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# Strengthening Low-Alloy Magnesium Sheet by Strain Softening and Annealing

By M. M. Tilman, R. L. Crosby, and L. A. Neumeier



UNITED STATES DEPARTMENT OF THE INTERIOR



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**UNITED STATES DEPARTMENT OF THE INTERIOR**  
Donald Paul Hodel, Secretary

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

h	hour	lb	pound
in	inch	min	minute
in/in	inch per inch	pct	percent
in/min	inch per minute	wt pct	weight percent
ksi	kips (thousand pounds) per square inch		

# STRENGTHENING LOW-ALLOY MAGNESIUM SHEET BY STRAIN SOFTENING AND ANNEALING

By M. M. Tilman,<sup>1</sup> R. L. Crosby,<sup>2</sup> and L. A. Neumeier<sup>3</sup>

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## ABSTRACT

The Bureau of Mines conducted research to determine the feasibility of strengthening experimental magnesium-base sheet alloys by strain softening induced by controlled cold rolling followed by selective heat treatment. Relatively small alloying additions having potential for producing strain softening were selected from metals that are readily available. Tensile properties comparable to minimum properties of the most commonly used magnesium-base sheet alloy AZ31B, in the H24 condition (strain-hardened and partially annealed), were obtained in an Mg-1MM (mischmetal)-0.5Mn alloy after strain-softening rolling followed by heat treatment. Tensile properties of the experimental alloys in certain conditions are also compared with minimum tensile properties of other standard magnesium sheet alloys.

Incidental to the principal objectives, apparent superplastic behavior was observed in three experimental magnesium-base alloys containing MM, MM plus manganese, and MM plus zirconium.

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<sup>1</sup>Metallurgist.

<sup>2</sup>Metallurgist (deceased).

<sup>3</sup>Research supervisor.

Rolla Research Center, Bureau of Mines, Rolla, Mo.

## INTRODUCTION

As part of the Bureau of Mines efforts to assure an adequate mineral base for the Nation's economy, research was undertaken to investigate the feasibility of devising magnesium-base sheet alloys having tensile properties comparable to those of the most commonly used AZ(Al,Zn)-type alloys, but with reduced requirements for imported alloying elements. Two ways by which metals can be strengthened are by relatively large alloying additions or by smaller additions in conjunction with thermal-mechanical processing. The latter approach specifically strain softening plus heat treating, was selected for this research.

The hexagonal crystal structure of common magnesium-base sheet alloy, AZ31B, upon progressive cold rolling, begins to "break up" or crack by excessive strain hardening at about 30 to 50 pct accumulative reduction. Menzen (7)<sup>4</sup> demonstrated that an Mg-1.5Mn alloy (AM 503) could be cold-rolled to as much as 90-pct reduction without intermediate annealing and noted an accompanying reduction in strength and increase in ductility. Ansel (3) and Hurst (5) reported a similar strain- or work-softening effect in cold-worked Mg-1.5Mn-0.1Ca; Ansel (3) reported a subsequent hardening effect after a low-temperature heat treatment but noted the absence of these effects in Mg-3Al-1Zn (AZ31). In subsequent work, McDonald (6) investigated the cold working and heat treating of Mg-0.3RE (rare

earth), Mg-0.6Zr, and Mg-1.5Mn-0.2RE alloys. McDonald observed a strengthening effect of a heat treatment on these alloys following work (strain) softening. The work softening was produced by cold rolling at 1.5-pct reduction per pass up to 85-pct total cold reduction. Couling (4) described the deformation mechanism in Mg-0.5Th and Mg-0.2MM (mischmetal)-0.4Zr alloys which permits "unlimited" rollability; the probable aging mechanism was discussed.

This research can be considered an extension of the previous referenced work and as an attempt to obtain optimum combinations of processing variables and promising alloying elements.

Efforts are described to develop practical magnesium-base sheet alloys exhibiting useful properties, by taking advantage of combined strain softening and annealing effects. The selection of alloying elements was limited to those for which there are domestic resources available, or in the case of manganese, potentially available from sea nodules. Initially, 16 magnesium-base compositions were cast with a 1.5-pct maximum alloy content of MM, Mn, Zr, and Ca in binary and ternary alloys. After preliminary testing, four of the most promising compositions were selected for further evaluation. Alloying elements in the four compositions were MM (56 to 58 pct Ce), Mn, and Zr.

## EXPERIMENTAL PROCEDURE

Based on the work of previous investigators (3-7), 16 binary and ternary compositions were selected containing individual or various combinations of Mn, MM, Zr, and Ca in total amounts not exceeding 1.5 wt pct. The 3.5-lb charges were melted in magnesia crucibles, under a commercially available flux cover, in an open-pot electrical-resistance furnace.

<sup>4</sup>Underlined numbers in parentheses refer to items in the list of references at the end of this report.

Manganese was added to the melts with a commercial flux containing 31 pct Mn. The mischmetal contained 56 to 58 pct Ce, balance La and other rare earths. Zirconium was added as a master alloy containing approximately 30 pct Zr and 70 pct Mg; calcium was added as 98-pct-pure Ca, balance CaCl<sub>2</sub>. The melts were cast into a preheated (400° C), bottom-feed carbon slab mold having cavity dimensions of 7.5 by 4.75 by 1.375 in. The slabs were annealed at 400° C for 90 min, and approximately 0.125 in was scalped from each

rolling surface. The scalped slabs were then rolled at 400° C to 0.125-in thickness at nominal 10-pct reduction per pass and sectioned for continued processing.

Sections of the hot-rolled plates were cold-rolled<sup>5</sup> at approximately 2 pct per pass to 0.025-in thickness for a total cold reduction of 80 pct. For comparison with the cold-rolled sheet, samples of the hot-rolled plates were further hot-rolled (at 400° C), also to 0.025-in thickness. Samples of both hot- and cold-rolled sheet were annealed<sup>6</sup> at 180° or 200° C for 24 h.

