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THERMAL TREATMENT OF ALUMINUM BASE ALLOYS

Edgar H. Dix, Jr., Oakmont, and Joseph A. Nock, Jr., Tarentum, Pa., assignors to Aluminum Company of America, Pittsburgh, Pa., a corporation of Pennsylvania

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This invention relates to an improvement in the art of heat treating certain aluminum base alloys, and it is especially concerned with improving their resistance to corrosion insofar as this property is affected by thermal treatment.

It is an object of our invention to improve the resistance to corrosion of certain solution heat treated and artificially aged aluminum base alloys. A particular object is to provide a method of improving the resistance to corrosion of precipitation hardened aluminum base alloys containing copper, magnesium and silicon. Still another object is to provide a method of controlling the initial precipitation of dissolved constituents in aluminum base alloys quenched from the solution heat treating temperature. Certain aluminum base alloys are greatly improved in strength and hardness by subjecting them to the well known thermal treatments in the neighborhood of 500° C., rapidly cooling to ordinary temperatures, and finally causing some of the dissolved constituents to precipitate by an aging treatment. The resistance to corrosion of alloys treated in this manner is greatly affected by the character of the thermal treatment. In general it has been believed that the more drastic the cooling from the elevated temperature, the better is the resistance to corrosive attack, all other factors remaining constant. Wherever corrosion resistance is important we have strongly advocated quenching in cold water. For instance, although it would be highly desirable to quench thin sheet, for aircraft construction, more slowly as in an air blast, in order to prevent distortion, we find that this practice is not permitted by reliable manufacturers. The resistance to corrosion of heavier sections is often not so important as that of thin sheet and slower quenching is sometimes permitted to minimize distortion. Even with cold water very heavy sections are not cooled as rapidly as lighter sections. The lowered resistance to corrosion sometimes encountered on heavy sections is attributed to this fact.

The precipitation treatment also exerts a pronounced influence upon the resistance to corrosion. It has been observed in general that an alloy containing a fine uniformly dispersed precipitate possesses a better resistance to attack than the same alloy with non-uniformly distributed larger particles of coalesced precipitate. The lower resistance to corrosion of copper bearing alloys which must be reheated to temperatures in the neighborhood of 120 to 180° C. after being quenched from the solution temperatures, is frequently ascribed to the size and arrangement of precipitated constituents and inequalities in the amount of constituent remaining in solution at the grain boundaries and within the grains. On the other hand alloys that age spontaneously ordinarily have small sized uniformly dispersed particles of precipitate and a better resistance to corrosion. On this account the latter alloys are sometimes preferred where resistance to corrosion is of primary importance. However, the alloys which must be reheated or aged artificially, as the precipitation hardening treatment is called, usually develop a higher strength and hardness which is very desirable for many purposes. It is particularly desirable that such alloys should also have a high degree of resistance to corrosion.

Another form of corrosion to which certain aluminum base alloys are sometimes subjected is that known as stress corrosion. This type of corrosion differs from the ordinary type of attack in that it is influenced by the stress, and it also proceeds at a more rapid rate. It often develops certain characteristic features in the alloy structure which can be identified by microscopic examination. Solution heat treated and artificially aged alloys of the kind herein described are sometimes subjected to conditions which promote acceleration of corrosion under stress, and hence it becomes important to minimize or eliminate this type of attack as far as possible.

We have now discovered a means of improving the resistance to corrosion of artificially aged aluminum base alloys containing copper, magnesium and silicon as the principal soluble hardening constituents. The improvement is effected by controlling the temperature of the quenching medium in such a manner as to induce an initial precipitation of the dissolved constituents. The alloys so treated are then aged in the usual manner to develop the desired strength and hardness. Our method is particularly applicable to aluminum base alloys containing from 3 to 6 per cent copper, 0.5 to 2 per cent silicon, 0.1 to 1 per cent magnesium and 0.1 to 1.5 per cent manganese.

