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3,741,827

AGE HARDENING PROCESS AND PRODUCT

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4 Claims

ABSTRACT OF THE DISCLOSURE

An aluminium - zinc-magnesium-copper alloy includes 0.12–0.20% zirconium and 0.2–0.4% silver and develops high strength and good stress corrosion characteristics when subjected to solution heat treatment and quenching, followed by artificial ageing. It is an advantage of this alloy that it is relatively insensitive to the rate of quenching. Particularly satisfactory properties are developed when the quenched alloy is heated to ageing temperature at a rate not exceeding 30° C./hour.

The present invention relates to aluminium alloys and in particular to aluminium alloys, which after appropriate heat treatment develop high mechanical strength and at the same time exhibit high resistance to stress corrosion cracking.

It is already known that aluminium alloys containing as the principal alloying elements zinc, magnesium and copper develop high strength when subjected to solution heat treatment, quenching and age hardening. It is already known that the stress corrosion characteristics of this class of alloys can be improved by the addition of a small amount of chromium and/or manganese. When this measure is adopted however the physical properties of the alloy are very dependent upon the thermal treatment and in particular upon the rate of quench. Thus it is found that, while a reduction of the quenching rate improves resistance to stress corrosion cracking, it also markedly reduces the mechanical strength of the alloy. The alloy containing manganese and chromium is markedly quench-sensitive and thus heavy sections of the alloy will exhibit different properties as regards both physical strength and stress corrosion at different levels because inevitably the quench rate is markedly slower at the centre of a heavy section than it is at the surface.

It is an object of the present invention to provide an improved aluminium alloy of the present class, which will develop high mechanical strength and simultaneously exhibit good resistance to stress corrosion cracking after appropriate heat treatment and which is less sensitive to quench rate than the known alloy, containing chromium and manganese, referred to above. In addition the heat treated alloy is required to exhibit satisfactory fracture toughness characteristics.

It is already known that the stress corrosion characteristics of the present class of alloy can be improved by raising the final ageing temperature, instead of reducing the quenching rate. That measure also leads to a reduction in the strength of the heat-treated alloy in the case of known alloys of the present class. It has heretofore therefore been impossible to heat-treat alloys of the present class to develop at the same time both high strength and good resistance to stress corrosion by applying a final ageing treatment at a relatively high temperature, such as 175° C. It has been conventional to perform the final artificial ageing treatment at a temperature of about 135° C.

The present invention provides a novel aluminium alloy

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containing zinc, magnesium and copper. A novel final ageing heat treatment for developing high strength and good stress corrosion characteristics in the alloy after it has been subjected to a conventional solution heat treatment and quench, has also been achieved.

According to the present invention an aluminium alloy comprises

	Percent
Zn -----	5.2–6.5
Mg -----	2.2–3.2
Cu -----	¹ 0.3–1.5
Zr -----	0.12–0.20
Ag -----	0.2–0.4
Fe -----	0.15 max.
Si -----	0.12 max.
Al and normal impurities -----	Balance

¹ Preferably.

It is preferred that the total impurities (including iron and silicon) should be a maximum of about 0.2% and this can readily be achieved by employing 99.8% commercial purity aluminium as the base for the alloy. Titanium, manganese and chromium are tolerable as impurities in amounts up to about 0.05%. To obtain the best results the silver content should be in the range of 0.30–0.35% and in practice it is preferred that there should be at least 0.25% silver.

According to a further feature of the invention, high strength and good stress corrosion characteristics are developed by subjecting the alloy after solution heat treatment and quenching which comprises heating the alloy to a final ageing temperature in the range of 165°–175° C., preferably 170°–175° C., at a very slow rate not exceeding 30° C./hr. and preferably at or below about 20° C./hr. After heating the alloy is held at the ageing temperature for at least 4 hours and preferably about 6 hours at a temperature in the range of 170°–175° C.

The very carefully selected balance of zirconium and silver results in an alloy which is relatively insensitive to rate of quench, as will be demonstrated below, and which develops high strength and good stress corrosion characteristics when subjected to the indicated heat treatment. It is also of considerable importance to maintain iron and silicon impurities within the values indicated, since the fracture toughness of the heat-treated alloy falls away rapidly if the maximum iron impurity exceeds the quoted level.