Standard ASTM E8, 8-in-long sheet tensile specimens (2) having a 2-in gage length were cut parallel to the rolling direction from each alloy sheet in the hot-rolled, cold-rolled, and the rolled-and-annealed conditions. Tensile testing was conducted on an Instron<sup>7</sup> machine at crosshead velocities of 0.02 in/min to the yield load (at 0.2-pct offset) and then at 0.2-in/min to fracture. Based on results of tensile testing, 4 of the original 16 compositions were selected for continued investigation. These four compositions are shown in table 1. Specimens of the four alloys, cold-rolled at 2 pct per pass, were annealed at 140° or 160° C for 24 h and tensile-tested. These results, together with those previously obtained, provided tensile data for

<sup>5</sup>All rolling at room temperature is referred to as "cold rolling"; however, it should be realized that strain softening occurring after initial strain hardening is behavior distinct from that typical of normal cold rolling, where strain hardening continues to accrue as the rolling progresses.

<sup>6</sup>The term "annealed" is used in preference to "aging" but does not indicate full annealing. The mechanism is somewhat obscure; microstructures are in a "strain-softened" rather than "strain-hardened" condition.

<sup>7</sup>Reference to specific equipment does not imply endorsement by the Bureau of Mines.

TABLE 1. - Alloy compositions, weight percent

Alloy	Nominal				Chemical analysis		
	MM <sup>1</sup>	Mn	Zr	Mg	MM <sup>1</sup>	Mn	Zr
A.....	1.0	0.5	0	Bal	0.90	0.38	ND
B.....	.5	.5	0	Bal	.44	.44	ND
C.....	.5	0	.5	Bal	.31	ND	0.56
D.....	1.5	0	0	Bal	1.23	ND	ND

ND Not determined.

<sup>1</sup>Mischmetal (56 to 58 pct Ce, balance La and other rare earths).

the four cold-rolled alloys annealed at 140°, 160°, 180°, or 200° C.

In an effort to determine the extent of cold rolling required to produce strain softening and possibly shorten the procedure, samples of the four hot-rolled alloys were cold-rolled at a nominal 2 pct per pass to total reductions up to 80 pct, and sectioned at accumulated increments of 10-pct reduction. As diamond pyramid hardness (DPH) measurements did not give a clear indication of the onset of the strain softening, the sections were heated at 180° C for 24 h to accentuate hardness differences and tested for DPH at 50-g load. Based on peak hardness measurements, samples of the hot-rolled alloys were cold-rolled to 30-, 40-, or 50-pct reduction at nominal 2-pct, reduction per pass and were then finish-cold-rolled at up to nominal 10-pct reduction per pass<sup>8</sup> to 80-pct total cold reduction (0.025 in thick). The cold-rolled samples were heat-treated at 180° or 200° C for 24 h and tensile-tested.

Metallographic samples were taken at appropriate steps during the processing. Following trials with a number of dip and electrolytic etches, the specimens were electrolytically etched in a one-to-three solution of phosphoric acid plus ethyl alcohol.

<sup>8</sup>The phrase "up to nominal 10-pct reduction per pass" indicates a gradual increase in percent reduction to avoid cracking, from nominal 5- to 7-pct and finally to 10-pct reduction per pass.



Upon observation of high room-temperature ductility in 80-pct cold-rolled sheet, a tensile test for superplastic behavior was conducted on one specimen each of alloys B, C, and D.

(Insufficient material precluded a test on alloy A.) The tests were conducted at 350° C and 0.02 in/in per minute initial strain rate.

#### RESULTS AND DISCUSSION

Results of tensile tests conducted on four alloys in various hot-rolled, cold-rolled, and rolled-and-annealed conditions are listed in table 2 and plotted in figures 1 and 2.<sup>9</sup> Each value represents an average of three tests. Figure 1 illustrates the tensile strength,

yield strength, and elongation of alloys A, B, C, and D in the hot-rolled condition, and the changes in the three values following annealing of the hot-rolled sheet at 180° and 200° C. Conducted mainly for the sake of completeness, the tests indicate very little increase in

<sup>9</sup>Care must be used in interpreting figures 1, 2, and 4. Diagonal lines representing yield strength slant to the

right, while those representing elongation slant to the left. Superimposing the two results in crosshatching.

TABLE 2. - Comparative tensile properties of alloy sheet in the as-hot-rolled, as-cold-rolled (strain-softened), and rolled-plus-annealed conditions

Condition	Strength, ksi		Elongation, pct	Condition	Strength, ksi		Elongation, pct
	Ten-sile	Yield			Ten-sile	Yield	
HOT ROLLED <sup>1</sup>				COLD ROLLED 80 PCT <sup>2</sup> --Continued			
As rolled:				Annealed 140° C:			
Alloy A.....	27.2	21.2	13	Alloy A.....	ND	ND	ND
Alloy B.....	27.6	20.3	13	Alloy B.....	32.8	26.9	6
Alloy C.....	27.8	21.4	16	Alloy C.....	34.4	29.3	2
Alloy D.....	27.4	20.1	12	Alloy D.....	34.8	29.0	3
Annealed 180° C:				Annealed 160° C:			
Alloy A.....	29.3	24.2	9	Alloy A.....	37.7	29.6	4
Alloy B.....	29.1	23.1	10	Alloy B.....	35.4	26.6	6
Alloy C.....	29.9	24.0	8	Alloy C.....	33.7	29.6	3
Alloy D.....	29.4	23.3	8	Alloy D.....	37.2	28.9	5
Annealed 200° C:				Annealed 180° C:			
Alloy A.....	29.4	24.0	10	Alloy A.....	40.3	31.5	3
Alloy B.....	28.9	22.6	11	Alloy B.....	37.5	28.0	5
Alloy C.....	30.2	24.9	8	Alloy C.....	38.1	30.3	3
Alloy D.....	29.9	23.2	6	Alloy D.....	39.1	30.4	4
COLD ROLLED 80 PCT <sup>2</sup>				Annealed 200° C:			
As rolled:				Alloy A.....	39.8	31.0	4
Alloy A.....	27.5	16.0	24	Alloy B.....	37.4	27.9	6
Alloy B.....	27.1	15.2	24	Alloy C.....	37.5	29.7	2
Alloy C.....	27.0	16.1	16	Alloy D.....	39.5	30.3	3
Alloy D.....	26.2	14.9	17				

ND Not determined.