While heretofore it has been the general practice to employ as drastic a quench as possible to obtain the maximum strength and resistance to corrosion, we have observed that a better resistance to corrosion is obtained in the artificially aged temper of the aforesaid alloys if the cooling rate is somewhat retarded. In other words, although it is generally advantageous to drastically quench many heat treated aluminum base alloys, yet this general practice is not desirable in the case of alloys herein described. In particular, artificially aged aluminum base alloys containing from 3 to 6 per cent copper, 0.5 to 2 per cent silicon; 0.1 to 1 per cent magnesium and 0.1 to 1.5 per cent manganese do not attain their maximum resistance to corrosion when quenched in the manner heretofore employed.

The artificial aging treatment which follows the quenching should be such as to cause a precipitation within the grains, and thus make the precipitation more nearly uniform through the alloy. In general the aging is to be accomplished by heating to between 120 and 180° C. for 5 to 24 hours, the shorter time being employed if the temperature is in the upper portion of the permissible range.

The effect of controlling the temperature of the quenching bath in improving the resistance to corrosion of the artificially aged alloy may be explained by the fact that a relatively uniform precipitation throughout the alloy minimizes, if it does not eliminate, differences in solution potential between the interior of the grains, the boundaries, and the precipitated constituent. By way of contrast, if precipitation is largely confined to the grain boundaries, and the alloy is exposed to a corroding medium, the attack will occur at the grain boundaries, because of the aforesaid differences in potential.

The rate of cooling of an article is determined by its mass, the size of the load, being quenched, the nature of the quenching medium, its temperature, and perhaps other factors. An article of thin section such as a sheet 0.064 inch in thickness may be quenched in hot oil, or a low melting point salt bath having a temperature of 225° C., whereas an article of heavy section should be quenched in a medium at a lower temperature. As the thickness and mass of the article increase, progressively more drastic quenching media should be employed. For example, to obtain a comparable effect in a plate 0.50 inch in thickness, it should be quenched in fused salt bath at about 200° C. In the table below are given various thicknesses of sheets or plates and the fused salt bath temperatures that were used in quenching single pieces of these sheets or plates in order to give the best resistance to corrosion where the samples were made from an alloy having a nominal composition of 4.4 per cent copper, 0.8 per cent silicon, 0.75 per cent manganese, 0.35 per cent magnesium, 0.5 per cent iron and the balance aluminum.

Thickness of sheet	Temperature of fused salt
Inches	° *C.*
0.064	225
0.125	225
0.250	215
0.375	215
0.500	200
1.00	190

Because of the wide variety in size and shape of articles and the number to be treated at one time, only an approximate rule can be given as to the temperature of the quenching bath and the temperatures given above may be altered, depending on the size of the load, that is to say the number of pieces quenched at one time. The precise conditions requisite to producing the maximum resistance to corrosion in a particular case can be determined by tests. In general, less drastic quenches should be used than would have heretofore been employed for the same sized article. The article need not be left in the quenching medium longer than sufficient to attain the temperature of the bath, say about 5 minutes. It should then be removed from the bath and cooled to room temperature either in air or by immersing in water. The latter practice has been found to be especially beneficial where articles of heavy cross section are being treated. The cooling effected by immersing in water appears to arrest further change in the alloy and retain the proper amount of dissolved constituents in solution. The immersion in water also serves to dissolve any coating of salt left on the article by the quenching bath.

