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SHEETS OF ALUMINUM ALLOY AND METHODS OF MANUFACTURING SAME

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No Drawing. Filed Sept. 28, 1964, Ser. No. 399,830

Claims priority, application Great Britain, Oct. 2, 1963, 38,861/63

14 Claims. (Cl. 29—197.5)

This invention relates to improvements in sheets of aluminium alloy and methods of manufacturing same.

The invention is concerned particularly with methods of producing aluminium alloy sheet materials intended to withstand high temperatures. Aluminium alloy sheet which is used, for example, for the skins of aircraft intended to fly at supersonic speeds requires to have a high level of static tensile strength after thousands of hours at the service temperatures to which such skins are exposed and good creep resistance and fatigue strength at the same temperatures. The sheets may be used in the unclad form or may be clad on one or both main faces with a layer of commercially pure aluminium or aluminium containing from about 0.8 to 1.2 percent by weight of zinc or with a layer of heat-treatable corrosion-resistant aluminium base alloy containing a similar amount of zinc. The thickness of each cladding layer is usually between 3 and 7 percent of the total thickness of the clad sheet.

Suitable alloys for heat-treatable corrosion-resistant claddings of the kind mentioned are the well known aluminium-magnesium-silicon type series of alloys (hereinafter referred to as the series hereinbefore defined) the compositions of which, by weight, fall within the range 0.4 to 1.4 percent magnesium, 0.2 to 1.3 percent silicon, 0.0 to 1.0 percent manganese, 0.0 to 0.3 percent chromium, the remainder being aluminium together with the normal amounts of impurities and grain refining elements found in such alloys, except that in the make-up of any of these alloys there is included an addition of between about 0.8 and 1.2 percent by weight of zinc. The preferred alloy for use as cladding is that according to British Standards Specification 1470:HS.30 with an addition of between 0.8 and 1.2 percent by weight of zinc.

It is well known that the creep resistance of aluminium alloys is effected by the grain-size of the material, the coarser the grain the better being the creep resistance. On the other hand, too coarse a grain is undesirable in sheet materials if the latter are to be formed by bending, stretching or drawing, because under these conditions the resulting product may suffer from the deleterious effect visible on the formed surfaces and known as "orange peeling." In the forged condition aluminium alloys can be produced with a controlled coarse grain-size which enhances the creep resisting properties and there is no difficulty associated with this grain-size because forgings are not normally formed after manufacture.

It is customary to make aluminium alloy sheet from recrystallised hot rolled slab by imposing heavy cold rolling reductions of the order of 50 to 60 percent or more, between inter-stage recrystallising anneals, and finishing to the required thickness with a final cold rolling reduction of the same order of magnitude. After solution heat-treatment, the resulting sheet has a relatively fine grain-size which ensures ease of formability.

The principal factors affecting the ultimate grain-size in sheet are the degree of reduction in the final cold roll-

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ing and the time and the temperature of the subsequent heat treatment. There is a critical range between about 1.5 percent and 11 percent in the reduction during final cold rolling which produces large grains on subsequent solution heat-treatment. As a result of experimental research, we have now discovered that, with certain aluminium alloys having a composition as hereinafter defined, by controlling the amount of reduction in the cold rolling stages and by a careful selection of the time and the temperature for solution heat-treatment there can be produced a sheet with approximately equiaxed grains which are small enough to prevent the incidence and adverse effects of "orange peeling" after forming and at the same time the material, after an artificial ageing treatment, possesses a very much better creep resistance than sheet produced in the normal way.

For the attainment of improved creep resistance, together with the retention of fine grains in the sheets, we have found it necessary to control the percentage cold rolling reductions imposed on the hot-rolled slabs during the sheet manufacture between the inter-stage recrystallising annealing treatments and in the final cold rolling reduction to the required gauge size to between about 12 and 50 percent at each stage. The preferred range of these cold rolling reductions is between 14 and 45 percent.