<sup>1</sup>Hot-rolled from 1.125 to 0.025 in thick at 400° C at 10-pct reduction per pass.

<sup>2</sup>Cold-rolled from 0.125 to 0.025 in thick (80-pct total cold reduction) at ~2-pct reduction per pass (initially hot-rolled from 1.125 to 0.125 in at 400° C).

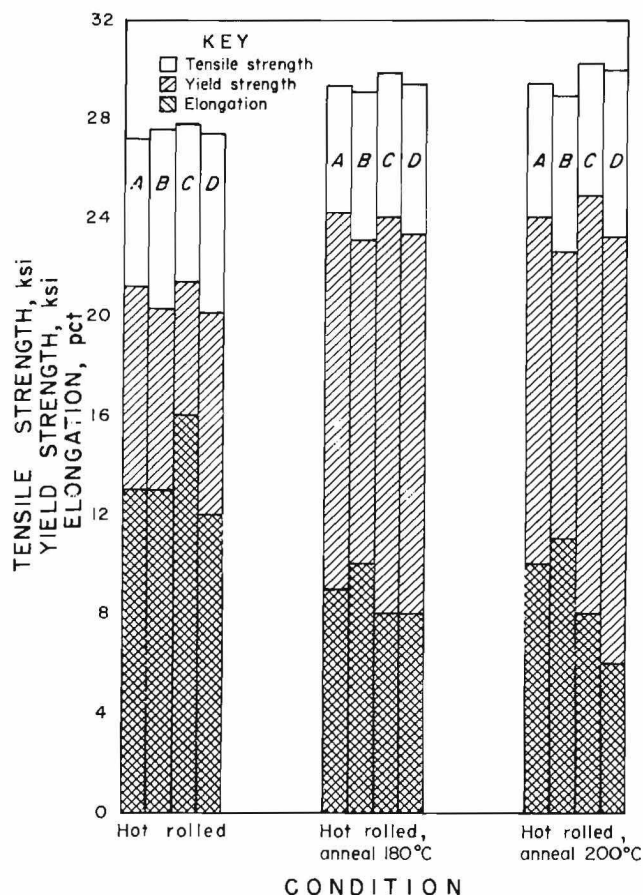


FIGURE 1. - Tensile properties of experimental magnesium alloy sheet in the as-hot-rolled and the hot-rolled-plus-annealed conditions. Nominal alloy compositions follow: A, Mg-1.0MM-0.5Mn; B, Mg-0.5MM-0.5Mn; C, Mg-0.5MM-0.5Zr; D, Mg-1.5MM.

strength and reduction of ductility after annealing the hot-rolled sheet at either 180° or 200° C.

The results in figure 2, however, show considerable change in tensile properties following cold rolling plus annealing. After 80-pct accumulative cold-rolling reduction at nominal 2-pct reduction per pass, the four alloys exhibit a 24- to 26-pct reduction in yield strength with an insignificant change in tensile strength. (In figure 2, it should be noted that the elongation bar values exceed the yield strength bar values for the cold-rolled sheet.) Percent elongation is increased following the cold reduction, except for alloy C, which remained constant.

Annealing the cold-rolled sheet for 24 h resulted in substantial increases in tensile and yield strength of all four alloys, compared to the hot- or cold-rolled values. A large increase is evident at temperatures as low as 140° C, but maximum yield strengths are obtained at 180° C, with slight apparent decrease at 200° C. The apparent maximum tensile strength occurred for the annealing at 180° C for alloys A, B, and C, and at 200° C for alloy D. The percent elongations of the 80-pct cold-rolled alloys are correspondingly reduced by the annealing treatments, to values between 2 and 6 pct, with the specific values apparently more sensitive to composition than to temperature. Time was not available to optimize the annealing results as to heating time, such as minimum period to attain the given results at 180° or 200° C, or perhaps raising the temperature somewhat to reduce the heating periods.

Cold-rolling and annealing experiments were conducted on each alloy to determine if the rolling schedule could be shortened without loss of properties. Samples of each alloy were cold-rolled at nominal 2-pct reduction per pass and tested for hardness at accumulated increments of 10-pct cold reduction to determine at what reduction strain softening occurred. Hardness values of the as-cold-rolled samples showed insignificant differences; however, subsequent annealing at 180° C for 24 h prior to hardness testing resulted in precipitation or other strengthening to the extent of indicating significant hardness differences. The hardness values are listed in table 3 and plotted in figure 3. No change in hardness was taken as indication of essential completion of strain softening (that is, little if any lattice strain to respond to heating). A decrease in hardness with increasing cold reduction was taken as an indication of incomplete extent of strain softening.

Based on the hardness values, hot-rolled sheet samples were cold-rolled at 2 pct per pass to 50-pct accumulative reduction (alloy A), 30-pct reduction

