paper, 6 and 1 μm oil based diamond pastes were used with Kerosene on nylon or paper. The final 0.06 μm polish was done with Mastermet, a liquid suspension that polishes simultaneously mechanically and chemically. Specimens were then etched using Kellers reagent in the proportions 2.5 % HNO_3 , 1.5% HCl , 0.5 % HF , and balance distilled water. For 2090+In #1 and 2, the HF concentration was increased to 1.0 % to better emphasize the grain structure. Photographs were taken with Polaroid film on an inverted Nikon metallograph.

2.3.2 Scanning Electron Microscopy

Fracture surfaces were observed via scanning electron microscopy (SEM). They were then compared qualitatively for variations in temperature and composition, and correlations to microstructure and properties.

The microscope used, located in building 62 of LBL, is an ISI model WB-6 with a tungsten filament. An accelerating voltage of 20 kV and approximate sample height of 20 mm were used. Samples were photographed near the notch at approximately 50x and 800x magnifications to compare lower and higher magnification.

An additional experiment was conducted with the SEM on selected Al-Li materials using EDX to determine if any particular elements, especially indium, in the form of large

intermetallics, were concentrated on the fracture surfaces of these materials. Alloys 2090-T6, 2090+In-T6, 2090+In #2, and 2090-T81 were observed at 300 and 77 K

Two samples were cut from each toughness specimen--one from the specimen bulk, away from the crack, and one from the fracture surface, including the notch. The pieces were approximately 1/4 in. tall. Each piece was then flattened with a hydraulic press to about 1/8 in. high. In order to obtain a smooth surface, the samples were pressed against a piece of carbide that had been polished to 6 μm with diamond paste and then cleaned with ethanol. The samples were then mounted with silver epoxy on an SEM holder. Parameters used included a 30 mm working distance, 20 kV accelerating voltage, 200 seconds real time X-ray collection, 144 x magnification, and analyses near the notch.

Results showed no significant changes in the indium concentration between specimens. Due to the nature of the EDX method in SEM, this negative result only suggests that no large particle differences were associated with the addition of indium. More work, such as Auger electron spectroscopy, would be necessary to determine if indium effects the fracture on a finer scale.

2.3.3 Transmission Electron Microscopy

An experiment was conducted using the transmission electron microscopy (TEM) to determine if the T_1 phase (Al_2CuLi) exhibits a significant qualitative difference in distribution between the 2090-T6 and 2090+In-T6 alloys, processed the same except for the indium addition.

The following study concentrated on the T_1 distribution within the grain interior. The focus was on the $[112]$ zone axis, used to show one T_1 variant edge-on, the $[\bar{1}\bar{1}1]$. Edge-on precipitates were also viewed in the $[110]$ orientation (variants with plane normals $[1\bar{1}1]$ and $[\bar{1}11]$), but these micrographs are not included here as they show identical information regarding the T_1 phase distribution. Dark field images are formed using $\bar{g} = 2/3 (\bar{2}20)$ on

the $[112]$ zone, which contains the $[\bar{1}\bar{1}1]$ and $[111]$ variants, while only the $[\bar{1}\bar{1}1]$ variant is seen edge-on. The $[111]$ variant is in the plane of the sample and, since the precipitates are thin plates, is not usually observable in this orientation, i.e. it does not diffract sufficiently to be visible. It was hoped that analysis using these conditions would be useful in determining differences in T_1 distribution, although it is recognized that there may be a preference for particular variants, recommending further work.

TEM sample preparation began with diamond blade cutting of sheets, 0.016 in. thick, from both 2090 and 2090+In in the long-transverse plane. Slices were ground to about 0.010 in., mechanically punched into 0.12 in. (3 mm) discs, and polished to remove punch burrs. Double jet electro-polishing was conducted using an electrolyte, in volume percent, of 77% methanol, 23% nitric acid, at -30°C . A voltage of 18-20 V and current of 0.16-0.2 A were used for polishing.

Microscopy was conducted on a Phillips 301 using a 100 kV accelerating voltage. Bright field and centered aperture dark field images were taken, and two samples were viewed from each alloy.

3 RESULTS AND DISCUSSION

The main purpose of this work is to characterize and compare the cryogenic mechanical properties and microstructures of several superplastically formable, low density aluminum alloys. The tensile strength and Kahn tear toughness properties were tested and an attempt is made in this section to correlate these with the various microstructures and fracture surfaces observed. This section also contains comments on the relative properties of these materials which are often used to toughness of these materials via the various methods used to distinguish a material as tough or brittle, as explained in the following paragraph.

In addition to the mechanical properties of toughness and ductility, there are many common features used to label a material ductile or brittle. For example via observation of the

fracture mode at low magnification, an oblique sheared surface is 'ductile' while a flat surface may be relatively 'brittle', assuming the fracture was not via ductile dimples. At higher magnification, the fracture is ductile if dimples are visible, while facets indicate a brittle fracture mode. Another classification is based on localized necking, which occurs for ductile materials. This can be seen by eye for extremely ductile materials while for less ductile materials it may be either measured directly from the specimen, seen from a plot of engineering stress versus strain, or in cases where the stress-strain plot appears flat before fracture, determined from whether the work hardening rate (slope of true stress versus true strain), meets the true stress prior to fracture. Labelling a material as ductile or brittle via these various classification systems is ambiguous, as some results are contradictory; thus, results are stated without making assumptions about the materials' relative ductilities.

3.1 Mechanical Testing

3.1.1 Tensile

Al-Li Alloys:

Numerical results from tensile tests done on the superplastically formable aluminum-lithium alloys, plus the reference material 2219-T87, are reported in Table 4. The table includes the test temperature, 0.2 percent offset yield strength, tensile strength, percent total elongation, and area reduction. Data are given for each specimen tested and the averages of the duplicate tests are in italics. Results for 6061-T6 are given in Appendix 3.

Strengths and elongations for the Al-Li alloys are plotted in Figure 6 for 300 and 77 K. The indium addition to 2090 does increase the strength of the T6 condition, as was hypothesized. All strengths increase with decreasing temperature, which is generally true due to reducing thermal vibrations which enhance dislocation motion around obstacles. The strengths of 2090+In #1 (SPF thermally treated only) are probably too low to be

Table 4: Al-Li and 2219-T87 tensile data.

Material	Tensile Specimen ID	Test Temp. (K)	Yield Strength (ksi)	Tensile Strength (ksi)	Total Elong. (%) *	Area Red. (%)
2090+In-T6	2It5-LR	300	60	68	7	11
	2It6-LR	300	58	67	8	12
	average	300	59	67.5	7.5	11.5
	2It7-LN	77	68	76	3	4
	2It8-LN	77	67	79	4	8
	average	77	67.5	77.5	3.5	6
2090+In-#1 (SPF temp.)	I1t1-LR	300	22	37	10	19
	I1t3-LR	300	21	37	10	18
	average	300	21.5	37	10	18.5
	I1t2-LN	77	25	49	25	27
	I1t4-LN	77	26	50	25	27
	average	77	25.5	49.5	25	27
2090+In-#2 (SPF temp., solutionize, peak age)	I2t1-LR	300	53	67	5	12
	I2t2-LR	300	52	67	7	12
	average	300	52.5	67	6	12
	I2t3-LN	77	58	78	7	11
	I2t4-LN	77	61	88	11	12
	average	77	59.5	83	9	11.5
2090-T6	2t2-LR	300	48	62	4	8
	2t4-LR	300	49	61	5	7
	average	300	48.5	61.5	4.5	7.5
	2t6-LN	77	55	77	15	14
	2t7-LN	77	54	77	15	16
	average	77	54.5	77	15	15
8090-T6	8t6-LR	300	48	62	9	10
	8t9-LR	300	49	63	8	8
	average	300	48.5	62.5	8.5	9.0
	8t8-LN	77	55	74	14	13
	8t10-LN	77	54	70	8	10
	average	77	54.5	72	11	11.5
2219-T87	2219t1-LR	300	52	64	12	32
	2219t3-LR	300	49	62	12	34
	average	300	50.5	63	12	33
	2219t2-LN	77	64	85	9	28
	2219t4-LN	77	63	82	12	26
	average	77	63.5	83.5	10.5	27

* 1 in. gage

useful, since indium was added to improve strengths over the 2090-T6 condition. The SPF alloy elongations and area reductions generally increased with decreased temperature, which is observed often in these materials. As it is shown later, the tear toughness is not increased, thus this is a reminder not to rely only on tensile ductility, and perhaps also not on the tear test, for toughness trends in these alloys. In contrast, the elongation and area reduction of 2090+In-T6 decreased significantly from 300 to 77 K; moreover, the average elongation value, 3.5 %, is very low.

From plots of the true stress and work hardening rate versus true strain, it was determined that the localized necking did not occur in most cases for the Al-Li SPF materials. The only case where it does occur in a material containing lithium was for room temperature tests on 2090+In-T6. Conversely, for 2219-T87, both 300 and 77 K samples necked locally.

Also noticeable from the strain versus work hardening rate plot is the consistently higher work hardening rate at 77 K compared to 300 K, a general trend in aluminum alloys ²⁶. Ongoing research at LBL has explored the relationship between work hardening rate and the increase in the strength-toughness combination with decreasing temperature for 0.5 in. 2090-T81 plate ⁷.

Another trend in the SPF Al-Li tension tests was the change in fracture appearance from room to liquid nitrogen temperatures, as observed unmagnified. At 300 K, the fracture surfaces were generally of a single or double oblique surface shear or cup and cone, regardless of whether or not local necking occurred, while at 77 K, the fracture was generally flat. A flatter fracture mode at lower temperature (higher strength) is common in aluminum alloys and represents a shift towards increasing plane stress conditions ²⁷⁻²⁹. The fracture surfaces of 2219-T87, at both 300 and 77 K, exhibited rough cup and cone and shear morphologies. More discussion regarding the fracture surface morphologies will be added in later sections on microscopy.

Figure 6: Elongations, yield, and tensile strengths are plotted for the Al-Li SPF alloys tested at 300 and 77 K.

Apparently, the elongation of 2090+In-T6 and the strength of 2090+In #1 are too low, but 2090+In #2 is an improvement on both these counts. Continued discussion of tensile properties are more appropriately left for after toughness results have been presented.

Al-Sc Alloys:

Al-Sc tensile results are given in Table 5. Results are averaged for those tests that were duplicated, and elongations and strengths at 77 and 4 K are plotted in Figure 7. As with the Al-Li alloys, strengths increase with decreasing temperature. While elongations and area reductions generally either decrease or remain constant from 77 to 4 K, all values were relatively high compared to those of the Al-Li alloys: for example, elongations were all 10 % or greater.

Table 5: Al-Sc tensile data.

Material	Tensile Specimen ID	Test Temp. (K)	Yield Strength (ksi)	Tensile Strength (ksi)	Total Elong. (%) *	Area Red. (%)
Al-0Mg-.5Sc	57t1-LN	77	55	66	14	32
	57t2-LN	77	63	70	10	32
	average	77	59	68	12	32
Al-2Mg-.5Sc	52t1-LN	77	66	82	20	29
	52t2-LH	4	76	95	10	28
Al-4Mg-.5Sc	54t1-LN	77	62	80	14	21
	54t2-LN	77	62	78	22	26
	average	77	62	79	18	23.5
Al-4Mg-.5Sc-.4Mn	56t1-LN	77	72	93	15	19
	56t2-LH	4	78	109	14	22
Al-6Mg-.5Sc	59t1-LN	77	62	83	16	18
	59t2-LH	4	81	109	10	17

* 1 in. gage

Explanations of strength trends as a function of composition and processing follow. The relatively lower strength of the binary Al-Sc alloy is expected because it alone lacks magnesium solid solution strengthening. Solid solution strengthening occurs with the addition of magnesium up to its solubility in aluminum at the solutionizing temperature. At the solutionizing temperature used, 288°C (550°F), embrittling intermetallic Al_xMg_y species form above about 5.5 weight percent magnesium³⁰. These secondary phases are probably responsible for the decrease in strength at 77 K and the similar strengths at 4 K caused by increasing magnesium from 4 to 6 weight percent (Al-4Mg-0.5Sc-0.4Mn to Al-6Mg-0.5Sc). The decrease in strength of Al-4Mg-0.5Sc from Al-2Mg-0.5Sc is due to the difference in processing, where the 4 Mg material was only warm rolled, rather than warm and cold rolled like the other four Al-(Mg)-Sc materials. In summary, the increase in magnesium up to solubility, cold rolling, and decrease in temperature all strengthen the Al-Sc system without causing elongation to become unacceptable. The effect of magnesium and cold rolling on room temperature properties were previously reported at Alcoa¹⁷.

Localized necking does occur for the 0 and 2 % magnesium alloys, and for the Al-4 Mg-0.5 Sc sample 54t2-LN, but not 54t1-LN. This follows the common decrease in ductility with increasing strength due to alloying additions. Serrated yielding occurred in all 4 K tests, also a common feature.

Figure 7: Elongations, yield, and tensile strengths are plotted for the Al-Sc alloys tested at 77 and 4 K.

The unmagnified fracture surfaces of these alloys were all sheared, although the 4 K specimens were rougher. Liquid helium tensiles also showed about 10 Lüders bands each, associated with the serrated yielding that was observed in the stress-strain curves, while orange peel effect (grain rotation)³¹ was observed in the binary and more extremely in the warm rolled 4 Mg specimens, all tested at 77 K only.

Figure 8: Elongations, yield, and tensile strengths are plotted for the Al-Sc, Al-Li, and reference alloys tested at 77 K.

Elongations and strengths are plotted in Figure 8 for the Al-Li, Al-Sc and reference materials 2219-T87 and 2090-T81. Comparison shows that the Al-Sc alloys are generally stronger than the SPF Al-Li alloys. The two highest strength alloys are Al-4Mg-0.5Sc-0.4Mn and 2090-T81. A currently used cryogenic tank material, 2219-T87, is comparable to Al-4Mg-0.5Sc which was only warm rolled, and 2090+In #2, thus there is not likely to be a problem to reach acceptable strengths with these alloys.

3.1.2 Toughness

Both 6061-T6 and 2219-T87 were tested for data comparison and standardization of the Kahn tear test, which had not previously been used at LBL. Methods used were similar to those at Alcoa, as the tear test information was found mostly in two Alcoa documents ^{25,32}. Alloy 6061, inexpensive and easily obtained, was used at first. The results for 6061-T6 tear tests are in Appendix 3. Later, 2219 was tested for a second comparison, specifically because it has been used for the applications of interest for this study.

Comparison of results with Alcoa data shows large differences in the unit initiation and propagation energies (UIE and UPE). For 6061, the UIE obtained at LBL is 74 in-lb/in², or 22%, higher than that obtained at Alcoa, while the UPE is 290 in-lb/in², or 32%, lower, and the tear and yield strengths are about the same for LBL and Alcoa. For 2219-T87, the UIE is 115 in-lb/in², or 52% higher, the UPE is 159 in-lb/in², or 68% higher, the tear strength is 18 ksi, or 26% higher, and the yield strength is 7 ksi, or 12% lower, giving a tear yield ratio 44 % higher than at Alcoa. Variations as a function of test location have been noticed previously. The factors which could contribute to these variations are in the following paragraphs.

Differences in material lot is one possible explanation for property variations ³³. This could have a large effect on material toughness, which is not an alloy specification, thus aluminum manufacturers may improve toughness of a material without renaming it. This factor, which could explain all toughness discrepancies but the large decrease in propagation energy for 6061, is believed to predominate, since other contributions would not effect the tear-yield ratio.

Differences in control mode is another possible cause for discrepancies. Tests at Alcoa were controlled via a manual valve which was set to increase the load at a constant rate up to the point of maximum load. At LBL, stroke control was used, so the strain rate was

kept approximately constant. Strain rate could have an effect on the properties, although the internal Alcoa report claims that various loading rates were tested and data was then taken from the range in which strain rate had no effect; thus, control mode is not a likely cause for the differences.

Another contribution to property variations is the difference in machine compliance. The compression tube for cryogenic testing is long, thus the setup used at LBL is likely to be more compliant than those that are normally used for ambient temperature tests. Higher compliance would increase the initiation and propagation energies, which are obtained from the area under the load-displacement curve, but would not effect the tear strength or tear yield ratio.

A final possible cause for variation in UPE is in the method of analyzing the test. Often, ductile samples sustain a small ligament for long periods of time. Data collection was thus stopped after the load on the specimen descended below 50 lbs. and for analysis, data was cut off if greater than or equal to 50 lbs. At Alcoa, the load-displacement curve was extrapolated to a certain load after the test was ended, where the exact value of the load is not mentioned in the available reports. This extrapolation would increase the propagation energy and is expected to have a greater effect for more ductile materials, such as 6061, thus this might explain the higher UPE reported for 6061 tested at Alcoa.

Al-Li Alloys:

Results from the Kahn tear tests on the Al-Li alloys, plus the reference material 2219-T87, are reported in Table 6. The table includes the test temperature, tear strength, UIE, UPE, and tear yield ratio.

Table 6: Kahn tear toughness data for all Al-Li alloys and 2219-T87.

