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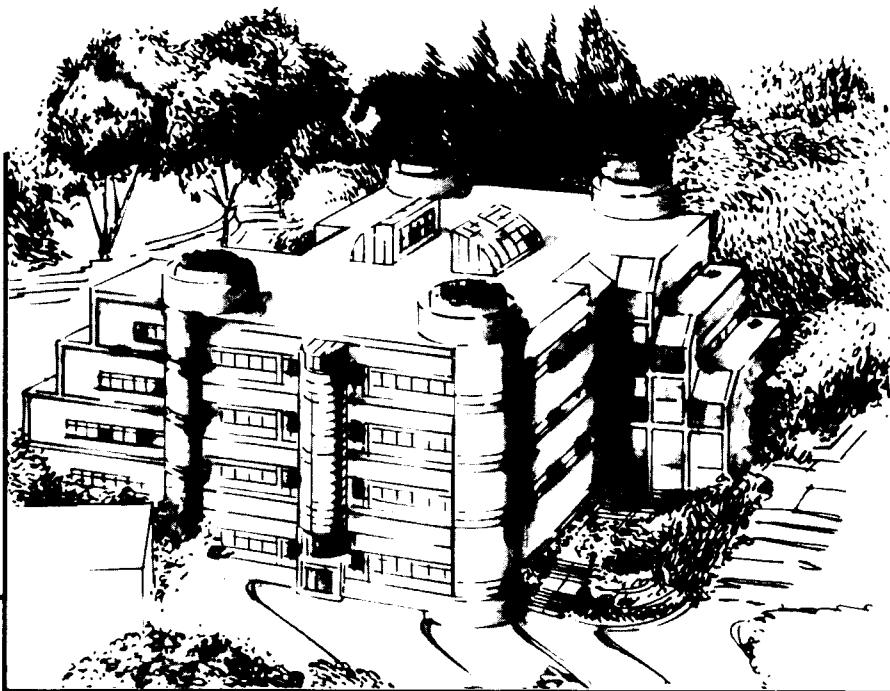
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Superplastic Al-Mg-Sc Alloys**

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# CRYOGENIC MECHANICAL PROPERTIES OF LOW DENSITY SUPERPLASTIC AL-MG-SC ALLOYS

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## ABSTRACT

Spacecraft cryogenic fuel tankage made from superplastic materials is a possible new application for low density aluminum alloys such as Al-Mg-Sc. Examples from this alloy system were examined for cryogenic strength and toughness. Alloys studied were received in the superplastically formable condition, in sheet form. Alloy 2219-T87 sheet was also tested for comparison, since 2219-T8X is currently used in cryogenic tankage. Five compositions of Al-Mg-Sc alloys were tested at 77 and 4 K. Alloys showed the expected increase in strength with decreasing temperature, accompanied by a general slight decrease in elongation and the Kahn tear-yield ratio toughness indicator; however, the strength-tear toughness relationship of this alloy class was as good as or better than that of 2219-T87. Correlations found between the properties, microstructure, and fracture surfaces are discussed.

## INTRODUCTION

In order to reduce energy costs for transportation vehicles, materials researchers are called upon to help reduce vehicle weight by discovering lighter, stronger, stiffer, more damage tolerant 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 weight savings via lowering alloy density, superplastically formed structures can be more efficient than conventional machined structures because they reduce material waste, decrease forming energy needs, and allow more complicated designs that support more load for a given weight <sup>1</sup>.

Cryogenic mechanical properties of superplastic Al-Mg-Sc alloys are the focus of this paper. For comparison, alloy 2219-T87 was also tested, since 2219-T8X is currently

used in cryogenic tankage of the space shuttle. Al-Mg-Sc materials can provide a significant density reduction over alloy 2219: the nominal densities of 2219 and Al-4Mg-0.5Sc (wt. %) are 2.72 and 2.65 g/cm<sup>3</sup>, respectively.

