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THERMAL TREATMENT OF ALUMINUM BASE ALLOY ARTICLE

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This application is a continuation-in-part of our application Serial No. 277,097, now abandoned, filed May 1, 1963, which was a continuation-in-part of application Serial No. 107,313, filed May 3, 1961, now abandoned.

This invention relates to the thermal treatment of articles composed of aluminum base alloys containing zinc, magnesium and copper as the principal added elements, and more particularly, a treatment of such articles which produces the combination of high resistance to stress corrosion cracking, resistance to tearing and high tensile properties, particularly in the short transverse direction of thick wrought sections. The expression "short transverse direction" applied to wrought products refers to the direction which is normal to the direction of metal working or flow and in the shorter dimension of a cross section taken in a plane normal to the direction of metal flow. The expression has no application to castings.

It is recognized in the aluminum base alloy field that alloys containing zinc, magnesium and copper in the proper proportion develop high strengths at room temperature when they have been worked and then subjected to a thermal treatment consisting of a solution heat treatment, quenching to a much lower temperature, usually room temperature, and finally precipitation or age hardening by reheating to a relatively low temperature for a period of time. Although the resistance to stress corrosion cracking of alloy products treated in the foregoing manner has been satisfactory for many applications, it has been found to be inadequate under severe conditions, especially in the short transverse direction of articles of relatively thick cross section. With the development of new products made from extrusions, forgings and the like and with more severe demands for performance under adverse conditions, prior thermal treatments have been found to be deficient in developing a high resistance to stress corrosion cracking even though the strength is at a desired level.

Another property which has become important in the utilization of high strength aluminum base alloys is that known as resistance to tearing. This is a measure of toughness, i.e., resistance to fracture or rapid propagation of cracks in articles under stress. This property can be measured for comparative purposes by a form of the Kahn tear test as described in The Welding Journal, published by the American Welding Society, volume 27, page 169–S (1958). In this test an autographic load-deformation curve is obtained while a crack is initiated in and propagated across the test specimen. The area under the load-deformation curve is a measure of the energy required to initiate and propagate a crack, conveniently expressed as inch-pounds per square inch, and the energy values thus obtained indicate the relative performance of different alloys and the effect of different thermal treatments. Tests such as these have shown that the conventional thermal treatments of aluminum-zinc-magnesium-copper type of alloys have not produced as high a resistance to tearing as is desired for some applications and consequently there has been a demand for improvement in this property as well as providing adequate strength and a high resistance to stress corrosion cracking. In referring to a high resistance to tearing, this means that the unit propagation energy exceeds 50 inch-pounds per square inch.

It is an object of this invention to provide a combination of thermal treatments which are effective in developing not only a high resistance to stress corrosion cracking but also a high strength and improved resistance to tearing in articles of particular aluminum-zinc-magnesium-copper alloys, especially in articles having a relatively thick cross section. Another object is to provide a combination of solution and two-step precipitation treatments which produces a highly corrosion resistant condition, a high resistance to tearing and a high strength in articles made from particular aluminum base alloys containing zinc, magnesium, copper and one or more hardening elements as the essential added alloying components, and more particularly in articles which, before treatment, exhibit directional stress corrosion properties. Still another object is to provide a sequence of aging treatments of solution heat treated articles made of those aluminum-zinc-magnesium-copper type alloys in which the amount of zinc exceeds the magnesium and copper contents, wherein the resistance to stress corrosion cracking is controlled to a large extent by the last aging treatment of the sequence and the minimum time at temperature is determined by the alloy composition and thickness of the article.