The effect of the quenching bath temperature upon the resistance to stressless corrosion of an aluminum base alloy containing copper, magnesium and silicon, may be seen in the following examples. For the purpose of these tests samples of sheet 0.064 inch in thickness were used. The alloy from which the sheet was made had a nominal composition of 4.4 per cent copper, 0.75 per cent manganese, 0.35 per cent magnesium, 0.8 per cent silicon, 0.5 per cent iron and the balance aluminum. All the samples were given a solution heat treatment at 498° C. for 15 minutes and then were divided into four groups for quenching in different media, one group being quenched in water at 25° C., another group in water at 100° C., another group in oil at 200° C., and the last group in oil at 225° C., all samples being cooled to room temperature in the air after removal from the quenching bath. All of the quenched samples were then aged at 171° C. for 10 hours. The samples were subjected to a 48 hour accelerated corrosion test consisting of alternately immersing in and removing them from an aqueous solution containing 5 per cent NaCl by weight and 0.3 per cent H_2O_2 by volume. The mechanical properties of the corroded and uncorroded specimens were compared, and the losses in tensile strength and elongation expressed in terms of per cent were calculated. The corrosion losses are shown in the table below.

Quenching medium		Percent loss in mechanical properties	
Kind	Temperature °C.	Tensile strength	Elongation
Water	25	−19	−66
Do	100	−18	−55
Oil	200	−9	−18
Do	225	−4	−39

From the foregoing test results it will be seen that the temperature of the quenching medium has a marked effect upon the resistance to corrosion, and that the most drastic quench does not produce the best resistance to attack as might be

ordinarily expected. The 19 per cent loss in strength and 66 per cent loss in elongation represents the result which will ordinarily be obtained following thermal treatments as previously known whereas the 4 per cent loss in strength and 39 per cent loss in elongation represents the desired improvement to be obtained by the present invention. Although we generally prefer to use a quenching medium maintained at a temperature between 190 and 225° C., yet an improvement in the corrosion resistance of the alloys becomes evident under certain conditions when the quenching medium is at a temperature as low as 100° C. as may be seen in the foregoing table.

The controlled quenching of aluminum base alloys of the kind herein described also has a marked beneficial effect upon the resistance to stress corrosion. As mentioned hereinabove, the solution heat treated and artificially aged aluminum base alloys containing from 3 to 6 per cent copper, 0.5 to 2 per cent silicon and 0.1 to 1 per cent magnesium are sometimes subject to this form of corrosive attack, and hence it is important to eliminate, if possible, even the chance of such an attack. The improvement in resistance to stress corrosion that can be effected by controlled quenching is illustrated in the following test results. For the purpose of this test sheets were used which were of different thicknesses and made from the same alloy as that mentioned in the preceding corrosion test. All of the sheets were first heat treated at about 500° C. A portion of the sheets 0.064 inch in thickness was quenched from 500° C. in water at room temperature in accordance with the usual practice of cooling the heat treated alloy as rapidly as possible. The remainder of the sheets was quenched in a fused salt bath at the temperatures indicated in the table below. The quenched samples were withdrawn from the salt bath and immediately immersed in water at room temperature. All of the samples were then aged at 160° C. for 18 hours. In the case of sheets thicker than 0.064 inch, specimens were cut from the sheets and machined to a thickness of 0.064 inch in order to provide specimens of the same size. These specimens were then subjected to an accelerated corrosion test consisting of immersing them in an aqueous solution of 5 per cent by weight of sodium chloride and 0.3 per cent by volume of hydrogen peroxide. The specimens were supported at both ends and a load producing a stress equivalent to 75 per cent of the yield strength of the alloy applied in the central portion of the specimens thus stressing them as simple beams. They were exposed to the attack for 24 to 72 hour periods as indicated in the table below. The tensile strength and elongation of the corroded specimens were then determined and compared with the properties prior to corrosion. The losses resulting from corrosion expressed in terms of per cent are given in the following table.

Table I

Original thickness of sheet in inches	Quenching medium	Temp. °C. of quenching medium	Period of test in hours	Percent losses	
				Tensile strength	Elongation
0.064	Water	20	24	−26	−78
0.064	Fused salt	225	24	−8	−45
0.064	do	225	72	−11	−63
0.125	do	225	72	−12	−40
0.250	do	225	72	−13	−54
0.375	do	200	72	−11	−58
0.500	do	200	72	−17	−62

For purposes of comparison we include also in Table II the results obtained from a companion set of samples, similar in all respects except that they were unstressed.