Reference is hereinafter made to the following tests.

TEST 1

Rolling ingots 8 in. x 24 in. x 1000 lbs. of the following composition were cast by direct chill (D.C.) process: Zn 6.09%, Mg 2.78%, Cu 1.01%, Zr 0.13%, Ag 0.30%, Fe 0.13%, Si 0.06%, Ti 0.06%, Al balance.

The ingots were stress-relief annealed at 430° C. for 12 hours in an electrically heated air-circulating furnace. They were then scalped, preheated at 460° C. for 16 hours and directly rolled to a final gauge of 2¾ in. at a reduction of ¼ in. per pass, the total reduction being 63.5%.

Pieces were removed from each plate and solution heat-treated at 465° C. for 3 hours. Half the pieces from each plate were quenched into water at 20° C. giving a quenching rate at the centre of each piece of 200° C./sec.

The remainder were quenched into a fluidised bed at 150° C.; the rate of quench now being 1° C./sec. Each piece was aged at room temperature (20° C.) for 7 days and then heated at a controlled rate of 20° C./hr. in an electrically heated air-circulating furnace to a final tem-

perature of 165° C., being maintained at this temperature for 8 hours.

The following tensile properties were obtained:

Cooling rate	Longitudinal			Long transverse			Short transverse		
	0.1% P.S., t.s.i.	U.T.S., t.s.i.	Elong. percent 4 A	0.1% P.S., t.s.i.	U.T.S., t.s.i.	Elong. percent 4 A	0.1% P.S., t.s.i.	U.T.S., t.s.i.	Elong. percent 4 A
200° C./second.....	32.95	35.65	12.6	30.75	34.2	10.7	30.25	33.8	7.3
1° C./second.....	24.8	29.9	12.8	24.5	29.55	8.8	24.45	29.4	6.3

NOTE.—T.s.i.=Ton/in.²

It will be seen from the above data that even with a quench rate as low as 1° C./sec. acceptable tensile properties are obtained. The quench rates occurring in practice in quenching are never as low as 1° C./sec. even when relatively thick heat-treated workpieces or plates are quenched. As a result of the low sensitivity to variations in quenching rate such thick materials will possess fairly uniform strength characteristics throughout.

TEST 2

Specimens 0.160 in. diameter were prepared in the short-transverse direction after both fast (200° C./sec.) and slow (1° C./sec.) quenching. All received an identical surface finish and were loaded axially at stresses of 90, 75, 50, 30 and 20% of their respective 0.1% Proof Stress. They were immersed in a 3% NaCl electrolyte and polarised anodically by application of a current density of 5 ma./sq. in Times to failure were recorded to the nearest minute.

ENDURANCES

Percent P.S.	Stress absolute (ton/in. ²)		Endurance (min.)	
	Cooling rate 1° C./sec.	Cooling rate 200° C./sec.	Cooling rate 1° C./sec.	Cooling rate 200° C./sec.
90.....	22.0	27.23	369	460
75.....	18.3	22.69	1,262	1,072
50.....	12.2	15.13	1,888	4,096
30.....	7.3	9.075	4,147	5,952
20.....	4.9	6.05	7,502	8,238

The above test results are comparable both for strength and endurance with those obtainable with the known alloy containing chromium and manganese as alloying elements, whilst the results show that the alloy of the present invention is relatively insensitive to the rate of quench to which it is subjected.

TEST 3

12 in. x 27 in. x 40 in. rolling ingots of the following compositions were cast by the direct chill (D.C.) process: Zn 5.99%, Mg 2.41%, Cu 1.03%, Zr 0.12%, Fe 0.13%, Si 0.06%, Ti 0.03%, Ag 0.29%, Al balance.