We have found that these objects and other advantages can be achieved by solution heat treating and aging in two steps articles made of aluminum base alloys consisting essentially of aluminum, 4.5 to 14% by weight of zinc, 1.5 to 3.8% by weight of magnesium, 0.75 to 2.5% by weight of copper, and at least one hardening element selected from the group composed of 0.05 to 0.4% chromium, 0.1 to 0.75% manganese, 0.05 to 0.3% zirconium, 0.05 to 0.3% vanadium, 0.05 to 0.3% molybdenum and 0.05 to 0.3% tungsten, the ratio of magnesium to zinc being 0.2 to 0.5 part of the former to 1 part of the latter. Although the combination of high strength, high resistance to stress corrosion and tearing can be developed in both chill cast and wrought articles of these alloys, we have found that the sequence of aging treatments is especially effective in wrought articles having a relatively thick cross section and in the short transverse direction. We have discovered that the response of the articles to the aging treatments is closely connected with the proportion of alloying elements and the cross-sectional thickness of the article particularly as it affects the resistance to stress corrosion cracking. Even small differences in composition of the alloys and thickness of section of the article affect the minimum time and temperature that will produce a high resistance to stress corrosion cracking. In referring to a high strength it is to be understood that this refers to tensile and yield strengths that usually exceed 60,000 and 50,000 p.s.i., respectively. Also, it is to be understood that the strength attained by our method does not represent the maximum that can be achieved under different aging conditions, but the strength is still adequate for the required structural purposes. This level of strength plus substantial freedom from stress corrosion cracking and a high resistance to tearing offers a combination of properties not heretofore obtained in the defined alloys.

Our method consists of initially subjecting the alloy articles to a solution heat treatment which involves heating them to a temperature within the range of 750 to 1000° F., but below the temperature of incipient fusion, and holding them within that range for a length of time sufficient to obtain substantially complete solution of the zinc, magnesium and copper components. Generally this can be accomplished within a period of from 3 or 4 minutes up to 10 hours depending on the thickness of the article being treated and whether the surface of the article is directly exposed to the heating medium, thus, an article having a thickness of ½ inch can be treated in a shorter time in an air atmosphere than one which has a thickness of 2 inches and a sheet $\frac{1}{16}$ inch in thickness can be treated in less time than a plate ½ inch in thickness in the same heating medium. At the conclusion of the solution heat treatment the articles are rapidly cooled to substantially room temperature, as by quenching in water at temperatures below 160° F. Cooling in this manner serves to retain a substantial portion of the dissolved components in a state of solid solution. However, by employing hot water instead of cold water it is possible to minimize stresses induced by quenching.

The articles which have been so quenched are then subjected to a two-stage precipitation hardening treatment, the first stage of which consists of heating the articles to between 175 and 275° F., the minimum temperature that can be used for a particular alloy being dependent upon the zinc content thereof. Thus, for alloys containing less than 7.5% zinc, the minimum temperature should be 200° F., while for alloys containing more than 7.5% zinc the minimum temperature is 175° F. Moreover, those alloys containing less than 7.5% zinc should be held within the specified temperature range for a period of 5 to 30 hours but in the case of the alloys containing more than 7.5% zinc the minimum holding time can be as short as 3 hours. At lower temperatures and for shorter periods of time the precipitation is insufficient to provide the proper metallurgical condition for the application of the succeeding precipitation hardening step. At higher temperatures and for longer periods of time precipitation is carried too far which adversely affects the precipitation produced in the second stage or step.

Following the first stage, the articles are heated to a higher temperature within the range of 315 to 380° F., the minimum length of time for holding at a given temperature to attain substantial freedom from stress corrosion cracking being determined by the zinc and hardener element content of the alloy and the thickness of the articles as defined below. The holding period should not be less than 2 hours nor more than 100 hours, it having been discovered that a slightly shorter as well as a much longer period also yield the desired results as compared to the shorter period stated in our preceding application. The minimum time and temperature limits essential to develop the desired freedom from stress corrosion cracking are expressed by the equation

$$t=2.0+\frac{k}{(T-280)^4}$$

where t is the time in hours, T is the temperature in ° F. and k is a constant related to the zinc and hardening element content of the alloy and thickness of the article being aged. The values of k for different alloys and for different thicknesses of the article are set forth in the following tabulation. It should be noted that in alloys containing 7.51 to 14% zinc, chromium is regarded as a separate addition and is not included in the group of hardeners zirconium, tungsten, molybdenum and vanadium.