It is customary to minimise the time for which the sheet is subjected to the temperature of solution heat-treatment to less than 15 minutes to avoid grain growth and, in the case of clad sheet, to prevent the excessive diffusion of the copper and magnesium alloying elements into the cladding and consequent reduction in the protection against corrosion afforded by the cladding layer, but we have discovered that it is necessary in our method to give a time of, for example, about 30 minutes in a salt bath in the case of sheet 0.064 in. thick in order to obtain optimum creep resistance. The time required for solution heat-treatment is dependent on the thickness of the sheet and varies from a minimum of about 15 minutes up to about 50 minutes within the solution heat-treatment temperature range. Although the solution heat-treatment temperature may lie between 525° C. and 545° C., the preferred temperature range is between 528° C. and 535° C. In order to produce the required level of tensile, creep and fatigue properties the sheets are quenched after solution heat-treatment and then artificially aged, preferably at a temperature of 175° to 210° C. for from 4 to 30 hours.

According to the present invention, therefore, there is provided a method of manufacturing aluminium alloy sheet wherein a hot-rolled slab of an aluminium alloy having a composition as hereinafter defined is subjected to a plurality of cold rolling reductions each of which is not substantially less than 12% and not substantially more than 50%, a recrystallising anneal being given after each reduction except the last, the cold-rolled sheet is subjected to solution heat-treatment for not substantially less than 15 minutes and not substantially more than 50 minutes at a temperature not substantially less than 525° C. and not substantially more than 545° C. and the sheet is then quenched and artificially aged.

We have also discovered that by "recovery annealing" the sheet after the final cold rolling reduction, for a selected time at a controlled temperature below that at which grain growth can substantially take place, there is produced a sheet in which, on subsequent solution heat-treatment and artificial ageing the said sheet in order to

produce the desired static tensile properties, the creep resistance of the material is unexpectedly even further improved. This improvement in creep resistance is more pronounced if the original cast rolling slabs are not homogenised prior to the hot rolling operation. Homogenisation is the thermal treatment which is sometimes given to cast rolling slabs in order to ensure that the soluble intermetallic constituents are substantially in solution or evenly dispersed in the alloy before the hot working is imposed on the material. It consists of soaking the slabs at, or just below, the solution heat-treatment temperature for a time which depends on the thickness and weight of the slabs and may be from about 10 to about 48 hours. After this treatment, the slabs may be allowed to cool to the required temperature and hot-rolled immediately, or cooled down to room-temperature and subsequently heated to the required temperature prior to hot-rolling.

The conditions which we have found satisfactory for the "recovery annealing" treatment are between 15 minutes and 3 hours at a selected temperature between 150° and 300° C., although a similar unexpected improvement has been observed in the creep resistance of sheet material stored at room temperature for several months after final cold rolling and prior to solution heat-treatment and artificial ageing.

To obtain the benefit of our discoveries, it is necessary, as hereinbefore mentioned, to control the composition of unclad material and the aluminium alloy core of clad sheet within very close limits. The aluminium alloy which we use is compounded of the following elements in the following proportions by weight:

	Percent
Copper	2.2 to 2.7
Magnesium	1.3 to 1.7
Silicon	0.12 to 0.25
Iron	0.9 to 1.2
Nickel	0.9 to 1.4
Titanium	0.02 to 0.15

One or more of the elements, antimony, beryllium, cerium, chromium, cobalt, manganese, molybdenum, niobium, silver, vanadium, zirconium and zinc may be present in an amount up to 0.3 percent by weight each but not more than 1 percent in total. The content of the silver may be increased above 0.3 percent up to 0.8 percent (in which case the aforesaid maximum total of 1 percent may be correspondingly increased) if the copper content is reduced below 2.5 percent by at least as much as the amount of silver exceeds 0.5 percent. One or more of the elements barium, calcium and strontium may be present in a total amount of not more than 0.2 percent. One or more of the following elements may be present up to a maximum amount of 0.1 percent total: tin, arsenic, bismuth, cadmium, boron, lithium, sodium and potassium. Aluminium the remainder.