Prior to scalping, the ingots were stress-relief annealed at 460° C. for 24 hours. They were preheated at 440° C. for 20–24 hours and directly rolled to 2½ in. plate, the total reduction being 77%. The plates were solution heat-treated at 465° C., cold water quenched and controlled stretched 2%. After a period of natural ageing at room temperature, they were then artificially aged in the following way:

Heated at approximately 20° C./hr. to 170±2° C. and held at this temperature for 6 hours.

The following tensile results were obtained.

Test direction	0.2% P.S., t.s.i.	U.T.S., t.s.i.	Elong. percent on 5D
Longitudinal.....	34.05	37.0	13
Long-transverse.....	30.4	33.8	10
Short-transverse.....	28.6	33.4	4.2

The fracture toughness of this material was determined in the long-transverse direction and the following result obtained:

$$K_{Ic}=29,600 \text{ lb./in.}^2\sqrt{\text{in.}}$$

In atmospheric stress-corrosion tests, in an industrial environment, using uni-axially loaded (to 0.7% of their respective short-transverse U.T.S.) specimens remained

unbroken after 25 days.

This result indicates a satisfactory fracture toughness.

- 15 By contract figures of K_{Ic} =circa 20,000 lb./in.²√in. are obtained when the iron and silica levels are increased above the indicated levels, as would result, for example, from the use of 99.5% commercial purity aluminium.

- 20 Such poor results are believed to be due to the presence of coarse intermetallic particles and indicate the importance of employing base metal of adequate purity for the production of the alloys of the invention.

- 25 The full advantages of the alloy of the invention are obtained as a result of the slow heating to the final ageing temperature. The inclusion of zirconium, as a replacement for chromium and manganese, results in the retention of a super-saturated solid solution after quenching. The prolonged heat treatment, in which the temperature is gradually increased to the final ageing temperature permits a fine uniform precipitate to form throughout the alloy and in conjunction with the final prolonged heating at a relatively high ageing temperature of 165°–175° C. allows stresses in the alloy to dissipate, thereby impeding stress corrosion crack propagation.

What is claimed is:

1. Procedure for producing an aluminum alloy having high strength and good stress corrosion characteristics, said procedure comprising artificially aging an alloy consisting essentially of

	Percent
Zn	5.2–6.6
Mg	2.2–3.2
Cu	0.3–1.5
45 Zr	0.12–0.20
Ag	0.2–0.4
Fe	0.15 max.
Si	0.12 max.
50 Al and normal impurities	Balance

after subjecting the said alloy to a conventional solution heat treatment and quench, said artificial aging comprising

- 55 (a) heating the alloy at a rate not exceeding 30° C./hour to a final temperature in the range of 165° C.–175° C. at a rate of not more than 30° C. per hour; and
 (b) holding the alloy at said final temperature for at least four hours.
- 60 2. An aluminium alloy having high strength and good stress corrosion characteristics comprising an alloy consisting essentially of the following composition:

	Percent
65 Zn	5.2–6.5
Mg	2.2–3.2
Cu	0.3–1.5
Zr	0.12–0.20
Ag	0.2–0.4
70 Fe	0.15 max.
Si	0.12 max.
Al and normal impurities	Balance

- 75 the said alloy having been subjected to a conventional solution heat treatment and quench followed by arti-

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cial ageing in which the alloy was heated at a rate not exceeding 30° C./hour to a final temperature in the range of 165° C.-175° C. at a rate of not more than 30° C. per hour, the alloy having been held at said final temperature for at least 4 hours.

3. An aluminium alloy according to claim 2 in which the impurity content, including iron and silicon, is held below about 0.2%.

4. An aluminium alloy according to claim 3 in which the final heat treatment is performed at a temperature within the range of 170°-175° C. for about 6 hours.

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RICHARD O. DEAN, Primary Examiner

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,741,827 Dated June 26, 1973

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It is certified that error appears in the above-identified patent
and that said Letters Patent are hereby corrected as shown below:

Column 2, line 17, after "Preferably" insert
--0.8 - 1.0-- .

Column 4, line 42, "5.2 - 6.6" should read --5.2 - 6.5-- .

Signed and sealed this 5th day of March 1974.

(SEAL)

Attest:

EDWARD M. FLETCHER, JR.
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