Percent Zn	Hardening elements present			Thickness of Section, inch	K value
	Mn	Cr	Zr, W, Mo, V and/or Cr		
4.5-7.5	X			¼-2	1.6×10^{8}
4.5-7.5			X	¼-2	6.0×10^{7}
4.5-7.5	X			Over 2	8.4×10^{7}
4.5-7.5			X	do	6.0×10^{7}
7.51-14	X			Under ¼	2.6×10^{6}
7.51-14		X		do	2.6×10^{6}
7.51-14			X	do	2.6×10^{6}
7.51-14	X			¼-1	1.4×10^{8}
7.51-14		X		¼-1	5.0×10^{6}
7.51-14			X	¼-1	3.8×10^{7}
7.51-14	X			Over 1	3.8×10^{8}
7.51-14		X		do	1.5×10^{8}
7.51-14			X	do	1.5×10^{8}

Where manganese and one or more of the other hardening elements are present and the k value for one is lower than the others, the lowest value should be employed in calculating the minimum temperature and time values. There are no k values for articles having thickness of less than ¼ inch which are made of alloys containing less than 7.5% zinc since the improvement in the resistance to stress corrosion cracking in the short transverse direction in articles having a thickness greater than ¼ inch is of primary interest and the two stage age hardening treatment, as applied to articles of the commercial alloys containing less than 7.5% zinc, does not afford any worthwhile improvement over the conventional single stage treatment in respect to attaining a satisfactory strength and increasing the resistance to stress corrosion cracking. The response of the alloys to the two staging treatment changes as the zinc content exceeds 7.5% and hence this is used as a dividing line in selecting the proper treatment.

Although values can be calculated which extend to fractions of an hour, as a practical matter it is advisable to go beyond the minimum and use a full hour's exposure or more thereby assuring substantial freedom from stress corrosion cracking.

The minimum time limits established by the equation that are essential to achieve freedom from stress corrosion cracking are based upon a large number of accelerated tests wherein the specimens were stressed to a value equal to 75% of their yield strength and exposed to alternate immersion in a 3½% NaCl solution according to established procedure for this test. Specimens that remained intact after 30 days of exposure were considered to be satisfactory. Likewise, if specimens of other alloys within our range survive such a test, the allow articles represented by the specimens would be considered to be substantially free from stress corrosion cracking. In our preferred practice the alloys containing less than 7.5% zinc are treated at 320 to 350° F. while those which contain more than 7.5% zinc are preferably treated at 320 to 360° F. Also in our preferred practice the second stage temperature should be at least 75° F. above the temperature used in the first stage. Although it is usually convenient to go directly from the first to the second stage, the articles can be cooled to room temperature at the end of the first stage and reheated to the second stage temperature.

As mentioned above, shorter times and lower temperatures for the second stage lower the resistance to stress corrosion cracking. On the other hand the use of temperatures above the maximum stated limits and longer periods of time result in excessive reduction in tensile and yield strengths.

The quenched articles should not be cold worked before the precipitation hardening treatment other than whatever working may be required to straighten them and remove any warpage and does not involve a reduction in thickness of more than about 2%.

While we are not able to offer a precise explanation of what occurs during the second stage of aging, it nevertheless appears that the precipitation differs from that produced in the first stage of precipitation hardening and the second stage creates a condition that distinctly improves the resistance to stress corrosion cracking and tearing.

In referring to stress corrosion cracking it is to be understood that this refers to the failure of the alloy article under the combined action of stress and corrosion whether the stress is applied externally or is in the nature of a residual stress. It is to be distinguished from corrosion which occurs in the absence of stress and is manifested in a general attack of the metal surface or causes pits to develop.

The alloys which respond to our novel thermal treatment and develop a high resistance to stress corrosion cracking, a high strength and high resistance to tearing contain from 4.5 to 14% by weight of zinc, from 1.5 to 3.8% of magnesium, 0.75 to 2.5% by weight of copper, and at least one hardening element selected from the group composed of 0.05 to 0.4% chromium, 0.1 to 0.75%

manganese 0.05 to 0.3% zirconium, 0.05 to 0.3% vanadium, 0.05 to 0.3% molybdenum and 0.05 to 0.3% tungsten, as stated hereinabove. The magnesium in any case should be present within the proportions of 0.2 to 0.5 part to 1 part of zinc and to attain a maximum strength and resistance to stress corrosion cracking we have found that in alloys containing less than about 10% zinc the ratio of magnesium to zinc should be 0.25 to 0.5 part of the former to 1 part of the latter. In alloys containing more than about 10% zinc the ratio should be 0.2 to 0.35 part of magnesium to 1 part of zinc. In addition to the foregoing relationship of magnesium to zinc, it is essential that the copper content of the alloy should not exceed the amount of magnesium. The composition limits and proportions between the alloying elements must be observed in order to secure the desired combination of properties yet provide an alloy that can be cast and worked.