Table II

Original thickness of sheet in inches	Quenching medium	Temp. °C. of quenching medium	Period of test in hours	Percent losses	
				Tensile strength	Elongation
0.064	Water	20	24	−23	−72
0.064	Fused salt	225	24	−8	−34
0.064	do	225	72	−7	−32
0.125	do	225	72	−11	−55
0.250	do	225	72	−12	−49
0.375	do	200	72	−13	−67
0.500	do	200	72	−15	−64

It should be noted that owing to the expense and time involved in this method of testing, these results are obtained on individual specimens and are not averages of a number of specimens as is generally desirable in corrosion testing, hence small differences can not be considered too significant. It may also be observed not only that the specimens quenched in the fused salt bath and corroded for 72 hours sustained lower losses than the water quenched samples corroded for only 24 hours, but that in most cases no acceleration of corrosion is produced by a stress equivalent to 75 per cent of the yield strength in the sheet quenched in fused salt.

It is true that there is no acceleration of the rate of corrosion by stress shown in the case of the water quenched sheet. This however is the result of the very low resistance to corrosion of this material which causes the specimen to rapidly yield under the load until it touches the bottom of the vessel containing the corrosive solution and in this way substantially relieves the stress.

Under less severe conditions of corrosion, as for instance exposure to an industrial atmosphere, the differential between the stressed and unstressed specimens quenched in cold water becomes more evident as shown from Table III. The specimens in this case were all 0.064 inch in thickness and were exposed to the atmosphere for a period of 6 months. The stressed specimens were stressed at 75 per cent of their yield strength.

TABLE III

Quenching medium	Temp. °C. of quenching medium	Aging treatment	Unstressed samples		Stressed samples	
			Percent losses tensile strength	Elongation	Percent losses tensile strength	Elongation
Water	20	18 hrs. at 160° C.	−7	−56	−13	−80
Oil	225	do	+1	−16	+1	−13
Water	20	10 hrs. at 171° C.	−8	−59	−16	−83
Oil	225	do	+2	−11	0	−17

It may be again observed that with specimens quenched in cold water and unstressed, the effect of corrosion is quite evident, and that under the mildly corrosive effect of an industrial atmosphere, coupled with an artificially applied stress, the losses induced by corrosion have been accelerated to a marked degree. It may also be observed that with the material quenched in oil at 225° C., the losses suffered in the unstressed condition are negligible as compared with the losses suffered by the water quenched material; furthermore this same material, under an equivalent stress and subjected for the same period of time to an industrial atmosphere suffered substantially no greater losses than in the unstressed condition. This clearly indicates the improvement that can be obtained by the practice of our invention.

Any quenching medium is suitable for our purposes which will afford the desired control of the quenching rate. Allowance must of course be made for the difference in cooling capacity of the various media with corresponding adjustments in bath temperature. Where a normally drastic quenching medium is used, and it is desired to reduce the severity of the cooling, the medium may be heated. Certain aqueous solutions may also be employed where the special cooling characteristics of such solutions are beneficial. Low melting point salt baths such as mentioned hereinabove are particularly useful at temperatures of 200° C. and higher since they eliminate a danger of fire from overheating, which might be encountered if oil is used.

The aging treatment subsequent to the quenching operation should be such as to promote precipitation within the grains to the extent that differences in solution potential are equalized between the body and boundary of the grains. In general, a longer time at a low temperature is preferred to a shorter time at a higher temperature to obtain the best resistance to corrosion. However, the composition of the alloy must also be considered, since precipitation is more easily induced in some cases than in others. The range of aging temperatures that produces the desired effect varies between about 120 and 180° C. and the time of exposure usually varies between 5 and 24 hours depending upon the temperature at which the aging is done. To produce equivalent strength and hardness in two lots of the same material, a longer period of exposure is needed at a low temperature than is required if a higher temperature is used.