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 <sup>2,3</sup>, and by the hypothesis that these alloys would remain exceptional at cryogenic temperatures, due to their strengthening mechanisms. These materials are strengthened directly both by small, spherical, coherent, Al<sub>3</sub>Sc precipitates, and by magnesium in solid solution. An additional component of strengthening is derived indirectly from grain structure refinement promoted by the Al<sub>3</sub>Sc precipitate, which has been shown to effectively pin grain boundaries <sup>3</sup>.

The main purpose of this work was to characterize the cryogenic strength and toughness of several Al-Mg-Sc alloys. Candidate alloys were tested in the unformed condition. The properties of formed parts should be the subject of near term future research; however, it was felt that in the absence of formed material, the properties of unformed material would present valuable information. In addition to mechanical testing, the microstructures and fracture surfaces were characterized and related to these properties where possible.

Toughness characterization was done using an indicator test, the Kahn tear, which is not often used; thus, background for the choice and results of this test are included below. The specimen was chosen mainly for two reasons. First, wide specimens, such as the center cracked panel, are problematic since the cryogenic test facility available for 4 K testing is three inches in diameter and since material was limited in most cases. Second, the choice of the Kahn tear over a notched tensile test was made primarily because of the large existing cryogenic data base on aluminum alloys tested with the tear method <sup>4</sup>.

During the tear test, load versus displacement data are collected. Three toughness indicators follow from the test: 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 percent 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.

## EXPERIMENTAL PROCEDURE

### Composition and Processing

The investigation of the Al-Mg-Sc alloys focused on the variation in cryogenic mechanical properties with temperature and composition for materials already tested at ambient temperature at Alcoa <sup>3</sup>. Table 1 shows the compositions of the Al-Mg-Sc alloys,

which range 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 1: Compositions, in weight percent, of Al-Mg-Sc alloys.

| I.D. (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 |      |

Materials were processed at Alcoa <sup>3</sup> for superplastic forming and tested at LBL in the unformed condition. The alloys were cast as 2.54 cm (1 in) thick ingots using semi-continuous DC (direct chill) techniques. Ingots were then trimmed to remove solidification defects, warm rolled to 8 mm (0.3 in), and then sections were removed from the warm rolled plates and cold rolled to 2.5 mm (0.1 in). Aging was then conducted for 4 hours 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.

#### Mechanical Testing

Subsized flat tensile specimens with a 2.54 cm (1 in) gauge were machined in the longitudinal direction. Samples 1.6 mm (0.063 in) thick were taken at T/2 from the 2.5 mm (0.1 in) sheets and at T/4 from the plate (Al-4Mg-0.5Sc). Alloy 2219 was in 3.2 mm (0.125 in) sheet and tensiles were taken near full thickness.

Tension and Kahn tear tests were conducted in stroke control at a rate of  $5 \times 10^{-3}$  mm/s ( $2 \times 10^{-4}$  in/s) on a hydraulic testing machine. The stroke rate was chosen to emulate that used at Alcoa for the initial tear test period <sup>5</sup>; however, tests at Alcoa were conducted in load control while those at LBL were stroke controlled, thus this was an approximation. Tensile strains were measured using a clip gage, spring loaded to hook onto two pins which are tightened onto the specimen.

Due to limited material, only two tensile tests could be obtained from each alloy. 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 (Al-0.5Sc and Al-4Mg-0.5Sc) were tested at 77 K.

Tear samples were machined in the L-T orientation. All samples were 1.6 mm (0.063 in) thick, taken at T/2 from the sheet materials and T/4 from the thicker material, Al-4Mg-0.5Sc. Prior to testing, samples were polished to 600 grit perpendicular to the crack propagation direction. Data for analysis was truncated below 220 N (50 lbs).

As in tension tests, the materials were first tested at 77 K. While one sample is generally not sufficient for the tear test, lack of material prevented duplication; furthermore, it was felt that the general trend of the Al-Mg-Sc materials would indicate whether further

investigation would be desirable. As with tensile tests, tear tests at 4 K 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.

### Microscopy

Optical microscopy was used to show qualitatively the grain and intermetallic size and distributions. Fracture surfaces were observed via scanning electron microscopy (SEM) and micrographs were taken at about 50, 200, and 800 times magnification. Photographs were then compared qualitatively for variations in temperature and composition, and correlations to microstructure and properties. A summary of this work is included herein and details have been recorded elsewhere <sup>6</sup>.

## RESULTS AND DISCUSSION

### Mechanical Properties

Al-Mg-Sc and 2219-T87 tensile results are given in Table 2, with results averaged for those tests that were duplicated. Results show the expected strength increase with decreasing temperature. In addition, general Al-Mg-Sc elongation and reduction in area generally either decrease or remain constant from 77 to 4 K, with good values at both temperatures, e.g. all elongations of 10 % or greater.

The relatively lower strength of the binary Al-Sc alloy is expected because it lacks mag-nesium solid solution strengthening. Theoretically, solid solution strengthening can be utilized with the addition of magnesium up to its solubility in aluminum at the elevated processing temperatures. At the aging temperature used, 288°C (550°F), embrittling intermetallic  $Al_xMg_y$  species form above about 5.5 weight percent magnesium <sup>7</sup>. These secondary phases probably explain in part why the strength does not increase when Mg is raised from 4 to 6 weight percent (Al-4Mg-0.5Sc-0.4Mn to Al-6Mg-0.5Sc). The lower strength of Al-4Mg-0.5Sc compared to 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 materials. In summary, and as predictable via strengthening theory <sup>6,8</sup>, the increase in magnesium up to solubility, cold rolling, and decrease in temperature all strengthen the Al-Sc system without causing ductility to become prohibitively low.

Table 2: Cryogenic Tensile Data

| Material  | Tensile specimen I.D. | Test Temp. K | Yield Strength MPa [Ksi] | Tensile Strength MPa [Ksi] | Total Elong. % * | Area Red. % |
|-----------|-----------------------|--------------|--------------------------|----------------------------|------------------|-------------|
| Al-0.5Sc- | 0Mg 57t1-LN           | 77           | 379 [55]                 | 455 [66]                   | 14               | 32          |
|           | 57t2-LN               | 77           | 434 [63]                 | 483 [70]                   | 10               | 32          |
|           | average               | 77           | 407 [59]                 | 469 [68]                   | 12               | 32          |
| 2Mg       | 52t1-LN               | 77           | 455 [66]                 | 565 [82]                   | 20               | 29          |
|           | 52t2-LH               | 4            | 524 [76]                 | 655 [95]                   | 10               | 28          |
| 4Mg       | 54t1-LN               | 77           | 427 [62]                 | 552 [80]                   | 14               | 21          |
|           | 54t2-LN               | 77           | 427 [62]                 | 538 [78]                   | 22               | 26          |
|           | average               | 77           | 427 [62]                 | 545 [79]                   | 18               | 23.5        |
| 4Mg-0.4Mn | 56t1-LN               | 77           | 496 [72]                 | 641 [93]                   | 15               | 19          |
|           | 56t2-LH               | 4            | 538 [78]                 | 752 [109]                  | 14               | 22          |
| 6Mg       | 59t1-LN               | 77           | 427 [62]                 | 572 [83]                   | 16               | 18          |
|           | 59t2-LH               | 4            | 558 [81]                 | 752 [109]                  | 10               | 17          |
| 2219-T87  |                       | 77           | 441 [64]                 | 586 [85]                   | 9                | 28          |
| 2219t4-LN |                       | 77           | 434 [63]                 | 565 [82]                   | 12               | 26          |
| average   |                       | 77           | 438 [63.5]               | 576 [83.5]                 | 10.5             | 27          |

\* 2.54 cm (1 in) gauge

Both 6061-T6 and 2219-T87 were tested for comparison and standardization of the Kahn tear test, which had not previously been used at LBL. Comparison of results with Alcoa data shows large differences in the unit initiation and propagation energies (UIE and UPE). Variations as a function of test location have been noticed previously. The factors which could contribute to these variations were examined and are discussed elsewhere<sup>6</sup>. As a result of this analysis, it was decided to focus on the tear-yield ratios as the toughness indicator.

Al-Mg-Sc and 2219-T87 tear data are shown in Table 3. With decreasing temperature, the Al-Mg-Sc tear-yield ratio generally decreased and the propagation energies increased, while most values were competitive with 2219-T87. 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 materials exhibit ratios well above 1.0 at both 77 and 4 K. The tear toughness of the Al-Mg-Sc system is generally exceptional, compared to other aerospace aluminum alloys<sup>4</sup>; therefore, although only one to two specimens were tested in each condition, the overall results suggest that the Al-Mg-Sc system has good cryogenic toughness and should be considered seriously for further study.

In evaluating the strength and toughness of materials, it is valuable to compare these properties together, as they are often coupled. The tear-yield ratio versus yield strength of the Al-Mg-Sc materials and 2219-T87 are plotted in Figure 1 for 300, 77, and 4 K. The plots contain lines connecting data from each test temperature, which was averaged where

duplicate tests were performed. These lines are for visual aid and do not indicate a linear relationship. Where there are only two points, data is from 300 and 77 K. Yield strength generally increases with decreasing temperature, thus, higher strength points correspond to lower temperature tests. The only exception to this is the Al-6Mg-0.5Sc alloy, which had a slight strength decrease from 300 to 77 K. The 300 K points are from the same lots of material, tested previously at Alcoa<sup>3</sup>. With decreasing temperature, some of the strength-toughness combinations increase, while others decrease, but again, all of the tear-yield ratios are well above 1.0 and strength-tear toughness relationships look promising compared to 2219-T87. From these data, the best material would probably contain 4-6 weight percent magnesium, since the strength and tear toughness values are good and density reduction would thus be significant.

Table 3: Cryogenic Kahn Tear Data

| Material  | Kahn Specimen I.D. | Test Temp. K | U.I.E. m-MPa | U.P.E. [in-lb/in*in] | Tear Strength MPa [Ksi] | Tear Yield Ratio |
|-----------|--------------------|--------------|--------------|----------------------|-------------------------|------------------|
| Al-0.5Sc- | 57k1-LN            | 77           | 113 [647]    | 161 [917]            | 669 [97]                | 1.64             |
|           | 57k2-LH            | 4            | 263 [1500]   | 204 [1163]           | 821 [119]               |                  |
| 2Mg       | 52k1-LN            | 77           | 140 [801]    | 128 [730]            | 731 [106]               | 1.61             |
|           | 52k2-LH            | 4            | 158 [905]    | 178 [1019]           | 821 [119]               |                  |
| 4Mg       | 54k1-LN            | 77           | 156 [892]    | 156 [892]            | 752 [109]               |                  |
|           | 54k2-LN            | 77           | 132 [753]    | 134 [767]            | 765 [111]               |                  |
|           | average            | 77           | 144 [823]    | 145 [830]            | 758 [110]               | 1.77             |
| 4Mg-0.4Mn | 56k1-LN            | 77           | 76 [434]     | 49 [282]             | 641 [93]                | 1.29             |
|           | 56k2-LH            | 4            | 89 [509]     | 25 [145]             | 662 [96]                |                  |
| 6Mg       | 59k1-LN            | 77           | 55 [312]     | 56 [318]             | 552 [80]                |                  |
|           | 59k2-LN            | 77           | 52 [298]     | 54 [309]             | 572 [83]                |                  |
|           | average            | 77           | 53 [305]     | 55 [314]             | 562 [81.5]              | 1.31             |
| 2219-T87  | 2219k1-LN          | 77           | 63 [359]     | 66 [376]             | 593 [86]                | 1.39             |
|           | 2219k2-LN          | 77           | 61 [349]     | 72 [411]             | 621 [90]                |                  |
|           | average            | 77           | 62 [354]     | 69 [393.5]           | 607 [88]                |                  |

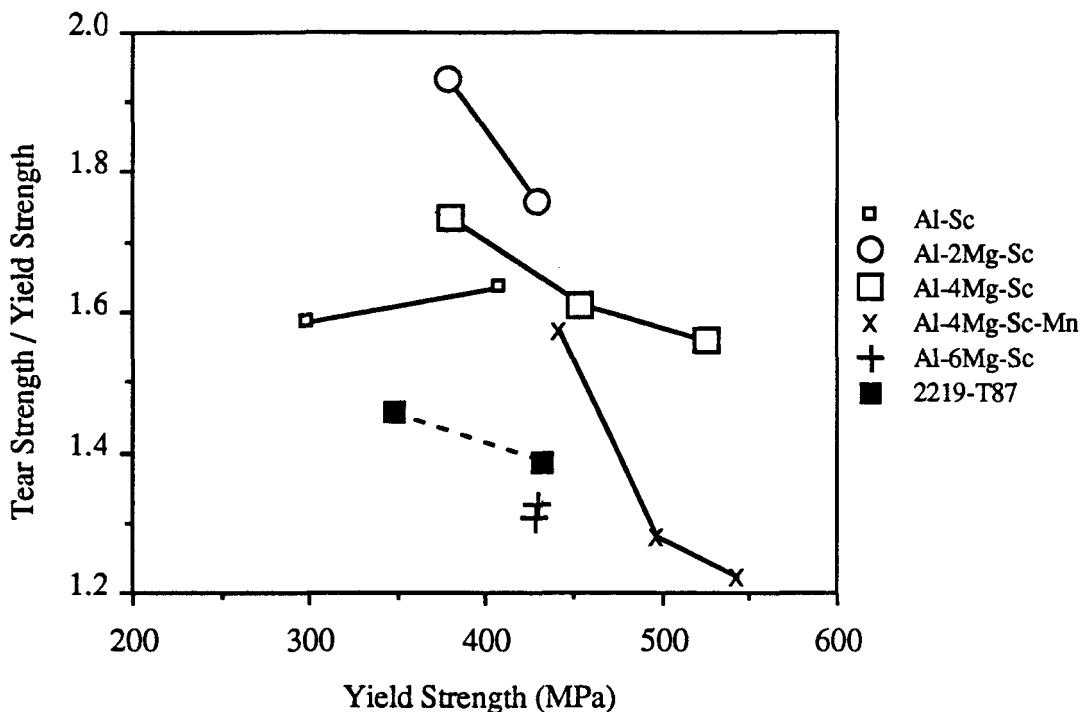


Figure 1: Yield strength versus tear-yield ratio

### Microscopy

Scanning electron microscopy (SEM) of the Al-Mg-Sc tear fracture surfaces are shown in Figure 2 to document the fracture morphologies. Fractographs taken at relatively lower and higher magnifications from each test condition are presented. Included here is a summary of the observations, as presented in detail elsewhere<sup>6</sup>. The binary alloy, not strengthened by magnesium, is the softest and exhibits ductile dimples at 77 and 4 K. The 2-4 weight percent magnesium alloys show increasingly tortuous fracture appearance with decreasing temperature and increasing alloying additions. Micrographs from the 77 K Al-4Mg-0.5Sc test are not included, as they were similar to the 77 K surfaces of Al-4Mg-0.5Sc-0.4Mn. Finally, the 6 percent magnesium alloy exhibits large dimples, which appear to nucleate at particles of a size seen in optical microscopy only in this alloy. These inclusions degrade the strength-tear toughness of Al-Mg-Sc alloys and, according to the phase diagram<sup>7</sup>, were probably the  $Al_xMg_y$  type that cannot be eliminated via solution heat treatment.

In summary, the increase in alloying additions generally decreased the tear toughness of the Al-Mg-Sc alloys, accompanied by fracture mode changes, while a decrease in temperature generally decreases the tear-yield ratio toughness, with a more tortuous fracture surface.

## CONCLUSIONS

From this study, one cannot be certain whether the materials tested will be useful for cryogenic applications, mainly because design criteria have not yet been disclosed. Even if the criteria were known, tension and fracture toughness 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.

For 2 to 6 weight percent magnesium, test results show that the strength-toughness (tear-yield ratio) combination of these alloys decreases with increasing alloying additions and decreasing test temperature. Furthermore, the Al-Mg-Sc system compared favorably with 2219-T87 in strength and tear toughness properties. These materials might thus be useful if strength and/or toughness were emphasized in a design for cryogenic application where density reduction is desired. Finally, the 4 and 6 weight percent magnesium alloys appear most promising because they provide more density reduction.

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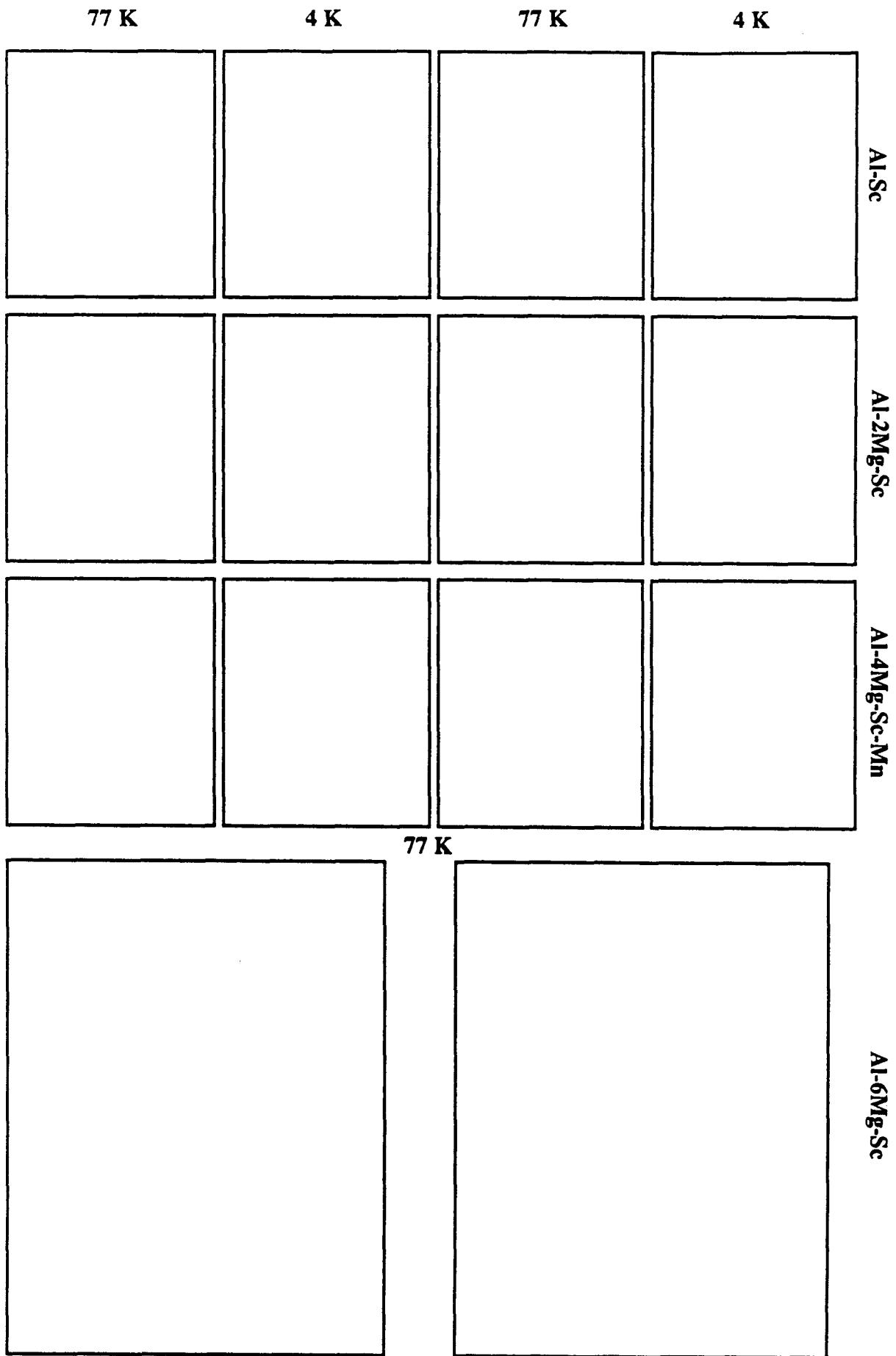


Figure 2: Scanning electron microscopy of Al-Mg-Sc alloys at cryogenic temperatures.