The presence of hardening elements within the stated limits is also essential in the aluminum-zinc-magnesium-copper alloys to assist in attaining the desired properties. Although these elements are substantially insoluble in solid aluminum they nevertheless affect the response of the alloy to thermal treatment, especially the precipitation treatment, and tend to hasten precipitation as compared to the same alloy without such elements. In addition, these elements improve the resistance to stress corrosion cracking.

The alloys just described may also contain one or more of the group of grain refining elements consisting of 0.01 to 0.2% titanium, and 0.0005 to 0.002% boron. These elements serve to produce a fine grain size in the cast form of the alloy which is generally advantageous to the mechanical properties. In addition, to minimize oxidation of the molten alloy it may be helpful to add 0.001 to 0.005% beryllium.

Iron and silicon are always present as impurities. Up to 0.3% iron can be tolerated and the silicon content should not exceed 0.2% and thus avoid the formation of any substantial amount of the intermetallic compound Mg_2Si.

Our thermal treatment serves to provide a high strength and improve the resistance to stress corrosion and resistance to tearing and still attain a high strentgh of wrought products made of the above-stated alloys, the benefits of the treatment being controlled by the zinc content and thickness of section of the article being treated as stated above. The articles which are particularly benefited by the thermal treatment are those which have a relatively thick cross section such as often found in forgings, extrusions, pressings and rolled plate or shapes. A thickness of more than ¼ inch is considered to be relatively thick. In all of these cases the articles possess definite directional properties as a result of the type of hot working employed. As a consequence the tensile properties are higher in the direction in which the article is worked, usually referred to as the longitudinal direction, than in the transverse direction, i.e. normal to the longitudinal direction. The difference is particularly evident in the short transverse direction. In addition, the resistance to stress corrosion cracking is also lowest in that direction. The combination of thermal treatments described above greatly improves the resistance to stress corrosion cracking in the short transverse direction and brings it close to that in the longitudinal direction, i.e. parallel to the working.

We have found that the thermal treatments described above are also beneficial to sheet products (less than ¼ inch in thickness) of alloys containing more than 7.5% zinc. Zinc contents of such a magnitude generally have an adverse effect upon the resistance to stress corrosion cracking in sheet products that are precipitation hardened by a single step, often referred to as isothermal aging. This adverse effect of zinc appears to increase with the zinc content and hence although high strengths may be achieved, resistance to stress corrosion is reduced. By means of our series of treatments both high strength and resistance to stress corrosion cracking are obtained.

In addition to improving wrought articles having a relatively thick cross section our novel thermal treatment is also beneficial to improving the resistance to stress corrosion cracking of thick drastically chilled cast bodies made from the above-described alloys. Drastically chilled cast bodies refers to ingots, either solid or hollow, which have been cast by the direct chill or similar casting procedures as distinguished from sand and permanent mold castings. To obtain the desired improvement it is usually necessary to employ a higher temperature or a longer time, or both for solution heat treatment, to produce the requisite solution of the soluble components than in the case of the wrought articles. After the solution heat treatment and quench, the same two-stage precipitation hardening treatment should be used as employed with the wrought articles.

The improvement gained by the use of our novel process in respect to strength and resistance to stress corrosion cracking is illustrated in the following examples. The composition of the alloys employed in the tests is given in Table I below.

Table I.—Composition of alloys

Alloy	Percent Zn	Percent Mg	Percent Cu	Percent Mn	Percent Cr	Percent Ti	
A	5.69	2.53	1.67	0.00	0.22	0.05	
B	5.75	2.58	1.67	0.50	0.00	0.04	
C	6.91	2.72	1.48	0.10	0.27	0.05	
D	5.92	2.63	1.62	0.04	0.17	0.02	
E	8.25	2.89	1.39	0.5	--------	--------	
F	9.1	2.16	1.5	0.5	--------	--------	
G	10.1	3.0	2	0.5	--------	--------	
H	12.5	3.68	1.59	0.5	--------	--------	
I	12.1	3.09	1.54	0.5	--------	--------	
J	11.9	3.42	1.52	0	0.24	--------	
K	12	3.5	1.52	0	--------	--------	0.26 Zr
L	11.9	3.48	1.5	0	--------	0.02	0.24 Mo
M	10.50	2.95	1.95	--------	--------	--------	0.21 Zr