Not all aluminum base alloys which are susceptible to improvement in physical properties by solution heat treatment and aging are amenable to improvement in resistance to corrosion in accordance with the method described hereinabove. Our invention is limited to artificially aged aluminum base alloys containing copper, magnesium and silicon as the principal alloying constituents. These alloys may also contain minor constituents such as chromium, titanium, nickel, molybdenum, tungsten, manganese, and iron, in total amounts of not over 1.5 per cent. Generally at least 0.05 per cent of these elements must be present in order to exert a beneficial effect. An alloy that we have found to be especially susceptible to improvement by our treatment is one having a nominal composition of 4.4 per cent copper, 7.75 per cent manganese, 0.8 per cent silicon, 0.35 per cent magnesium and 0.5 per cent iron, the balance being aluminum. This alloy finds particular use in the making of heat treated and aged forgings.

Having thus described our invention and certain embodiments, thereof,

We claim:

1. In the art of heat treating aluminum base alloys, the method of producing maximum resistance to corrosion in a solution heat treated and artificially aged aluminum base alloy containing copper as the chief added alloying constituent and lesser amounts of magnesium and silicon, said method comprising quenching the alloy from the solution heat treating temperature in a medium maintained at a temperature above 100° C. to induce an initial precipitation of dissolved constituents in said alloy, and thereafter artificially aging the alloy.

2. In the art of heat treating aluminum base alloys, the method of producing maximum resistance to corrosion in a solution heat treated and artificially aged aluminum base alloy containing up to 6 per cent copper as the chief added alloying constituent and lesser amounts of magnesium and silicon, said method comprising quenching the solution heat treated alloy in a quenching medium maintained at such a temperature above 100° C., as to induce an initial precipitation of dissolved constituents, removing the alloy from the medium, cooling to room temperature, and thereafter artificially aging the alloy.

3. In the art of heat treating aluminum base alloys, the method of producing maximum resistance to corrosion in a solution heat treated and artificially aged aluminum base alloy containing up to 6 per cent copper as the chief added alloying constituent and lesser amounts of magnesium and silicon, said method comprising quenching the solution heat treated alloy in a quenching medium maintained at such a temperature above 100° C. as to induce an initial precipitation of dissolved constituents, removing the alloy from the quenching medium, immersing it in water, removing it from said water bath, and thereafter artificially aging the alloy.

4. In the art of heat treating aluminum base alloys, the method of producing maximum resistance to corrosion in a solution heat treated and artificially aged aluminum base alloy containing up to 6 per cent copper as the principal added alloying constituent and lesser amounts of magnesium and silicon, said method comprising quenching the alloy from the solution heat treating temperature in a medium maintained at a temperature above 190° C. and proportioned to the mass of the alloy being quenched in order to induce an initial precipitation of dissolved constituents, removing the alloy from the medium, cooling the alloy to room temperature and thereafter artificially aging the alloy.

5. In the art of heat treating aluminum base alloys, the method of producing maximum resistance to corrosion in a solution heat treated and artificially aged alloy containing up to 6 per cent copper and lesser amounts of magnesium and silicon, said method comprising quenching the alloy from the solution heat treating temperature in a quenching medium maintained at such a temperature between 190 and 225° C. as to induce an initial precipitation of dissolved constituents, holding the alloy in the medium until it reaches the temperature of said medium, removing the alloy therefrom, cooling to room temperature and thereafter artificially aging the alloy.

6. In the art of heat treating aluminum base alloys, the method of producing maximum resistance to corrosion in a solution heat treated and artificially aged alloy containing up to 6 per cent copper and lesser amounts of magnesium and silicon, said method comprising quenching the alloy from the solution heat treating temperature in a quenching medium maintained at such a temperature between 190 and 225° C. as to induce an initial precipitation of dissolved constituents, removing the alloy from the quenching bath, cooling to room temperature and thereafter reheating the alloy to between 120 and 180° C. for 2 to 24 hours to complete the aging.

7. In the art of heat treating aluminum base alloys, the method of producing maximum resistance to corrosion in a solution heat treated and artificially aged aluminum base alloy containing from 3 to 6 per cent copper, 0.5 to 2 per cent silicon, 0.1 to 1 per cent magnesium and 0.1 to 1.5 per cent manganese, said method comprising quenching the alloy from the solution heat treating temperature in a quenching medium maintained at such a temperature above 100° C. as to induce an initial precipitation of dissolved constituents, removing the alloy from the quenching bath, cooling to room temperature and thereafter reheating the alloy to between 120 and 180° C. for 2 to 24 hours to complete the aging.

8. In the art of heat treating aluminum base alloys, the method of producing maximum resistance to corrosion in a solution heat treated and artificially aged aluminum base alloy containing from 3 to 6 per cent copper, 0.5 to 2 per cent silicon, 0.1 to 1 per cent magnesium and 0.1 to 1.5 per cent manganese, said method comprising quenching the alloy from the solution heat treating temperature in a quenching medium maintained at such a temperature above 100° C. as to induce an initial precipitation of dissolved constituents, removing the alloy from the quenching bath, immersing it in water at room temperature, until it reaches the temperature of the water, removing it from said water, and thereafter reheating the alloy to between 120 and 180° C. from 2 to 24 hours to complete the aging.

9. In the art of heat treating aluminum base alloys, the method of producing maximum resistance to corrosion in a solution heat treated and artificially aged aluminum base alloy containing from 3 to 6 per cent copper, 0.5 to 2 per cent silicon, 0.1 to 1 per cent magnesium, and from 0.05 to 1.5 per cent of at least one of the group of metals composed of chromium, manganese, titanium, nickel, molybdenum, tungsten, vanadium, zirconium, and iron, said method comprising quenching the alloy from the solution heat treating temperature in a quenching medium maintained at such a temperature above 100° C. as to induce an initial precipitation of dissolved constituents, removing the alloy from the quenching bath, cooling to room temperature and thereafter reheating the alloy to between 120 and 180° C. for 2 to 24 hours to complete the aging.

10. In the art of heat treating aluminum base alloys, the method of producing maximum resistance to corrosion in a solution heat treated and artificially aged wrought aluminum base alloy consisting of about 4.4 per cent copper, 0.75 per cent manganese, 0.35 per cent magnesium, 0.8 per cent silicon, 0.5 per cent iron, and the balance aluminum, said method comprising quenching the alloy from the solution heat treating temperature in a quenching medium maintained at such a temperature above 100° C. as to induce an initial precipitation of dissolved constituents, removing the alloy from the quenching bath, cooling to room temperature and thereafter reheating the alloy to between 120 and 180° C., to complete the aging.

11. In the art of heat treating aluminum base alloys, the method of producing maximum resistance to corrosion in a solution heat treated and artificially aged aluminum base alloy containing from 3 to 6 per cent copper, 0.5 to 2 per cent silicon, 0.1 to 1 per cent mangesium, and 0.1 to 1.5 per cent manganese, said method comprising quenching the alloy from the solution heat treating temperature in a quenching medium maintained at a temperature between about 190 and 225° C., holding said alloy in the quenching medium for a time sufficient to induce an initial precipitation of dissolved constituents, removing the alloy from the quenching bath, cooling it to room temperature, and thereafter reheating the alloy to between 120 and 180° C. for 2 to 24 hours to complete the aging.

EDGAR H. DIX, JR.
JOSEPH A. NOCK, JR.

CERTIFICATE OF CORRECTION.

Patent No. 2,159,010. May 23, 1939.

EDGAR H. DIX, JR., ET AL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 4, first column, line 73, for the numeral "7.75" read 0.75; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 11th day of July, A. D. 1939.

Henry Van Arsdale

(Seal)

Acting Commissioner of Patents.

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