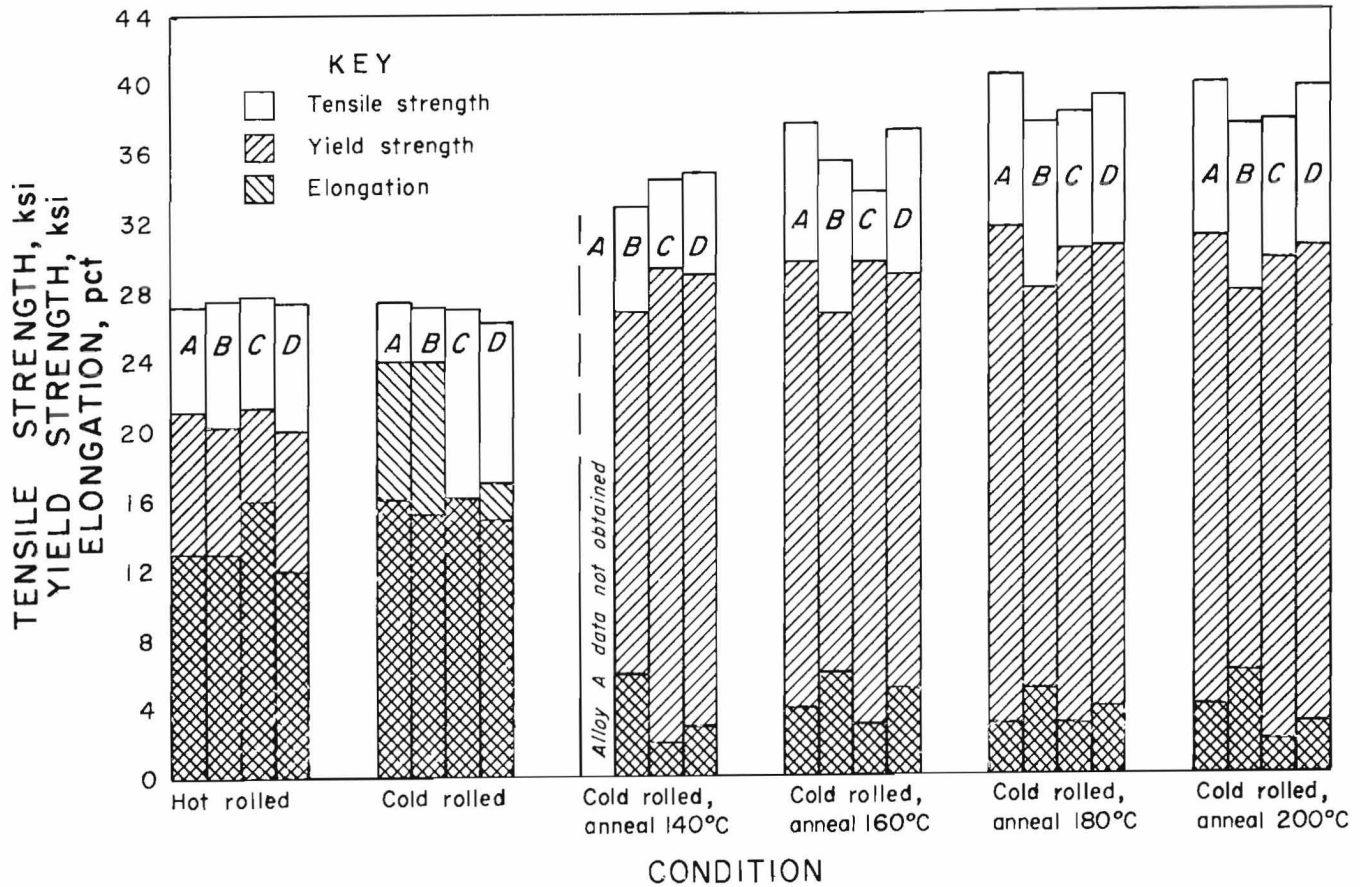


FIGURE 2. - Tensile properties of experimental magnesium alloy sheet in the as-hot-rolled, the as-cold-rolled, and the cold-rolled-plus-annealed conditions. Cold rolling was at nominal 2-pct reduction per pass to 80-pct total reduction. Nominal alloy compositions follow: A, Mg-1.0MM-0.5Mn; B, Mg-0.5MM-0.5Mn; C, Mg-0.5MM-0.5 Zr; D, Mg-1.5MM.

TABLE 3. - Diamond pyramid hardness (50-g load) as a function of accumulative cold-rolling reduction<sup>1</sup>

Reduction, <sup>2</sup> pct	Diamond pyramid hardness			
	Alloy A	Alloy B	Alloy C	Alloy D
10	43.1	39.6	40.2	39.7
20	42.2	44.6	44.6	42.2
30	42.2	48.1	48.1	44.4
40	46.6	45.2	47.7	47.3
50	49.9	45.6	46.0	46.0
60	48.1	46.6	47.7	45.2
70	51.6	46.6	47.7	46.2
80	54.9	43.3	49.9	47.6

<sup>1</sup>Specimens were annealed 24 h at 180° C following the indicated reduction. Each value is an average of 5 measurements.

<sup>2</sup>At nominally 2-pct reduction per pass.

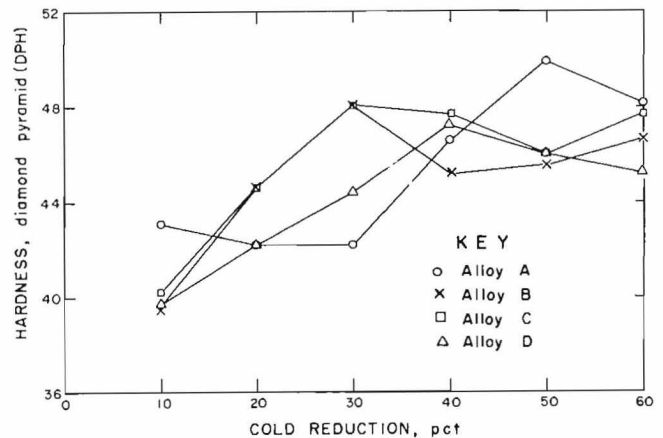


FIGURE 3. - Hardness versus percentage accumulative cold reduction for experimental magnesium alloy sheet. Alloys were rolled at nominal 2-pct reduction per pass and annealed 24 h at 180° C prior to hardness testing.

(alloys B and C), and 40-pct reduction (alloy D). The sheet samples were then finish-rolled cold to 80-pct total reduction at up to 10-pct reduction per pass and annealed for comparison with the sheet processed entirely at 2-pct reduction per pass and annealed. Annealing temperatures of 180° C (alloys C and D) and 200° C (alloys A and B) were selected to obtain the best combination of strength and ductility for each alloy. Average results of tensile testing of the four alloys annealed after cold rolling to 80-pct reduction at 2 pct per pass, or cold rolling at up to 10 pct per pass for the later passes, are shown in table 4 and in figure 4. Three specimens were tested for each alloy condition. There is little difference in tensile properties resulting from the two procedures. (Compare conditions II and IV.)

Strengths of all four annealed alloys following either rolling-plus-annealing procedure (conditions II and IV) were within the general range of strengths of AZ31B sheet and plate in the H24 condition (strain-hardened and partially

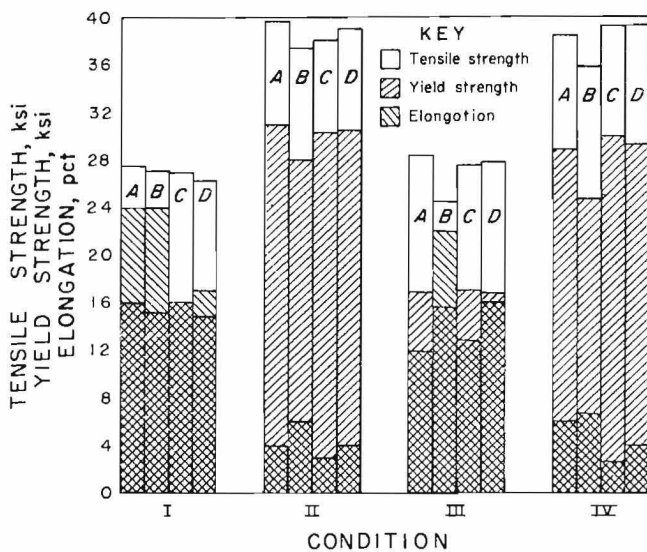


FIGURE 4. - Tensile properties of experimental magnesium alloy sheet resulting from various processing conditions. Conditions are explained in table 4. Nominal alloy compositions follow: A, Mg-1.0MM-0.5Mn; B, Mg-0.5MM-0.5Mn; C, Mg-0.5MM-0.5Zr; D, Mg-1.5MM.

TABLE 4. - Comparative tensile properties of sheet strain-softened with light cold reduction passes (condition I), sheet strain-softened with light plus heavy cold reduction passes (condition III), and annealed sheet of each (conditions II and IV, respectively)

Condition <sup>1</sup>	Strength, ksi		Elongation, pct
	Tensile	Yield	
Alloy A:			
I.....	27.5	16.0	24
II.....	39.8	31.0	4
III.....	28.3	16.9	12
IV.....	38.4	28.8	6
Alloy B:			
I.....	27.1	15.2	24
II.....	37.4	27.9	6
III.....	24.5	15.8	22
IV.....	35.8	24.7	7
Alloy C:			
I.....	27.0	16.1	16
II.....	38.1	30.3	3
III.....	27.4	17.0	14
IV.....	39.3	29.9	3
Alloy D:			
I.....	26.2	14.9	17
II.....	39.1	30.4	4
III.....	27.8	16.8	16
IV.....	39.3	29.2	4

<sup>1</sup>Conditions are as follows:

I--Cold-rolled at 2 pct per pass to 80-pct total reduction.

II--Cold-rolled at 2 pct per pass to 80-pct total reduction; annealed 24 h at 200° C (alloys A and B) and 180° C (alloys C and D).

III--Cold-rolled at 2 pct per pass to 50-pct reduction (alloy A), 30-pct reduction (alloys B and C), and 40-pct reduction (alloy D). All alloys finish-cold-rolled at 10 pct per pass to 80-pct total reduction.

IV--Cold-rolled at 2 pct per pass to 50-pct reduction (alloy A), 30-pct reduction (alloys B and C), and 40-pct reduction (alloy D). All alloys finish-cold-rolled at 10 pct per pass to 80-pct total reduction and annealed 24 h at 200° C (alloys A and B) and 180° C (alloys C and D).

annealed) (1).<sup>10</sup> The AZ31B wrought alloy was of particular interest because this alloy is by far the most widely used. Annealed alloys A and B had average percentage elongations within the general range for AZ31B-H24, following the shortened rolling procedure (condition IV). Percentage elongations of alloy B also fell within this range after annealing following the longer procedure (condition II), but alloys C and D with only 3- and 4-pct elongation, respectively, after annealing, following either procedure, were outside the range.

Tensile properties determined for the experimental compositions, which were rolled to 0.025-in thickness, were near but not equal to minimum tensile properties listed by ASTM for AZ31B sheet for the specific sheet thickness range of 0.016- to 0.249-in (1). ASTM specifications for this thickness range in the H24 condition are 39-ksi minimum tensile strength, 29-ksi minimum yield strength, and 6-pct minimum elongation.

Effects of reduced annealing time on tensile properties following the shortened rolling procedure were investigated for alloys A and C. Results for 1- and 24-h annealing are listed in table 5. These data represent a limited number of specimens; however, optimum properties evidently occur after an annealing time of only 1 h for both alloys A and C, with properties decreasing when annealing for 24 h.

Alloys A and C met the minimum required tensile properties for AZ31B sheet in the strengthened H24 condition--39-ksi tensile strength, 29-ksi yield strength, and 6-pct elongation. This was after initial cold rolling at 2-pct reduction per pass, finish cold rolling to 80-pct total cold reduction at up to 10-pct reduction per pass, and annealing at 200° C (alloy A) or 180° C (alloy C) for 1 h.

<sup>10</sup>ASTM specifications for AZ31B sheet and plate in the H24 condition are tensile strength 34 to 39 ksi, yield strength 18 to 29 ksi, and elongation 6 to 8 pct in 2 in for thicknesses from 0.016 to 3.000 in.

TABLE 5. -- Change in tensile properties with annealing time

Annealing time, h	Strength, ksi		Elongation, pct
	Tensile	Yield	
Alloy A:			
1.....	139.0	129.5	18
24.....	38.4	28.8	6
Alloy C:			
1.....	40.0	33.0	7
24.....	39.3	29.9	3

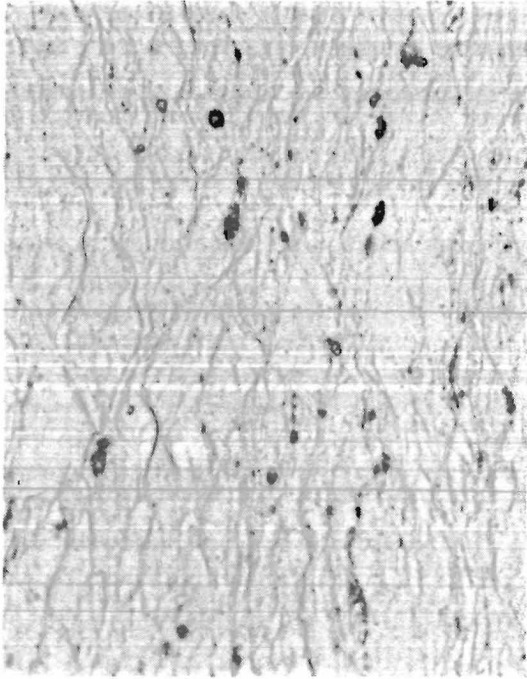
<sup>1</sup>Single test only.

NOTE.--Sheet cold-rolled at 2 pct per pass to 50-pct (alloy A) or 30-pct (alloy C) reduction; finish-rolled to 80-pct total reduction at up to 10 pct per pass; and annealed at 200° C (alloy A) or 180° C (alloy C). Averages of 3 specimens unless otherwise indicated.

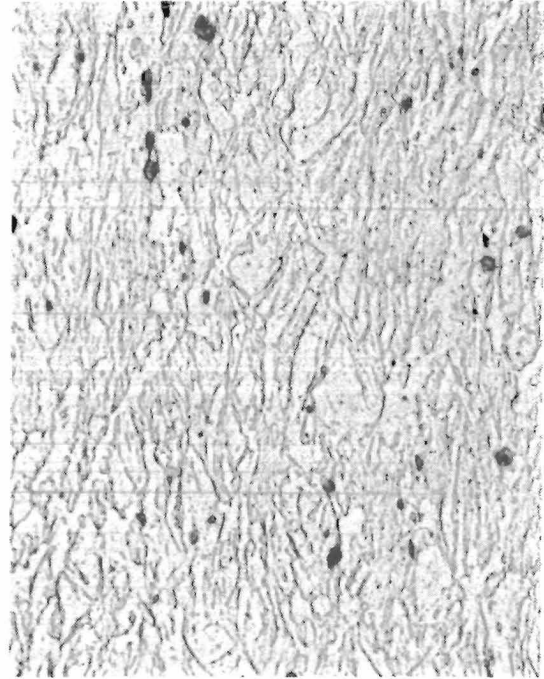
It should also be possible to bring alloys B and D to the minimum requirements or greater for AZ31B in the H24 condition. Further work to optimize annealing conditions could probably improve properties of all four alloys.

Figures 5 and 6 illustrate microstructures of alloys A through D in two different conditions of rolling plus annealing, that is, conditions I and IV of table 4 and figure 4. The banded structures are similar in appearance to those observed by Couling for Mg-Th and Mg-MM-Zr alloys (4). Condition IV processing has produced finer structures than those resulting from condition I processing in alloys A and C and possibly in alloys B and D. The microstructures are difficult to reveal, but repeated polishing and etching produced the structures shown. Condition I micrographs represent cold-rolled, strain-softened structures, whereas condition IV micrographs are representative of cold-rolled, strain-softened structures that have been annealed. The annealed condition IV structures have much higher tensile and yield strengths and reduced ductilities than the strain-softened structures of condition I.

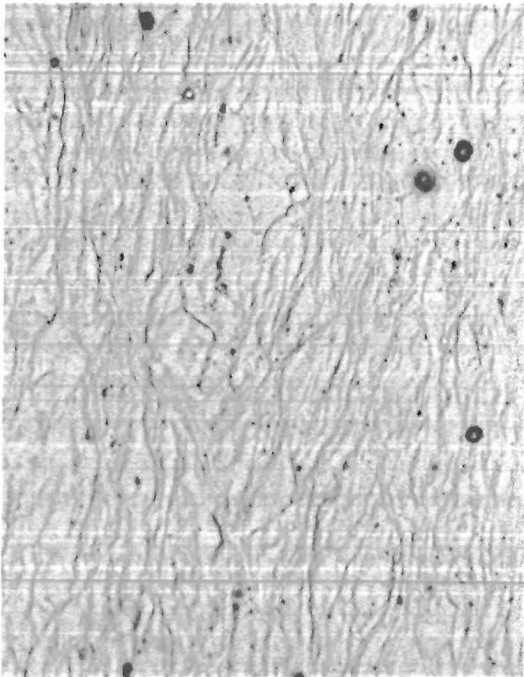
Viewing the experimental data in comparison with the minimum tensile requirements of conventional magnesium sheet



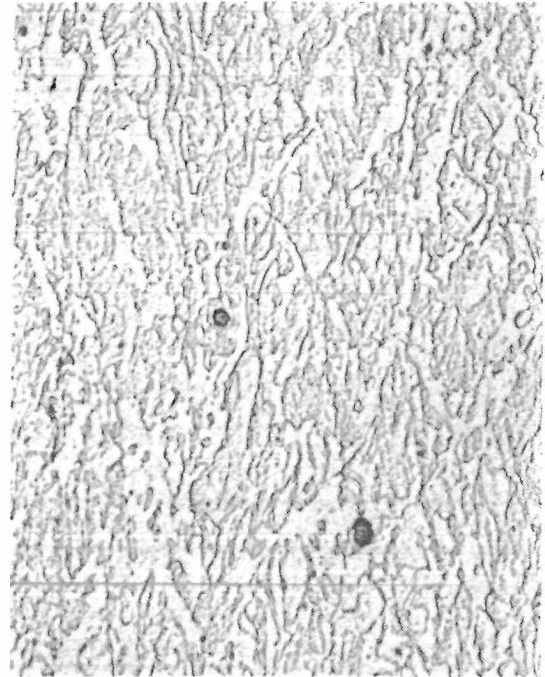
**Alloy A, Condition I**



**Alloy A, Condition IV**

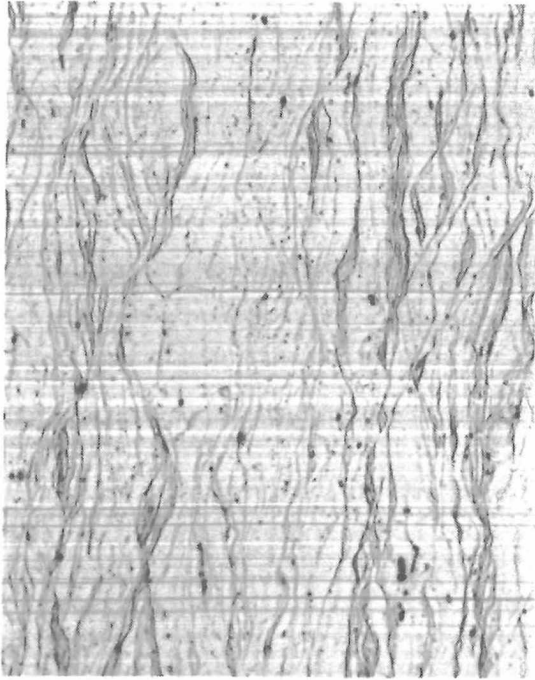


**Alloy B, Condition I**

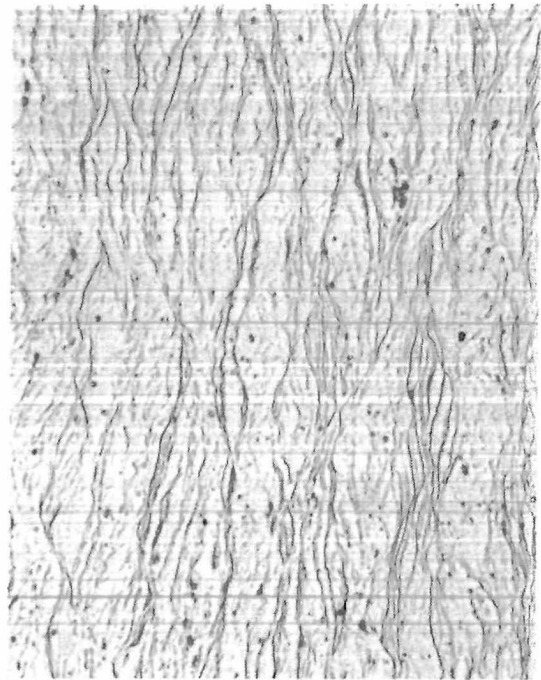


**Alloy B, Condition IV**

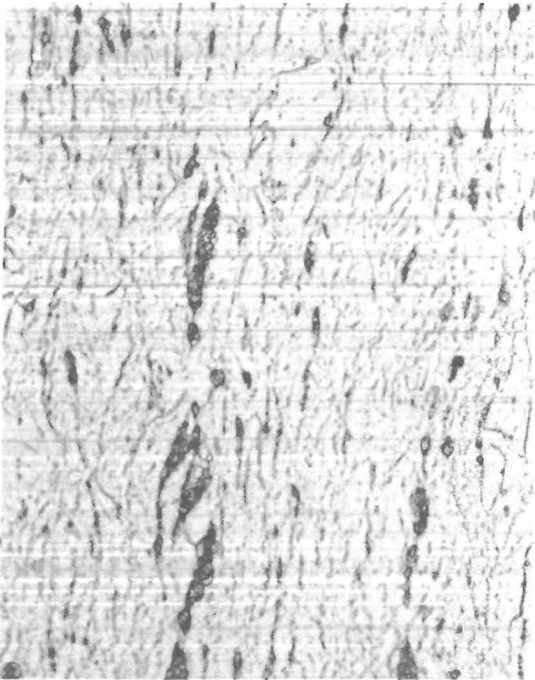
FIGURE 5. - Microstructures of alloys A and B in conditions I and IV (X500). Conditions are explained in table 4.



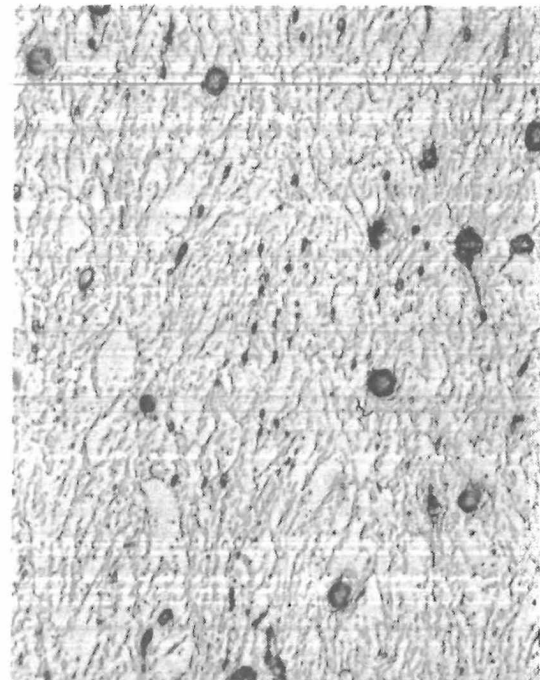
**Alloy C, Condition I**



**Alloy C, Condition IV**



**Alloy D, Condition I**



**Alloy D, Condition IV**

FIGURE 6. - Microstructures of alloys C and D in conditions I and IV (X500). Conditions are explained in table 4.

(generally <0.25 in thick), the alloys listed in table 6 yield some interesting interpretations. For purposes of discussion and strictly on the basis of overall room-temperature tensile properties, all four hot-rolled experimental alloys (table 2) have strengths and elongations that exceed those for the T7 strengthened condition (see footnote 1, table 6) of the LA141A alloy--19-ksi tensile strength, 15-ksi yield strength, and 10-pct elongation. The hot-rolled B alloy annealed at 180° C and the A and B alloys annealed at 200° C also equal or exceed the properties, as do all four alloys as cold rolled to 80-pct reduction (table 2), except that the yield strength of alloy D is 14.9 ksi instead of 15 ksi. As for an extremely ductile condition along with the >15-ksi yield strength, the as-cold-rolled alloys A and B exhibit 24-pct ductility, which is significantly higher than the most ductile annealed condition for the AZ31B, HK31A, and ZE10A alloys (condition 0 of table 6). It is expected that controlled annealing of the cold-rolled (80-pct) alloys A and B could produce a combination of 18-ksi yield strength plus 15-pct elongation as listed in table 6 for the annealed ZE10A alloy.

For the strengthened H24 condition for the ZE10A alloy in table 6, the minimum tensile properties required are 36-ksi tensile strength, 25-ksi yield strength,

and 4-pct elongation. A number of the experimental alloys (table 2), annealed after cold rolling at 2 pct per pass to 80-pct reduction, meet or exceed these criteria: the A and D alloys annealed at 160° C, the B and D alloys annealed at 180° C, and the A and B alloys annealed at 200° C. The properties are also exceeded for alloys A and D for the alternate cold-rolling schedule followed by annealing at 200° and 180° C, respectively (condition IV, table 4).