Alloys A, B and C were melted, cast into ingots and forged to bars having a cross section of 2 inches by 4 inches by conventional practice. The bars of these alloys were solution heat treated at 870° F. for 6 hours and quenched in water at a temperature of 70° F. Alloy D was cast in the form of an ingot for plate and sheet rolling rolled to 2 inch thick plate solution heat treated at 870° F. and quenched in a water spray in customary manner. Alloys E through L were melted, cast into ingots and extruded into rod ¾ inch in diameter in accordance with usual practices. Alloy M was also extruded into a sector shaped bar, each side of which was about 2 inches in length. Samples cut from the rods and the bar for test purposes were heated to 870° F. and held for 2 hours and quenched in water at 75° F.

The precipitation hardening treatments applied to the bars or rods of each of the alloys are given in Table II below.

Table II.—Precipitation hardening treatments

Alloy	First stage treatment		Second stage treatment	
	Temp., ° F.	Hours	Temp., ° F.	Hours
A (1)	225	6	350	8
(2)	250	12	375	6
(3)	270	6	375	6
B (1)	230	7	370	7
(2)	240	13	375	8
(3)	270	7	370	7
C (1)	225	6	350	8
(2)	225	6	375	4
D	250	24	325	48
E	225	6	350	8
F	225	6	350	8
G	225	6	350	8
H	225	6	350	8
I	225	6	350	8
J	225	6	350	8
K	225	6	350	8
L	225	6	350	8
M	225	6	315	96

Tensile test specimens were cut from the forged bars of Alloys A, B and C in the short transverse direction, that is, in the 2 inch dimension. Short transverse specimens were also cut from the Alloy D plate and Alloy M extrusion. The average tensile properties of these specimens are listed in Table III.

Table III.—Tensile properties in short transverse direction

Alloy	Treat ment	Tensile strength p.s.i.	Yield strength, p.s.i.	Percent elongation
A	1	72,000	61,000	9
	2	68,900	55,600	12
	3	67,200	54,300	8
B	1	70,900	60,400	8
	2	69,500	56,100	12
	3	70,700	58,900	8
C	1	72,100	60,500	6
	2	69,300	57,300	7
D		69,500	58,700	2.5
M		73,600	65,700	6.5

These results clearly indicate that a high strength was obtained as compared to a typical short transverse tensile strength of 80,000 p.s.i. and a yield strength of 68,000 p.s.i. for Alloy A in the conventionally solution heat treated and precipitation hardened condition.

Tensile test specimens of standard size were cut from the extruded and heat treated rods in the longitudinal direction and the tensile properties determined. The average values for each of the alloys are given in Table IV below.

Table IV.—Tensile properties of extruded rods

Alloy	Tensile strength, p.s.i.	Yield strength, p.s.i.	Percent elongation
E	83,000	75,900	11
F	75,400	66,700	13
G	85,800	79,100	10
H	82,600	76,800	10
I	82,300	75,600	10
J	80,700	74,300	11
K	81,400	77,100	10.5
L	82,400	76,200	10

The foregoing tensile properties compare favorably with the typical properties of extrusions of the commercial alloy having a nominal composition of 5.6% zinc, 2.5% magnesium, 1.6% copper, 0.3% chromium, and balance aluminum that have received solution heat treatment at 870° F. and have been aged 24 hours at 250° F. The typical properties of such extrusions are: 90,000 p.s.i. tensile strength, 84,000 p.s.i. yield strength and 12% elongation. The presence of chromium, zirconium and molybdenum, respectively, in Alloys J, K and L had about the same effect upon the tensile properties as manganese in Alloys H and I where the same sequence of precipitation treatments were employed.

Additional short transverse tensile test specimens of Alloys A, B and C were subjected to an accelerated stress corrosion test which consisted of applying a stress to the specimens equal to 75% of the yield strength and exposing them to an aqueous 3½% to NaCl solution by the alternate immersion method over a period of 84 days. Three specimens were used in each case. No failures occurred during the entire period. In comparison, test specimens of the same alloys solution heat treated at 870° F. and aged in one step by heating at 250° F. for 24 hours failed in 3 to 5 days in the same test. It is therefore evident that the two-step aging treatment produced substantial immunity to stress corrosion cracking in the short transverse direction.