Material	Kahn Specimen ID	Test Temp. (K)	U.I.E. (in-lb/in*in)	U.P.E.	Tear Strength (ksi)	Tear Yield Ratio
2090+In-T6	2Ik14-LR	300	125	30	53	0.92
	2Ik16-LR	300	173	41	55	
	average	300	149.0	35.5	54.0	
	2Ik17-LN	77	50	20	32	0.48
	2Ik18-LN	77	52	10	33	
	average	77	51.0	15.0	32.5	
2090+In-#1 (SPF temp.)	I1k3-LR	300	158	262	40	1.84
	I1k4-LR	300	152	232	39	
	average	300	155.0	247.0	39.5	
	I1k1-LN	77	249	474	47	1.90
	I1k2-LN	77	275	405	50	
	average	77	262.0	439.5	48.5	
2090+In-#2 (SPF temp., solutionize, peak age)	I2k3-LR	300	226	144	66	1.22
	I2k4-LR	300	200	132	62	
	average	300	213.0	138.0	64.0	
	I2k1-LN	77	103	29	54	0.93
	I2k2-LN	77	98	54	57	
	average	77	100.5	41.5	55.5	
2090-T6	2k23-LR	300	141	54	53	1.10
	2k24-LR	300	146	123	54	
	average	300	143.5	88.5	53.5	
	2k25-LN	77	107	60	54	1.04
	2k26-LN	77	155	65	59	
	average	77	131.0	62.5	56.5	
8090-T6	8k25-LR	300	133	163	50	1.01
	8k27-LR	300	133	122	48	
	average	300	133.0	142.5	49.0	
	8k26-LN	77	131	125	54	1.03
	8k29-LN	77	136	112	58	
	average	77	133.5	118.5	56.0	
2090-T81	2-8k1-LR	300	264	163	67	0.98
	2-8k2-LR	300	176	111	62	
	average	300	220.0	137.0	64.5	
	2-8k3-LN	77	278	217	72	0.98
	2-8k4-LN	77	237	219	77	
	average	77	257.5	218.0	74.5	
2219-T87	2219k3-LR	300	347	348	73	1.46
	2219k4-LR	300	327	374	74	
	average	300	337.0	361.0	73.5	
	2219k1-LN	77	359	376	86	1.39
	2219k2-LN	77	349	411	90	
	average	77	354.0	393.5	88.0	

The tear test load for most Al-Li materials (8090-T6, 2090-T6, 2090+In-T6, and 2090-T81) drops abruptly after crack initiation, indicating rapid crack propagation. This generally undesirable behavior is, as expected, in agreement with the low UPE toughness values. The same plots for the 2090+In #1 and 2219-T87 exhibit much tougher behavior, as the crack propagates gradually after initiation, and the curves are smooth. In general, except for variations in the area under the curves with change in temperature, the curves were similar for any particular material as a function of temperature.

As with the tensile fracture surfaces, the unmagnified tear surfaces showed shearing for 300 K and flat surfaces for 77 K tests. Cracks of some specimens traveled at large angles off load normal, thus the propagation energy values for these specimens are elevated. Alloy 2090+In #2 was the worst such case, subtending an angle of 15° for sample I2k1-LN, having a midway jog of about 15° for sample I2k2-LN, and a 45° angle for both 300 K samples. A 2090-T6 sample, 2k24-LR, and an 8090-T6 sample, 8k25-LR, both had cracks 30° off load normal. Because of this problem, plots of strength versus tear toughness shown later use the tear-yield ratio rather than the UPE. Further discussion of the tear results is left for the following section, which combines strength and toughness.

The unmagnified fracture surfaces of 2090-T81, at 300 and 77 K, were rough, delaminated, and straight, except for sample 2-8k1-LR, which jogged 20° for the first half of the specimen fracture path, while those of 2219 were straight and sheared at 300 and 77 K.

Al-Sc Alloys:

Al-Sc Kahn tear toughness data is shown in Table 7. Toughness values generally increased with decreasing temperature; furthermore, for the cases in which it decreased, toughness was still high relative to the Al-Li materials tested.

The load-displacement curves are generally smooth for these alloys. The manganese containing alloy did show some discontinuity as load dropped. Also, the binary Al-Sc alloys shows a load drop at 4 K but not at 77 K; however, the 4 K toughness energy data were higher.

Table 7: Kahn tear toughness data for the Al-Sc alloys.

Material	Kahn Specimen ID	Test Temp. (K)	U.I.E. (in-lb/in*in)	U.P.E.	Tear Strength (ksi)	Tear Yield Ratio
Al-0Mg-.5Sc	57k1-LN	77	647	917	97	1.64
	57k2-LH	4	1500	1163	119	-
Al-2Mg-.5Sc	52k1-LN	77	801	730	106	1.61
	52k2-LH	4	905	1019	119	1.57
Al-4Mg-.5Sc	54k1-LN	77	892	892	109	
	54k2-LN	77	753	767	111	
	average	77	823	830	110.0	1.77
Al-4Mg-.5Sc-.4Mn	56k1-LN	77	434	282	93	1.29
	56k2-LH	4	509	145	96	1.23
Al-6Mg-.5Sc	59k1-LN	77	312	318	80	
	59k2-LN	77	298	309	83	
	average	77	305	314	81.5	1.31

The fracture paths of these specimens were all straight, while the fracture surfaces varied. The binary alloy fractured via a rough shear at both 77 and 4 K. The Al-2Mg-0.5Sc alloy fractured via shear at 77 and 4 K. This was macroscopically smooth for the 77 K specimen and rougher for the 4 K test. The Al-4Mg-0.5Sc material, tested only at 77 K, showed a rough shear similar to that of Al-2Mg-0.5Sc at 4 K. The strongest material, Al-4Mg-0.5Sc-0.4Mn, exhibits less macroscopic shearing: at 77 K, the surface is predominantly flat and rough with a slight shear angle; furthermore, at 4 K the surface is completely flat and shows some delamination. Al-6Mg-0.5Sc, tested only at 77 K, also showed some delamination, along with roughness and a slight shear angle

Tear-yield ratios above 1.0 indicate that, in the presence of a blunt flaw, the material will yield prior to tearing; thus, a tear-yield ratio of less than 1.0 is generally undesirable. All

the Al-Sc materials exhibit tear-yield ratios well above 1.0 at both 77 and 4 K, while the Al-Li alloys have values below or very close to 1.0. The tear toughness values of all the Al-(Mg)-Sc materials are in general exceptional; therefore, although only one to two specimens were tested in each condition, the overall results indicate that the Al-(Mg)-Sc system has more than adequate cryogenic tear toughness.

3.1.3 Strength-Toughness

In evaluating the strength and toughness of materials, it is valuable to compare the properties together, as they are often coupled, such that a change in one property affects the other. The strength-toughness combinations are explored in the following section via plots of tear-yield ratio versus yield strength. The plots contain lines connecting average data from each test condition. These lines are for visual aid and do not indicate a linear relationship. In all cases, yield strength increases with decreasing temperature, thus, higher strength points correspond to lower temperature tests. There are two points for each Al-Li alloy, tested at 300 and 77 K, and two to three points for the Al-Sc data, for 300 and 77 K tests or 300, 77, and 4 K tests. The 300 K points are from the same lots of material and tested at Alcoa.

Al-Li Alloys:

A broad view of the Al-Li results, Figure 9, suggests that the materials could be on a strength-toughness trade-off line as a function of composition, processing, and in some cases, test temperature. If this is true, variations of this sort will give predictable combinations of strength and toughness; thus, one could decide without further variations whether or not these types of alloys are useful for a desired application as soon as design requirements are known. Conversely, designers could attempt to work up a design based on properties on the given trend line.

Figure 9: Yield strength versus tear-yield ratio for SPF Al-Li alloys.

Alloys 2090-T6 and 8090-T6 are slightly above the tear-yield ratio of 1.0. Alloy 2090+In-T6 is below 1.0 at both temperatures and 2090+In #2 is below at 77 K. Finally, 2090+In #1 is extremely tough, however, its strength is probably too low, since indium was added to increase the strength over that of 2090-T6. According to the tear results, an intermediate strength of the 2090+In alloys would probably be most desirable. Finally, the addition of indium does increase strength but not the strength-toughness relationship as a function of

temperature; in fact, it appears to degrade the relationship for the high strength conditions tested in this work.

Figure 10: Yield strength versus tear-yield ratio for SPF Al-Sc alloys.

The strength toughness plot of the Al-Sc materials is in Figure 10. With decreasing temperature, some of the strength-toughness combinations increase, while others decrease,

but all of the tear-yield ratios are well above 1.0. From these data, the best material choice would probably contain 4-6 weight percent magnesium, since all the toughness appear acceptable and density reduction is the main goal.

Al-Li and Al-Sc:

The combined strength-toughness data of Al-Li and Al-Sc alloys, along with 2219-T87 are shown in Figure 11. The bands represent the envelope around the 300 and 77 K data for each alloy. The Al-Sc system has a generally superior strength-toughness combination compared to the Al-Li SPF materials, as the bands do not even overlap. In addition, the Al-Sc alloys compare favorably with 2219-T87, while the tested conditions of the Al-Li alloys do not.

Figure 11: Strength-toughness comparison of Al-Li, Al-Sc and 2219-T87.

A final plot is included in this section to show possible plane-stress toughness values of the SPF alloys. Correlations between tear and fracture toughness have been found empirically to be linear for some ranges³². Figure 12 is a plot of the tear toughness versus K_c fracture

toughness of 1/16 in. thick specimens. The data for 2xxx and 7xxx series alloys was taken from the Alcoa report that discusses similar correlations. Fracture toughness data were from of 16 in. wide, center-cracked panel, fatigue precracked specimens. A line is constructed from this data, which fits with little scatter, and the tear-yield ratios of the SPF alloys are placed on this line. Al-Li alloys have been shown to exhibit greater notch sensitivity than conventional aerospace alloys ³⁴, such as those used in the plot, thus decreasing the confidence in these numbers. More work would certainly need to be done to determine if this empirical correlation holds for Al-Li and Al-Sc alloys, but it is perhaps interesting to see what the K_c values would be if the correlation does hold.

Figure 12: Tear-yield ratio versus K_c fracture toughness.

3.2 Microscopic Analysis

3.2.1 Optical Metallography

Al-Li Alloys:

Thermomechanical processing caused varying degrees of recrystallization in the Al-Li superplastically formable alloys. Figure 13 of the 8090-T6 LS cross section, also representative of 2090-T6 and 2090+In-T6, shows all small grains in the outer half of the sheet, whereas the center material is composed of small and large grains. The second lot of 2090+In, from which the two SPF thermal treatments were taken, was recrystallized more equally throughout the thickness, with only an occasional large grain. This lot also had a very thin layer of larger grains on the surfaces that is likely to be from solute depletion, as has been shown by others³⁵⁻³⁶. The microstructures of all the materials are shown at higher magnification in Figure 14.

Figure 13: Metallography of 8090-T6 (LS orientation) shows grain size variation.

Figure 14: Metallography of superplastically formable Al-Li alloys.

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Figure 15: Metallography of grains and intermetallics in SPF Al-Li alloys.

Photographs were taken at 400x, and are shown in Figure 15, to document qualitatively the relative amount of intermetallics. The only significant difference is the much greater amount found in 2090+In #1, which was not surprising since it was the only material that did not received a solution heat treatment.

Al-Sc Alloys:

With increasing magnesium, an increase in the number of Al-Mg intermetallics is expected. Figure 16 shows optical micrographs from Al-0.5Sc, Al-4Mg-0.5Sc, and Al-6Mg-0.5Sc in the polished condition. Al-4Mg-0.5Sc showed an increase in the number of intermetallics, while Al-6Mg-0.5Sc intermetallics increased in size and number. The three materials, Al-0.5Sc, Al-2Mg-0.5Sc, and Al-4Mg-0.5Sc-0.4Mn, showed no significant difference in the intermetallic amount or size. The high magnesium alloy revealed large etch pits, sometimes polygonal shaped, such as triangles and rectangles. These are thought to be particles rather than dislocation etch pits, since in the observation shown at the bottom of Figure 16, two of these features appear to have broken from one, e.g. during the rolling operations. In addition, etch pits generally form in self similar shapes on closest packed (i.e. lowest surface energy) planes.

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Figure 16: Relative intermetallic morphologies of the Al-(Mg)-Sc alloys.

Specimens polished and etched show the generally large, irregular grain structure of these alloys, which can be seen by three perpendicular orientations of the binary Al-0.5Sc in Figure 17. This was similar to micrographs shown by Alcoa ¹⁷. Etching also highlights fabrication deformation, which appears in two general forms and is shown in Figure 18. One form is more angular than the other and predominated in the Al-0.5 Sc binary alloy. The other, wavier form was predominate in both Al-2Mg-0.5Sc and Al-4Mg-0.5Sc. Both deformation morphologies appeared in Al-4Mg-0.5Sc-0.4Mg and Al-6Mg-0.5Sc. The angular deformation was the less uniform and, in the binary alloy micrograph, the region photographed was one of the few areas with visible deformation. In contrast, the other materials showed more uniform deformation (generally advantageous for properties) from the more finely spaced, wavier deformation.

Figure 17: Optical metallography of the Al-0.5Sc alloy.

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Figure 18: Two morphologies of fabrication deformation in the Al-Sc alloys.

3.2.2 Scanning Electron Microscopy

Scanning electron microscopy (SEM) was used to document the fracture surface morphologies. In the following section, fractographs taken at relatively lower ($\cong 50\times$) and higher ($\cong 800\times$) magnifications are presented and discussed with respect to properties, processing, and microstructure. Whenever pop-in (the plane-strain fracture at the notch) was observed, high magnification photographs shown were taken from within this region. Specimens without pop-in were photographed in regions similarly close to the notch, which in all cases is at the top edge of each SEM micrograph as shown.

Pop-in and delamination, two features of discussion, require some explanation. The transition from pop-in with shearing at room temperature to flatter fracture surfaces with decreasing temperature is common in aluminum and other alloys and is attributed to a shift from plane stress towards plane strain²⁷⁻²⁹. The stress state is a function of yield strength, which increases as temperature decreases, causing the transition. The second feature,

delamination of grains perpendicular to the direction of crack propagation, may contribute positively to the fracture properties^{12,13,37,38}. Delamination is also caused by the increased yield strength because the resulting increase in through thickness constraint causes stresses in the through thickness direction to reach grain boundary fracture strength.

Al-Li Alloys:

The fracture surfaces of the Al-Li materials are shown in Figures 19-24 and 2219-T87 is in Figure 25. The fracture mode of 2090-T6, Figure 19, is tougher in appearance at 300 K than at 77 K. Pop-in and shear failure occurred at 300 K, while the 77 K fracture is flat with very little delamination. This relationship is accompanied by a slight decrease in tear toughness. At higher magnification, relatively equiaxed features on the order of the grain size indicate a predominantly intergranular fracture at both 300 and 77 K. In addition, the 77 K specimen exhibits some rough features (upper left corner of 25 μm photograph) which might be transgranular.

Alloy 2090+In-T6, Figure 20, has similar low magnification surfaces to 2090-T6, except for more delamination at 77 K. This fracture mode transition is accompanied by a large decrease in toughness with decreasing temperature. Delamination is mostly observed in the 77 K tests. The higher magnification photos of 300 and 77 K show some fine grain intergranular features, some larger, delaminated features, and much of the rough mode that was observed in 2090-T6 at 77 K.

Alloy 2090+In #1, Figure 21, has the finest, least delaminated fractures of all the SPF Al-Li alloys. This material also shows pop-in only at 300 K, but tear toughness in this case increases as temperature decreases. This can be attributed to the entirely different fracture, void coalescence, of this alloy. Voids form around particles and join to form the fracture surface by deforming, which may be more difficult at lower temperature, since the yield strength is higher. The observed mode is not surprising considering both the much greater

number or intermetallic particles in this material, due to lack of solution heat treatment, and the softness, due to lack of artificial aging.

Figure 22, of 2090+In #2, also shows pop-in only at 300 K, and flat, delaminated fracture at 77 K. The higher magnification photographs at both temperatures show a fine intergranular fracture much like that of 2090-T6. The fracture transition is again accompanied by a decrease in tear toughness with temperature.

Fracture surfaces of 8090-T6, Figure 23, appear more directional than those of the other SPF Al-Li materials. Pop-in again occurs only at 300 K, and delamination was extensive at 77 K. Higher magnification shows some fine intergranular features and some larger, flatter features reminiscent of the intergranular / transgranular fracture in 2090-T81 plate. Tear toughness remains approximately constant with temperature, possibly due to a balance in the contributions from delamination and fracture mode, which operate in opposite directions.

Figure 24 shows the fracture surfaces from 2090-T81. Features were similar to those in the J_{ic} specimens, with the directional trans- and intergranular fracture⁵. Both 300 and 77 K fractures were flat and delaminated, with more delamination at 77 K. The flat fracture at both temperatures is probably due to the relatively higher strength of this alloy at both 300 and 77 K, giving more plane-strain characteristic at both temperatures than the room temperature SPF Al-Li alloys. The propagation energies increase slightly with decreasing temperature, while the tear-yield ratio remains constant. This is in contrast to the J_{ic} fracture toughness which increases dramatically from 300 to 77 K⁵, however it agrees with Charpy impact energy results, which remained constant in this temperature range³⁹. This similarity between Kahn and Charpy results, which use samples of such different thickness, suggests that the difference in the trend may be due to the difference in notch

sharpness, since the Charpy and Kahn samples have machined notches while the J_{1c} has a fatigue precrack.

The fracture surfaces of 2219-T87 are shown in Figure 25. Both 300 and 77 K show pop-in and no delamination. Higher magnification shows fracture to be via linking of ductile dimples.

In summary, the SPF Al-Li materials generally exhibit pop-in with shearing at 300 K and flat fractures with varying amounts of delamination at 77 K. The surfaces at high magnification showed significant variation with composition, but not with temperature. At both 300 and 77 K, alloy 2090-T81 was flat and delaminated and 2219-T87 shows pop-in and ductile dimple failure. The changes in properties with temperature are discussed in the proceeding section.

Al-Sc Alloys:

Fractographs of the Al-(Mg)-Sc alloys are in Figures 26-30. The Al-0.5Sc binary alloy, Figure 26, fractured via void coalescence with pop-in at both 77 and 4 K, however the surface was rougher at 4 K. The propagation energies increase with decreasing temperature, which could be, as described above for 2090+In #1, due to the increase in yield strength causing more difficulty for void linking.

The Al-2Mg-0.5Sc alloy fractures, Figure 27, appear brittle relative to Al-0.5Sc, but at 77 K, fracture is still at least partially via void coalescence. The surfaces exhibit pop-in at 77 K and perhaps at 4 K, while delamination was absent at 77 K and extensive at 4 K. Tear toughness increases in propagation energies and remains approximately constant in tear-yield ratio with decreased temperature. The increase in propagation energies is possibly due to the delamination at 4 K.

Al-4Mg-0.5Sc was warm rolled only. Its 77 K fracture surface, Figure 28, is similar to that of Al-2Mg-0.5Sc, i.e. to an alloy of lower magnesium content with cold working; however, the tear properties of the warm rolled material are slightly higher. The surface has pop-in and some delamination, and has some possible void formation.

Figure 29 shows the fracture surfaces of the Al-4Mg-0.5Sc-0.4Mn alloy, which is extensively delaminated at both 77 and 4 K, with more delamination at 4 K. The fracture surfaces appear brittle relative to all the other Al-(Mg)-Sc fractures, and the UPE and tear-yield ratios are the lowest of these materials. This material should contain the greatest volume fraction of embrittling intermetallics excepting the 6 % magnesium containing alloy.

Finally, the 77 K fracture of Al-6Mg-0.5Sc, Figure 30, is flat and delaminated at low magnification, but failed with some void coalescence, as seen at high magnification. This is probably due to the large intermetallics formed only in this alloy, since it had the highest alloying content. The tear properties are similar to those of Al-4Mg-0.5Sc-0.4Mn.

The increase in alloying additions and decrease in temperature generally decreased the tear toughness of the Al-(Mg)-Sc alloys, accompanied by fracture mode changes. However, it is important to remember that all the tear properties are higher than those of the Al-Li SPF alloys and similar to or better than those of 2219-T87.

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Figure 19: SEM fractographs of 2090-T6 Kahn tears.

XBB 892-873

Figure 20: SEM fractographs of 2090+In-T6 Kahn tears.

XBB 892-875

Figure 21: SEM fractographs of 2090+In #1 Kahn tears.

XBB 892-874

Figure 22: SEM fractographs of 2090+In #2 Kahn tears.

XBB 892-872

Figure 23: SEM fractographs of 8090-T6 Kahn tears.

XBB 892-880

Figure 24: SEM fractographs of 2090-T81 Kahn tears.

XBB 892-876

Figure 25: SEM fractographs of 2219-T87 Kahn tears.

XBB 892-870

Figure 26: SEM fractographs of Al-0.5Sc Kahn tears.

XBB 892-869

Figure 27: SEM fractographs of Al-2Mg-0.5Sc Kahn tears.

XBB 892-866

Figure 28: SEM fractographs of Al-4Mg-0.5Sc Kahn tears.

XBB 892-867

Figure 29: SEM fractographs of Al-4Mg-0.5Sc-0.4Mn Kahn tears.

XBB 892-868

Figure 30: SEM fractographs of Al-6Mg-0.5Sc Kahn tears.

3.2.3 Transmission Electron Microscopy

The distribution and amount of the T_1 phase (Al_2CuLi) was compared for alloys 2090-T6 and 2090+In-T6 to determine if changes in the amount and/or distribution were contributing to the observed increase in strength with the indium addition. The amount of T_1 phase within the grain interiors can be determined directly through T_1 imaging or indirectly via diffraction patterns. T_1 superlattice spots were difficult to locate in 2090-T6 and much easier to find in 2090+In-T6 samples, an indication that the amount of T_1 was greater in 2090+In-T6. Bright field-dark field pairs showed that 2090+In-T6, Figure 31, indeed contains a greater amount of intragranular T_1 phase than does 2090-T6, Figure 32, and the phase is distributed more uniformly in the indium containing alloy. This coincides with and explains, at least in part, the greater strength of the indium containing material.

To complete an investigation of this type, all variants of T_1 and grain boundary precipitation should also be considered. Other investigations have also found ⁴⁰, as shown in Figure 32, that T_1 populates the grain boundaries of 2090-T6. Inter- versus intragranular precipitation should be the subject of future work with the indium containing material being compared to 2090-T6 and T8. In addition, the distribution of other phases, such as δ' (Al_3Li), β' (Al_3Zr), and especially θ' (Al_2Cu), another main strengthening phase in these alloys, could have effects on the 2090+In properties and should be studied. Some of this has been done by L. Blackburn at NASA Langley, with similar results for T_1 and θ' ¹⁵.

XBB 886-6262

Figure 31: Bright field-dark field pair on the $[112]$ zone axis shows evenly distributed T_1 plates in 2090+In-T6.

XBB 886-6263

Figure 32: Bright field-dark field pair on the $[112]$ zone axis shows unevenly distributed T_1 plates in 2090-T6.

4 MECHANISTIC DISCUSSION

Due to the large amount of data and detail presented herein, the following section is included to generalize and discuss possible mechanisms for the strength and toughness behavior.

In accordance with general strengthening theory, flow stress increases both as a function of increasing number and effectiveness of obstacles to dislocation motion ^{41,42}. Strength was observed to increase with increasing magnesium in the Al-Mg-Sc alloys and with the addition of indium to the Al-Li alloys, which caused an improvement in distribution of strengthening precipitates.

The increasing strength observed with decreasing temperature was common to all the materials tested. Strengthening mechanisms, i.e. dislocation-obstacle interactions, will be enhanced as temperature decreases, since thermal vibrations decrease, making it more difficult for dislocations to bypass obstacles. Obstacles that may be too small to impede dislocation motion at one temperature may be effective at a lower temperature.

The toughness trends will be discussed in slightly more detail, since much research has been done to determine the mechanisms for the improvement in fracture toughness with decreasing temperature observed in some aluminum-lithium alloys. The main factors--other mechanical properties, primary fracture mode, and delamination--which might contribute to the toughness behavior obtained are reviewed below and their effects discussed.

The various theories of strain-controlled fracture predict an increase in plane-strain fracture toughness with increasing yield strength, elongation, elastic modulus, and strain hardening rate ^{11,43}. In this study, all of these properties generally increase with decreasing temperature for the Al-Li alloys, and all but the elongation increase in the Al-Sc alloys;

thus, a toughness increase might be expected with decreasing temperature as long as the primary fracture mode is strain-controlled and unchanged. Toughness trends of the Al-Sc alloys, which do not clearly change fracture mode with changing temperature, might be explained by this mechanism. However, the magnesium containing alloys tested at two temperatures also exhibit delamination, complicating interpretation. The Al-Li alloys are also affected by their changing properties, which cause a change in the macroscopic fracture mode and complicate interpretation for these alloys as well.

An increase in the yield strength and strain hardening rate may cause the primary fracture mode (as distinguished from secondary cracking such as delamination perpendicular to the primary crack) to change to a lower toughness mode. This may occur microscopically or macroscopically. An example of a microscopic transition occurs when void coalescence is characteristic at a higher temperature and transgranular or intergranular cleavage, at a lower temperature. The transitions observed in this study occur on the macroscopic level. With decreasing temperature (increasing strength, etc.) the stress state moves towards plane-strain conditions, causing the fracture to change from 45° shearing to a flat surface. This phenomenon was observed in all the Al-Li SPF alloys, which generally exhibited 45° shear surfaces at 300 K and flat surfaces at 77 K. Thus the change in stress state probably dominates the toughness behavior of the high strength Al-Li alloys, which is generally constant or decreasing with decreasing temperature.

The third factor, intergranular delamination perpendicular to the primary fracture path, probably increases with decreasing temperature (increasing flow stress) because the increasing fraction of plane-strain fracture allows through thickness stresses to ascend beyond the grain boundary fracture stress and/or because the grain boundary fracture stress decreases ¹¹. This could contribute to material toughness by decreasing the effective thickness of the sample, i.e. moving towards plane-stress conditions ^{12,13,37,38}.

Delamination generally increases slightly in the Al-Li materials with decreasing temperature--in most cases delamination is absent at the higher temperature and present at the lower temperature--but toughness generally remained constant or decreased; thus, if delamination had an effect, it was counteracted by the change in macroscopic fracture mode due to the change in stress state. In addition, the amount of delamination, and thus effect on toughness of this mechanism, is expected to be less in the SPF alloys than in 2090-T81 plate studied because the difference in microstructures. The SPF Al-Li alloys are composed primarily of recrystallized, equiaxed grains, while 2090-T81 has elongated, flat grains. The amount of delamination could be correlated to the changes in tear toughness of the magnesium containing Al-Sc materials; however, delamination was extensive and might mask changes in primary fracture mode.

In summary, the strength increases observed, with decreasing temperature and increasing number and improved distribution of strengthening phases, are predictable via strengthening theory. The toughness trends, some increasing and some decreasing, are less well understood. The material properties, fracture mode, and delamination probably act together in most cases, making it difficult to determine the relative importance of these mechanisms. However, for most of the materials tested in this work, it is thought that the increase in mechanical properties, such as strength and strain hardening rate, are mainly responsible for the fracture behavior as a function of temperature. This effect may occur either directly, as predicted by fracture theories, or indirectly, by changing the stress state and thus the macroscopic fracture mode.

5 CONCLUSIONS

From this study, it is impossible to be certain whether the materials tested will be useful for cryogenic tank applications, mainly because design criteria have not yet been disclosed. Even if the criteria were known, tension, fracture toughness, and fatigue properties of the superplastically formed material, at minimum, should be tested in order to determine whether a large scale material study would be logical. In addition, economical aspects of these materials with respect to existing or other new materials, such as material cost increase versus production and operation cost savings, must also be addressed. These issues are beyond the scope of this study, which focuses on rating the conditions tested.

For designs where high stiffness or low density are most important, the Al-Li alloys are superior. Additions of indium increased the strength of these alloys and decreased the Kahn tear toughness; furthermore, the materials studied do not compare favorably with 2219-T87 in their strength-toughness combinations. On the contrary, the indium containing Al-Cu-(Mg)-Li alloys have not been optimized, so more promising properties may be obtainable, while designs which take advantage of the SPF characteristics may be able to accept their lower strength or toughness.

In contrast to Al-Li, the Al-Sc materials compare favorably with 2219-T87 in strength-tear toughness combinations. These materials would thus be favorable if strength and/or toughness were emphasized in design. All the Al-Sc materials had excellent tear toughness, thus, since density reduction is desired, the 4 and 6 weight percent alloys are most promising.

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8 APPENDICES

8.1 Appendix 1: Fortran code for tensile data analysis

The following appendix gives the Fortran code used for tensile data analysis and plotting, called Tenplt. This program, written primarily by the author, works on an IBM personal computer. Only the comments have been modified from the operating program.

The program takes load and strain (or stroke) data from the tensile test file and calculates the yield strength, tensile strength, elongation, and work hardening rate. Only the strain portion of this program has been thoroughly tested, as stroke data were not used for analysis. Plotting routines, written in Halo, were inserted between each major data manipulation. Each plot includes the newest manipulated set of data with the previous data. The plots were used both for debugging and for determination of the linear range of strain from which to zero the plot, and calculate the yield strength and elongation. Plots could be printed to a dot matrix printer which was used primarily for recording the work hardening rate curves.

```
c PROGRAM TENPLT
```

```
c
```

```
c Comments:
```

```
c   Raw data is recorded in 5 columns:
```

```
c       1. time, 2. load, 3. stroke, 4. strain, 5. valve current.
```

```
c   Five Halo stress-displacement plots show data and how it is modified:
```

```
c       1. original data, except for stress < 0
```

```
c       2. original + data averaged over displacement interval
```

```
c       3. zeroed averaged data + calculated modulus
```

```
c       4. averaged + smoothed data (only if data is smoothed)
```

```
c       5. averaged (smoothed or not) + strain hardening rate
```

```
c   Averaging occurs in x (strain, stroke) and y (load).
```

```
c   Modulus is computed over a range specified by user.
```

```
c   Each smoothing operation drops a beginning and end point,
```

```
c       e.g. smoothing 10x deletes 20 data points.
```

```
c
```

```
c
```

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