The experimental alloys A, B, and D in at least one of the cold-rolled-plus-annealed conditions (such as table 4, conditions II and IV) exceeded the required minimum properties for the HK31A alloy in the H24 condition--34-ksi tensile strength, 26-ksi yield strength, and 4-pct elongation. Alloys A and B exceeded the minimum required properties for the HM21A alloy for the T8 (footnote 1, table 6) strengthened condition, when in one or more of the annealed conditions after the 80-pct cold rolling (condition II or IV in table 4). The HK and HM alloys are the recommended magnesium alloys for elevated temperature service. The scope of the present investigation did not permit a full evaluation of the elevated-temperature stability of the experimental alloys with regard to stressing, nor with reference to room- or

TABLE 6. -- Comparative minimum tensile requirements for sheet of selected magnesium-base alloys according to ASTM standard specifications (1)

Alloy	Temper <sup>1</sup>	Main alloy additions, pct	Thickness, in	Strength, ksi		Elongation, pct
				Tensile	Yield <sup>2</sup>	
AZ31B.....	0	3Al-1Zn.....	0.016-0.500	32.0	ND	12
AZ31B.....	H24	3Al-1Zn.....	.016-.249	39.0	29.0	6
HK31A.....	0	3.2Th-0.7Zr.....	.016-.250	30.0	ND	12
HK31A.....	H24	3.2Tr-0.7Zr.....	.016-.125	34.0	26.0	4
HM21A.....	T8	2.0Th-0.8Mn.....	.016-.250	33.0	18.0	6
LA141A....	T7	14Li-1.2Al.....	.010-.090	19.0	15.0	10
ZE10A.....	0	1.25Zn-0.17RE <sup>3</sup> ...	.016-.060	30.0	18.0	15
ZE10A.....	H24	1.25Zn-0.17RE <sup>3</sup> ...	.016-.125	36.0	25.0	4

ND Not determined.

<sup>1</sup>0 = annealed; H24 = strain hardened and partially annealed; T7 = solution-treated and aged; and T8 = solution-treated, cold-worked, and aged.

<sup>2</sup>0.2-pct offset method.

<sup>3</sup>RE = rare earths.



elevated-temperature corrosion or oxidation behavior.

These comparisons illustrate that the selection of compositions conducive to response to the strain-softening phenomenon, and carrying out strain-softening and selective annealing under carefully controlled conditions, can induce a wide variety of tailored tensile properties--from weak-and-ductile to strong-and-tough. Other compositional variations and strain softening plus controlled annealing can be expected to produce even a wider variety or specifically desired combinations of properties.

It should be emphasized that the properties of the experimental alloys and the HK and HM commercial sheet alloys are compared for purposes of discussion of room-temperature properties. The HK and HM alloys are the recommended magnesium alloys for elevated-temperature service, and the experimental alloys would not be expected to match elevated-temperature strength of these alloys, particularly above about 200° C. Other than brief tests of superplastic behavior, no elevated-temperature measurements of tensile properties were made. Similarly, the LA alloys were originally developed primarily as an armament alloy resistant to rapid strain (cubic-structure-induced); no tests of rapid strain resistance were made for the experimental alloys.

The increased ductility of alloys A, B, and D at room temperature resulting from cold rolling suggested the possibility of superplastic behavior at elevated temperatures. Superplastic alloys characteristically exhibit deformation strain-rate sensitivity, fine grain sizes, and grains

resistant to recrystallization and/or grain growth during elevated-temperature deformation. Although not directly related to the strengthening of low-alloy magnesium sheet, a few tests were conducted to investigate potential superplasticity. Based on previous experience with superplastic magnesium alloys (8), tensile tests were conducted at 350° C and 0.02 in/in per minute initial strain rate on single specimens of alloys B, C, and D. (Alloy A sheet stock was depleted before these tests.) Elongations of over 100 pct are considered by a number of investigators as indicative of superplastic behavior. Using this criterion, alloys B, C, and D, with total elongations at fracture of 132, 246, and 309 pct, respectively, could be considered superplastic. Determination of optimum conditions of superplasticity could reveal greater ductility with increased potential for commercial superplastic forming. Although not verified in this investigation, it is anticipated that because of the structure refinement induced by the strain softening, the strain-softened and subsequently annealed sheet may exhibit less anisotropy than conventional rolled magnesium alloys; this should be examined in future studies.

A further point bears noting. Magnesium alloy sheet has traditionally been produced by energy-intensive hot rolling of thick slab castings to near final relatively thin gages. The method of strain softening plus annealing, which requires starting sheet thickness of not more than about twice the final gage to generate maximum properties, would fit any scheme of relatively thin-gage slab casting that may be devised.

## CONCLUSIONS

Experimental magnesium alloys containing not more than 1.5 total wt pct of MM (mischmetal), Mn, and/or Zr can be processed to sheet exhibiting a wide range of properties by utilizing the strain-softening phenomenon in conjunction with carefully selected annealing conditions.

Tensile properties comparable to those of Mg alloy AZ31B-H24 may be obtained in an Mg-1MM-0.5Mn alloy by cold rolling to 50-pct reduction at 2-pct reduction per pass, finish cold rolling to 80-pct total cold reduction at up to 10-pct reduction per pass, and annealing at 200° C.

Tensile properties comparable to those of other standard Mg alloys are obtainable in Mg-0.5MM-0.5Mn, Mg-0.5MM-0.5Zr, and Mg-1.5MM, by various strain-softening and annealing combinations.

A limited number of tests indicated superplastic behavior in Mg-0.5MM-0.5Mn, Mg-0.5MM-0.5Zr, and Mg-1.5MM alloys at

350° C and 0.02 in/in per minute initial strain rate. The Mg-1.0MM-0.5Mn alloy was not tested for superplastic behavior, but based on its structural and property similarities to the other alloys processed similarly, could be expected to also exhibit some degree of superplasticity.

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