Transverse specimens from the extruded rods, the sector-shaped bar and the rolled plate were subjected to the same stress corrosion test for a period of 30 days and all survived the test thus demonstrating a high resistance to stress corrosion cracking.

The examples of improved resistance to stress corrosion given above demonstrate the value of the two-stage precipitation treatment as compared to the conventional aging practice of heating at 250° F. for 24 hours. It is also important to observe the minimum conditions for the second stage treatment as defined by the equation stated hereinabove. This is illustrated in the treatment of transverse specimens cut from the ¾ inch thick wall of an extruded tube of an alloy having the nominal composition of 5.6% zinc, 2.5% magnesium, 1.6% copper, 0.3% chromium and balance aluminum. The extruded tube was solution heat treated at 870° F. and quenched in cold water. Sections were cut from the tube for precipitation hardening by two different practices. One group was heated 3 hours at 225° F. and then for 4 hours at 325° F. A second group was heated 7 hours at 225° F. and then 9 hours at 350° F. When subjected to the stress corrosion test described above the specimens from the first group failed in 6 to 8 days whereas those from the second group remained intact at the end of 84 days. In the first group the time at temperature was too short while in the second group the time at temperature exceeded the minimum.

The effect of our treatment upon the resistance to tearing is illustrated in the following examples wherein ingots of the different alloys containing more than 7.5% zinc were rolled to sheet 0.064 inch in thickness according to conventional practice. Specimens were cut from the sheet in both longitudinal (L) and transverse (T) directions to provide samples for the Kahn type of test. The samples were solution heat treated at 870° F. for a period of 30 minutes and quenched in water at 75° F. One set of samples (1) was precipitation hardened by heating them to 250° F. and holding for 24 hours, the conventional type of treatment, while a second set (2) was given a two-step treatment consisting of heating to 225° F. and holding for 6 hours and then heating to 350° F. and holding for 8 hours. The sheet samples were tested and the unit crack propagation energy was calculated in terms of inch pounds per square inch. The composition of the alloys used for making the sheet and the unit propagation energy in both longitudinal and transverse directions are given in Table V below.

Table V.—Alloy composition and resistance to tearing

Alloy composition, percent					Direction	Aging treatment	Unit prop. energy, inch-lbs./sq. in.
Zn	Mg	Cu	Zr	Cr			
7.95	3.46	1.52	0.22	----	L	1	0
7.95	3.46	1.52	0.22	----	T	1	0
7.95	3.46	1.52	0.22	----	L	2	295
7.95	3.46	1.52	0.22	----	T	2	110
9.98	2.98	1.50	0.17	----	L	1	0
9.98	2.98	1.50	0.17	----	T	1	0
9.98	2.98	1.50	0.17	----	L	2	345
9.98	2.98	1.50	0.17	----	T	2	200
11.90	3.46	1.52	0.21	----	L	1	0
11.90	3.46	1.52	0.21	----	T	1	0
11.90	3.46	1.52	0.21	----	L	2	105
11.90	3.46	1.52	0.21	----	T	2	75

It is apparent that the two-step precipitation hardening increased the resistance to tearing although the treatments were not equally effective in each case. The improvement is significant, however, and is particularly useful in the design and us of highly stressed structures. The presence of zirconium is not essential to gaining the improvement, its benefit being confined to improving the structure of the ingot from which the sheet was rolled.

The benefit of our novel process as applied to a drastically chilled cast body is illustrated in the following example. An alloy having the nominal composition of 5.6% zinc, 2.5% magnesium, 1.6% copper, 0.3% chromium and balance aluminum was cast by the direct chill procedure in the form of an ingot having a thickness of 4 inches. Test bars cut from the ingot were solution heat treated at 960° F. for 24 hours, quenched and aged in two steps, 6 hours at 225° F. and 8 hours at 350° F. The average tensile strength was 74,100 p.s.i., the yield strength 66,200 and the elongation 4.5%. Bars subjected to the alternate immersion corrosion test referred to above and stressed an amount equal to 75% of the yield strength showed no failures over a period of 133 days.