This alloy is hereinafter referred to in this specification and in the claims as "an aluminium alloy having a composition as hereinbefore defined."

Aluminium core alloys of the same general nature as that defined above are normally cast by the semi-continuous or continuous casting process and it is desirable that the alloys which we use should be cast by one of said processes.

Sheet can be produced by the method according to this invention with an average intercept diameter of the grains in the core of between 0.0399 millimeter, which is equivalent to A.S.T.M. No. 6.0 determined by the standard A.S.T.M. method of estimating the average grain-size of metals, and 0.0168 millimetre, which is equivalent to A.S.T.M. No. 8.5. The method used for determining the A.S.T.M. numbers for the average grain-size is that described for the comparison procedure in the American Society for Testing and Materials Standard Designation: E.112-61. The sheets are formable without the production of the "orange peeling" effect and their creep resist-

ance is very much better than that of sheets processed in the normal way. When heat-treated sheet has a grain-size coarser than about A.S.T.M. No. 5.0, the "orange peeling" effect is likely to occur during the subsequent forming operations by bending, drawing or stretching. The more severe forming operations may even result in visible intergranular cracking on the surface of the sheet.

Examples of clad aluminium alloy sheets and methods of producing the same in accordance with the invention will now be described in detail, and their properties compared with those of sheets prepared by standard and non-standard procedures outside the scope of the present invention.

Example 1

In the following seven clad sheets A to G, inclusive, the composition of all the aluminium alloy cores was within the limits:

	Percent
Copper	2.35 to 2.65
Magnesium	1.35 to 1.65
Silicon	0.18 to 0.22
Iron	0.95 to 1.15
Nickel	1.00 to 1.30
Titanium	0.05 to 0.10

Aluminium and not more than traces of one or more other elements hereinbefore mentioned—the remainder.

A series of melts was made up to the above composition and cast into rolling slabs which were not homogenised but were processed and clad with cladding sheets in the normal way by hot rolling down to an appropriate thickness. The cladding was on both main faces of the sheet and each layer was a nominal 5 percent of the total thickness of each sheet.

The cladding used for sheets A, B, C and D was compounded of commercially pure aluminium and from 0.8 to 1.2 percent of zinc by weight. For sheets E, F and G, the cladding was a heat-treatable corrosion resistant alloy made from commercially pure aluminium and about 0.7 percent of magnesium, 1.0 percent of silicon, 0.55 percent of manganese with an addition of about 1.0 percent of zinc, all by weight, together with the normal impurities present in such alloys. Apart from the zinc addition, this latter alloy conformed to the compositional requirements of the British Standards Specification 1470: HS.30.

The sheets were then subjected to cold rolling reductions. The percentage reductions used during each of the inter-stage cold rolling operations and in the final cold rolling operation are shown in Table 1. The partially rolled sheets were given a recrystallising anneal of 6 to 8 hours at 360° to 385° C. between the inter-stage reductions by cold rolling. Sheet A was solution heat-treated on a salt bath for 10 minutes at 530° C. Sheets B to G, inclusive, were solution heat-treated for 40 minutes at 530° C. After quenching, the minimum amount of cold work was imposed on the sheets during the normal flattening or levelling and stretching operations because excessive amounts of cold work at this stage have a deleterious effect on the subsequent creep resistance. All the sheets were finally artificially aged for 20 to 24 hours at 190° C. in a forced air circulation furnace.

Suitable specimens were cut from the heat-treated sheets and prepared and assessed for grain-size of the core material by the method described for the comparison procedure in A.S.T.M. Standard: E. 112-61, and the results are shown in Table 1. Specimens were also prepared in a direction transverse to the direction of final rolling for testing under standard tensile creep test conditions in accordance with British Standards Specification 3500: Part 3: 1962, at 150° C. under a stress of 12 tons per square inch. The percentage total plastic creep strains, after 500 hours, for this series of tests are also shown in Table 1.

TABLE 1

Sheet Identification.....	Cladding						
	Al+1% Zn				HS.30+1% Zn		
	A	B	C	D	E	F	G
Percentage Inter-Stage Cold Reductions Prior to Last Recrystallising Anneal.....	55	37	41	37	37	37	37
Percentage Cold Reduction After Last Recrystallising Anneal.....	47	15	24	27	55	15	27
Thickness of Sheet in Inches.....	0.064	0.065	0.064	0.055	0.035	0.065	0.055
A.S.T.M. Grain-Size Number After Full Heat Treatment.....	7.5	6.5	7.0	7.5	8.0	6.5	7.0
Percentage Total Plastic Creep Strain With a Load of 12 Tons/Sq. Inch at 150° C. After 500 Hours.....	0.195	0.093	0.106	0.120	0.131	0.076	0.107

In comparing the sheets clad with the non-heat treatable cladding Al+1% Zn, it can be seen that the sheets B, C and D made according to the present invention have a suitable small grain-size and at the same time the creep resistance of these sheets is very much better than that shown by sheet A, made according to the conventional or standard procedure. Similarly, sheets F and G, made according to the present invention and clad with the heat-treatable HS.30+1% Zn cladding, have a small grain-size and their creep resistances are very much superior to that of the corresponding sheet E made according to a non-standard procedure but outside the scope of this invention.

Example 2

As an example of the effect of "recovery annealing" without first homogenising the cast rolling slab, some of the sheets made according to the procedure used for sheet C and sheet F in Example 1 were given an anneal for two hours at 275° after the final cold rolling reductions shown in Table 1 and prior to solution heat-treatment in a salt bath for 30 minutes at 530° C. After quenching, the sheets were then artificially aged for 20 hours at 190° C. and tested for grain-size and tensile creep tested under a stress of 12 tons per square inch at 150° C. as in the previous example. The A.S.T.M. grain-size number of sheet C was found to be 7.0 and the percentage total plastic creep strain after 500 hours was only 0.083. In the case of sheet F, after this treatment, the A.S.T.M. grain-size number was 6.5 and the percentage total plastic creep strain after 500 hours was only 0.068.

Example 3

The effects of prior homogenisation of the cast rolling slabs, with and without subsequent "recovery annealing" of the finally cold rolled sheet, are shown by the following example:

A cast rolling slab was made up according to the composition given in Example 1 and homogenised for 20 hours at 520° C. before hot rolling and cladding, as in sheets A to D inclusive, on both main faces with a layer equal to about 5 percent of the total thickness of the sheet, with an alloy compounded of commercially pure aluminium and nominally 1 percent of zinc by weight. The hot-rolled slab was cold rolled in substantially the same way as for sheet C in Example 1. One portion of the resulting sheet was not "recovery annealed" after the final cold rolling operation and this was designated sheet J. A second portion was "recovery annealed" for 2 hours at 285° C. after the final cold rolling and this was designated sheet K.

Suitable specimens were cut from both sheets J and K and solution heat-treated and artificially aged at the same temperatures and for the same times as used in Example 2. The specimens were then tested for grain-size and tensile creep tested under a load of 12 tons per square inch at 150° C. for 500 hours. The results of these tests are shown in Table 2.

TABLE 2

Sheet Identification.....	Cladding	
	Al+1% Zn	
	J	K
Percentage Inter-Stage Cold Reductions Prior to Last Recrystallising Anneal.....	42	42
Percentage Cold Reduction After Last Recrystallising Anneal.....	24	24
Thickness of Sheet in Inches.....	0.064	0.064
A.S.T.M. Grain-Size Number After Full Heat Treatment.....	7.0	6.5
Percentage Total Plastic Creep Strain With a Load of 12 Tons/Sq. Inch at 150° C. After 500 Hours.....	0.098	0.090

I claim:

1. A method of manufacturing aluminium alloy sheet comprising the steps of forming a slab suitable for cold-rolling into a sheet of an alloy consisting essentially of 2.2 to 2.7 percent by weight copper, 1.3 to 1.7 percent by weight magnesium, 0.12 to 0.25 percent by weight silicon, 0.9 to 1.2 percent by weight iron, 0.9 to 1.4 percent by weight nickel, 0.02 to 0.15 percent by weight titanium and the remainder aluminium, subjecting said slab to a plurality of cold rolling reductions each of which is not substantially less than 12% and not substantially more than 50%, subjecting the cold-rolled sheet to solution heat-treatment for not substantially less than 15 minutes and not substantially more than 50 minutes at a temperature not substantially less than 525° C. and not substantially more than 545° C., and quenching and then artificially ageing the sheet.
2. A method according to claim 1 wherein each of the cold-rolling reductions is between 14 and 45%.
3. A method according to claim 1 wherein the solution heat-treatment is effected at a temperature between 528° C. and 535° C.
4. A method according to claim 1 wherein the artificial ageing is effected at a temperature not substantially less than 175° C. and not substantially more than 210° C. for not substantially less than 4 hours and not substantially more than 30 hours.
5. A method according to claim 1 wherein said slab is homogenised.
6. A method of manufacturing aluminium alloy sheet comprising the steps of forming a slab suitable for cold-rolling into a sheet of an alloy consisting essentially of 2.2 to 2.7 percent by weight copper, 1.3 to 1.7 percent by weight magnesium, 0.12 to 0.25 percent by weight silicon, 0.9 to 1.2 percent by weight iron, 0.9 to 1.4 percent by weight nickel, 0.02 to 0.15 percent by weight titanium and the remainder aluminium, subjecting said slab to a plurality of cold rolling reductions each of which is not substantially less than 12% and not substantially more than 50%, subjecting the cold-rolled sheet to recovery annealing for not substantially less than 15 minutes and not substantially more than 3 hours at a tempera-

ture not substantially less than 150° C. and not substantially more than 300° C., subjecting the sheet to solution heat-treatment for not substantially less than 15 minutes and not substantially more than 50 minutes at a temperature not substantially less than 525° C. and not substantially more than 545° C., and quenching and then artificially ageing the sheet.

7. A method according to claim 6 wherein said slab is unhomogenised.

8. A method of manufacturing clad aluminium alloy sheet comprising the steps of forming a hot-rolled slab of an alloy consisting essentially of 2.2 to 2.7 percent by weight copper, 1.3 to 1.7 percent by weight magnesium, 0.12 to 0.25 percent by weight silicon, 0.9 to 1.2 percent by weight iron, 0.9 to 1.4 percent by weight nickel, 0.02 to 0.15 percent by weight titanium and the remainder aluminium, and having cladding of material selected from the group consisting of commercially pure aluminium, aluminium containing not substantially less than 0.8 and not substantially more than 1.2 percent by weight of zinc, and a heat-treatable corrosion-resistant aluminium base alloy consisting essentially of 0.4 to 1.4 percent by weight magnesium, 0.2 to 1.3 percent by weight silicon, 0.0 to 1.0 percent by weight manganese, 0.0 to 0.3 percent by weight chromium, about 0.8 to about 1.2 percent by weight zinc and the remainder aluminium, subjecting said slab to a plurality of cold rolling reductions each of which is not substantially less than 12% and not substantially more than 50%, subjecting the cold-rolled sheet to solution heat-treatment for not substantially less than 15 minutes and not substantially more than 50 minutes at a temperature not substantially less than 525° C. and not substantially more than 545° C., and quenching and then artificially ageing the sheet.

9. A method of manufacturing aluminium alloy sheet comprising the steps of forming a hot-rolled slab of an alloy consisting essentially of 2.2 to 2.7 percent by weight copper, 1.3 to 1.7 percent by weight magnesium, 0.12 to 0.25 percent by weight silicon, 0.9 to 1.2 percent by weight iron, 0.9 to 1.4 percent by weight nickel, 0.02 to 0.15 percent by weight titanium and the remainder aluminium, subjecting said slab to a plurality of cold rolling reductions each of which is not substantially less than 12% and not substantially more than 50%, a recrystallising anneal being given after each reduction except the last, subjecting the cold-rolled sheet to solution heat-treatment for not substantially less than 15 minutes and not substantially more than 50 minutes at a temperature not substantially less than 525° C. and not substantially more than 545° C., and quenching and then artificially ageing the sheet, said cold-rolling and solution heat-treatment resulting in an average intercept grain diameter in the final sheet of between 0.0399 millimetre and 0.0168 millimetre.

10. Aluminium sheet produced by the steps of forming a slab suitable for cold-rolling into a sheet of an alloy consisting essentially of 2.2 to 2.7 percent by weight copper, 1.3 to 1.7 percent by weight magnesium, 0.12 to 0.25 percent by weight silicon, 0.9 to 1.2 percent by weight iron, 0.9 to 1.4 percent by weight nickel, 0.02 to 0.15 percent by weight titanium and the remainder aluminium, subjecting said slab to a plurality of cold rolling reductions each of which is not substantially less than 12% and not substantially more than 50%, subjecting the cold-rolled sheet to solution heat-treatment for not substantially less than 15 minutes and not substantially more than 50 minutes at a temperature not substantially less

than 525° C. and not substantially more than 545° C., and quenching and then artificially ageing the sheet.

11. Clad aluminium sheet produced by the steps of forming a hot-rolled slab of an alloy consisting essentially of 2.2 to 2.7 percent by weight copper, 1.3 to 1.7 percent by weight magnesium, 0.12 to 0.25 percent by weight silicon, 0.9 to 1.2 percent by weight iron, 0.9 to 1.4 percent by weight nickel, 0.02 to 0.15 percent by weight titanium and the remainder aluminium, and having cladding of material selected from the group consisting of commercially pure aluminium, aluminium containing not substantially less than 0.8 and not substantially more than 1.2 percent by weight of zinc, and a heat-treatable corrosion-resistant aluminium base alloy consisting essentially of 0.4 to 1.4 percent by weight magnesium, 0.2 to 1.3 percent by weight silicon, 0.0 to 1.0 percent by weight manganese, 0.0 to 0.3 percent by weight chromium, about 0.8 to about 1.2 percent by weight zinc and the remainder aluminium, subjecting said slab to a plurality of cold rolling reductions each of which is not substantially less than 12% and not substantially more than 50%, subjecting the cold-rolled sheet to solution heat-treatment for not substantially less than 15 minutes and not substantially more than 50 minutes at a temperature not substantially less than 525° C. and not substantially more than 545° C., and quenching and then artificially ageing the sheet.

12. A method according to claim 1 wherein said alloy includes a minor amount of at least one other element selected from the group consisting of antimony, beryllium, cerium, chromium, cobalt, manganese, molybdenum, niobium, silver, vanadium, zirconium and zinc, the amount of each said element selected being not more than 0.3 percent by weight, the total amount of all said selected elements being not more than 1.0 percent by weight.

13. A method according to claim 1 wherein said alloy includes a minor amount of at least one other element selected from the group consisting of antimony, beryllium, cerium, chromium, cobalt, manganese, molybdenum, niobium, vanadium, zirconium and zinc, the amount of each said element selected being not more than 0.3 percent by weight, and silver in an amount greater than 0.3 percent by weight and not more than 0.5 percent by weight, the total amount by weight of all said selected elements and silver being not more than 1.0 percent plus the amount by which the silver exceeds 0.3 percent.

14. A method according to claim 1 wherein said alloy includes a minor amount of at least one other element selected from the group consisting of antimony, beryllium, cerium, chromium, cobalt, manganese, molybdenum, niobium, vanadium, zirconium and zinc, the amount of each said element selected being not more than 0.3 percent by weight, and silver in an amount greater than 0.5 percent by weight and not more than 0.8 percent by weight, the amount of copper being at least as much below 2.5 percent as the amount of silver exceeds 0.5 percent, the total amount by weight of all said selected elements and silver being not more than 1.0 percent plus the amount by which the silver exceeds 0.3 percent.

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