Having thus described our invention and certain embodiments thereof, we claim:

1. The method of thermally treating articles composed of an alloy consisting essentially of aluminum, 4.5 to 14% zinc, 1.5 to 3.8% magnesium, 0.75 to 2.5% copper and at least one hardening element selected from the group composed of 0.05 to 0.4% chromium, 0.1 to 0.75% manganese, 0.05 to 0.3% zirconium, 0.05 to 0.3% vanadium, 0.05 to 0.3% molybdenum and 0.05 to 0.3% tungsten, wherein the proportion of magnesium to zinc is within the range of 0.2 to 0.5 part of magnesium to 1 part zinc and the copper content does not exceed that of magnesium, said treatment comprising

heating said articles to a temperature between 750 and 1000° F., but below the temperature of incipient fusion, for a sufficient length of time to produce substantially complete solution of the soluble components,

quenching said articles to substantially room temperature,

precipitation hardening said quenched articles in two steps according to the following schedule:

first step: for alloys containing less than 7.5% zinc, heating the articles to 200 to 275° F. and holding for a period of 5 to 30 hours;

for alloys containing more than 7.5% zinc, heating the articles to 175 to 275° F. and holding for a period of 3 to 30 hours;

second step: heating the articles to 315 to 380° F. for a period of 2 to 100 hours, the minimum time and temperature relationship to achieve high resistance to stress corrosion being expressed by the equation:

$$t=2.0+\frac{k}{(T-280)^4}$$

where t is the time in hours, T is the precipitation temperature and k is a constant that varies with the zinc and hardening element content of the alloy and the thickness of the article being treated as follows:

Percent Zn	Hardening elements present			Thickness of Section, inch	K value
	Mn	Cr	Zr, W, Mo, V and/or Cr		
4.5-7.5	X			¼-2	1.6×10^8
4.5-7.5			X	¼-2	6.0×10^7
4.5-7.5	X			Over 2	8.4×10^7
4.5-7.5			X	do	6.0×10^7
7.51-14	X			Under ¼	2.6×10^6
7.51-14		X		do	2.6×10^6
7.51-14			X	do	2.6×10^6
7.51-14	X			¼-1	1.4×10^8
7.51-14		X		¼-1	5.0×10^6
7.51-14			X	¼-1	3.8×10^7
7.51-14	X			Over 1	3.8×10^8
7.51-14		X		do	1.5×10^8
7.51-14			X	do	1.5×10^8

cooling said articles to room temperature,

whereby the treated articles acquire a high strength, a high resistance to stress corrosion cracking and a high resistance to tearing.

2. The method according to claim 1 wherein the solution heat treatment is applied to wrought products over a period of 3 minutes up to 10 hours.

3. The method according to claim 1 wherein the second stage of precipitation hardening for articles of alloys containing less than 7.5% zinc consists of heating the articles to between 320 and 350° F. and for alloys containing more than 7.5% zinc, the articles are heated to between 320 and 360° F.

4. The method according to claim 1 wherein the second stage of precipitation hardening consists of heating to a temperature at least 75° F. above that of the temperature used in the first stage.

5. The method according to claim 1 wherein the alloy also contains at least one of the grain refining elements of the group composed of 0.01 to 0.2% titanium and 0.0005 to 0.002% boron.

6. The method according to claim 1 wherein the alloy also contains from 0.001 to 0.005% beryllium.

7. The method according to claim 1 wherein the article to be treated is in wrought form and possesses directional properties.

8. The method according to claim 1 wherein the article is in the form of a drastically chilled casting.

References Cited by the Examiner

FOREIGN PATENTS

544,329 4/42 Great Britain.

OTHER REFERENCES

"Precipitation From Solid Solution," by the A.S.M., Cleveland, Ohio, pages 179–181.

DAVID L. RECK, *Primary Examiner.*

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,198,676 August 3, 1965

Donald O. Sprowls et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 3, line 75, for "lower-" read -- lower --; column 4, line 3, for "thickness" read -- thicknesses --; line 14, for "staging" read -- stage aging --; line 31, for "allow" read -- alloy --; column 5, line 1, after "manganese" insert a comma; column 9, line 5, for "us" read -- use --; column 10, line 54, for "544,329" read -- 544,439 --.

Signed and sealed this 15th day of February 1966.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents