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# HEAT TREATMENT OF ALUMINUM ALLOYS

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The present invention relates to a novel artificial age hardening treatment for age hardenable aluminum alloys which undergo spontaneous age hardening at room temperatures to the detriment of subsequently performed artificial aging treatments. More particularly the invention relates to the treatment of aluminum alloys whose principal alloying elements are magnesium and silicon in approximately the stoichiometric proportion of $Mg_2Si$ and wherein the proportion of $Mg_2Si$ is between 0.5% and 3.0% and containing no more than about 0.3% of copper and preferably not more than 0.1% of copper. These Al-Mg-Si alloys may also contain silicon in excess of the stoichiometric proportion $Mg_2Si$, manganese in amounts up to 1.5% and not more than a total of 1.0% of other alloying elements such as for example, copper less than 0.3% preferably less than 0.1%, Zn less than 0.5%, Cr less than 0.5% and Fe less than 0.5%. Alloys of the following composition are especially suitable for treatment in accordance with the invention; Mg 0.3% to 1.5% preferably 0.7% to 1.0%, Si 0.2% to 1.5% preferably 0.7% to 1.2%, Mn less than 1.5% preferably between 0.6% to 1.0% Cu less than 0.3% preferably less than 0.1%, Zn less than 0.3%, Cr less than 0.3%, iron less than 0.5% and the remainder substantially aluminum.

Alloys of this type are normally subjected to a solution heat treatment at 520° to 570° C. and are then quenched and subsequently aged either at room temperature or artificially aged at temperatures between 120° and 200° C. preferably 140° to 160° C. to improve their mechanical properties. When such alloys are aged at room temperatures the following average mechanical properties are obtained in the alloys: yield strength (0.2% offset) 10 to 15 kg./mm.$^2$, tensile strength 20 to 26 kg./mm.$^2$, elongation (Delta 10) 20% to 12% and a Brinell hardness of 50 to 60 kg./mm.$^2$. When said alloys are artificially aged at elevated temperatures the following average values are obtained for their mechanical properties: yield strength 18 to 28 kg./mm.$^2$, tensile strength 28 to 32 kg./mm.$^2$, elongation 14% to 10% and a Brinell hardness of 70 to 95 kg./mm.$^2$ (measured with 2.5 mm. ball with a load of 32.5 kg. for thirty seconds).

It has been known for some time that when Al-Mg-Si alloys which are poor in copper are permitted to age at room temperatures for substantial periods of time before artificial aging at elevated temperatures, the mechanical properties achieved are not as good as when the artificial aging is commenced immediately after the alloys have been quenched. This property of the Al-Mg-Si alloys has lead to problems in their heat treatment, for it is not always desirable or practical to subject the products to an artificial aging immediately after quenching. For example, if test samples of an alloy composed of 0.02% Cu; 0.79% Mg; 0.79% Mn; 0.93% Si; 0.49% Fe and its remainder Al are subjected to a solution heat treatment at 550° C. for about 1 hour and quenched in water at 20° C. and a portion of such test samples is immediately subjected to artificial aging in an air oven at 150° to 155° C. for eight hours and the other portion is permitted to remain at room temperature for an hour before being subjected to the same artificial aging treatment the following properties are obtained in the samples:

| | Yield Strength, kg./mm.$^2$ | Tensile Strength, kg./mm.$^2$ | Elongation, percent |
|---|---|---|---|
| Artificially aged immediately after quenching | 34.25 | 37.2 | 10 |
| Artificially aged 1 hour after quenching | 26.4 | 33.5 | 15.8 |

Room temperature storage before artificial aging has the same detrimental effect upon all alloys of the



Al-Mg-Si type as long as they do not contain substantial quantities of copper. The amount of copper suppressing the detrimental effect of room temperature storage varies but is to a certain degree proportional to the amount of $Mg_2Si$ present.

It is an object of this invention to provide a novel process which will permit the artificial aging of Al-Mg-Si alloys of the above type to produce products of uniformly improved mechanical properties.

It is a further object to provide a novel process which will increase the corrosion resistance to artificially aged Al-Mg-Si alloys.

Still another object of the invention is to provide a process for the heat treatment of Al-Mg-Si alloys which will permit subsequent artificial age hardening to produce excellent mechanical properties even though such alloys are stored at room temperature between the time they are subjected to solution heat treatment and the artificial aging treatment.

It was found Al-Mg-Si alloys even when quenched from a solution heat treatment in water at 20° C. and, under laboratory conditions, immediately subjected to artificial aging at elevated temperatures do not achieve as favorable mechanical properties as those achieved by the process in accordance with the invention. In operations upon a commercial scale it is not practically possible to subject the alloys to artificial aging at elevated temperatures as quickly as is possible in operations on a laboratory scale and usually at least above five minutes intervene between the quenching and the artificial aging.

In accordance with the invention it has been discovered that substantial advantages are obtained when Al-Mg-Si alloys are quenched from a solution heat treatment in a quenching medium which is maintained at the temperature required for the artificial aging of the alloys namely between 120° C. and 200° C. preferably between 140° C. and 180° C. and are maintained at such temperature until the desired properties of the alloy are produced. The time for which the alloy is maintained depends upon the properties which are sought in the alloy. The artificial aging may be completed without permitting the article treated to cool down to room temperature but in some instances it has been found desirable to interrupt the artificial aging treatment to permit other operations to be performed before the final artificial aging takes place. In the latter instance it has been found preferable to continue the preliminary artificial aging until the article possesses a hardness which does not increase during the period the article is held at room temperature between the preliminary artificial aging treatment and the final artificial aging treatment.

The following example illustrates the advantages of the process in accordance with the invention. A series of test specimens of an alloy composed of 0.02% Cu, 0.79% Mg, 0.79% Mn, 0.93% Si, 0.49% Fe and the remainder Al were subjected to a solution heat treatment at 550°± 5° C. for one hour. One series of the specimens was then quenched in water at room temperature and immediately subjected to artificial aging in an air oven at 150° C. one portion for eight hours and the other for fifteen hours. The other series of specimens was subjected to the same solution heat treatment but was quenched in oil at a temperature of 150° C. and placed in the same air ovens for aging as soon as they reached equilibrium temperatures, one portion also was aged for eight hours and another for 15 hours. The following chart indicates the differences in the mechanical properties obtained in the two series of tests. In the chart the designation NW indicates the series quenched in water and the designation IW indicates the series quenched in oil.

| Length of Artificial Aging | Yield Strength, 0.2%, kg./mm.$^2$ | | Tensile Strength, kg./mm.$^2$ | | Elongation, 1=50 mm., percent | |
|---|---|---|---|---|---|---|
| | NW | IW | NW | IW | NW | IW |
| 8 hours | 34.25 | 35.7 | 37.25 | 38.3 | 10 | |
| 15 hours | 35.6 | 38.1 | 39.0 | 40.0 | 13.6 | 1 |

These values show the improvement obtained by the process in accordance with the invention when compared with normally used processes carried out under the most favorable conditions, which conditions, however, cannot be practically achieved in commercial scale operations.

A further series of tests were carried out with eight specimens of the same alloy which had been subjected to a solution heat treatment. In each series of the tests one specimen was quenched in water at room temperature and after an intervening period of exactly ten minutes placed in an air oven and heated to 150° C. and artificially aged therein for fifteen hours and the other specimen was quenched in oil at 150° C. and immediately placed in the air oven and aged for fifteen hours. Specimens 1, 3, 5 and 7 were those quenched in water and specimens 2, 4, 6 and 8 were those quenched in oil.

| Specimens | Yield Strength, 0.2% kg./mm.² | Tensile Strength, kg./mm.² | Elongation, 1=50 mm., percent |
|---|---|---|---|
| | 34.2 | 38.1 | 14 |
| | 36.9 | 39.5 | 10 |
| | 35.9 | 39.0 | 12.4 |
| | 36.9 | 39.8 | 10.4 |
| | 35.4 | 38.4 | 13.6 |
| | 37.1 | 39.6 | 11 |
| | 36.0 | 39.2 | 13.8 |
| | 37.4 | 39.9 | 12 |

It will be seen from a comparison of the latter chart with the former chart that the values obtained when there is a 10 minute delay between the quenching in water and the artificial aging are only slightly different from the values obtained when the specimen is immediately subjected to age hardening after quenching in water. It may, however, be clearly seen that the process in accordance with the invention produces a recognizable improvement in the properties of the alloy treated. The average values of the properties obtained for the tensile strength and yield strength with the process in accordance with the invention are not only considerably better but the variations are in a considerably narrower range.

It is furthermore interesting that the progress of the hardening obtained with artificial aging in accordance with the present invention substantially parallel that obtained when the alloys are quenched to room temperature and immediately artificially aged so that for given periods of artificial aging substantially the same hardnesses are obtained.

Corrosion tests have also shown that the corrosion resistance of alloys artificially aged in accordance with the invention is considerably greater than when prior art processes are employed. For example, corrosion tests were made with test specimens of an Al-Mg-Si alloy of the type specified, one series being quenched in water at room temperature and immediately artificially aged at 150° C. for various periods and the other series being quenched in oil at 150° C. and artificially aged by being maintained at such temperature for varying periods of time.

The aging periods for each series were selected as four hours, eight hours, sixteen hours and seventy two hours.

The corrosion test consisted of immersing suitably prepared specimens in an aqueous half normal NaCl solution containing ½% of concentrated HCl and measuring the amount of hydrogen evolved. After three hours' treatment the following volumes of hydrogen were evolved:

| Duration of Artificial Aging | 4 hrs. | 8 hrs. | 16 hrs. | 72 hrs. |
|---|---|---|---|---|
| Specimens Oil quenched | 13½ | 13 | 12 | 12 |
| Specimens water quenched | 25½ | 21 | 17 | 14 |



While the above examples refer to treatments in which the total desired artificial aging is obtained in one uninterrupted aging treatment in conjunction with the quenching from a solution heat treatment, the process in accordance with the invention may be modified to permit final artificial aging to the desired properties some time after the quenching when for practical reasons, such as, lack of aging oven capacity in the plant where the alloys are subjected to solution heat treatment and quenching or where it is desired to subject the alloys to treatments which are not practical before the solution heat treatment nor after the artificial aging treatment.

It has been found in accordance with the invention that the detrimental effects of room temperature storage of Al-Mg-Si alloys of the type described between quenching and artificial aging may be substantially avoided if such alloys are quenched only to the artificial aging temperature, that is between 120° and 200° C. and are maintained at such temperature until the artificial aging effected thereby provides a hardness which is greater than that which would be obtained directly after quenching such alloy in water at room temperature and the final aging takes place before its hardness increases because of room temperature storage. Preferably the preliminary artificial aging treatment in accordance with the invention is of such duration that the hardness of the alloy obtained does not substantially exceed that which would ultimately be obtained if the alloy were quenched to room temperature and aged at room temperature. The preliminary aging is carried out to such ultimate hardness in such cases where the preliminarily aged alloy is to be furnished to others for fabrication and final artificial aging as the period of room temperature storage in such instances is uncertain.

While the preliminary artificial aging treatment should be long enough that the hardness of the alloy does not substantially increase during room temperature storage before it is given the final artificial aging treatment in order to secure the optimum mechanical values it has been discovered that substantially improved mechanical values may be obtained in accordance with the invention even though the preliminary aging treatment is insufficient to maintain a constant hardness for the alloy during its entire storage at room temperatures as long as the hardness of such preliminarily artificially aged alloy is still below that which would be obtained in the alloy if it were stored at room temperature for an equivalent period of time after quenching at room temperature.

For example, a test sample of an Al-Mg-Si of the type described composed of 0.02% Cu, 0.66% Mg, 0.72% Mn, 0.93% Si, 0.22% Fe and the remainder Al were solution heat treated at 550° C. for 1¼ hours. Five of such samples were quenched in water at room temperatures and four other test samples were quenched in oil at 150° C., one of such samples being held in such quenching oil for 1 minute and the three others for 10 minutes, 15 minutes, and 20 minutes respectively.

One of the test samples which was quenched in water originally had a Brinell hardness of about 54 kg./mm.² which increased to 58 kg./mm.² after 1 hour room temperature storage, to 70 kg./mm.² after 15½ hours' storage, to 74 kg./mm.² after 41 hours' storage and to 78 kg./mm.² after 84 hours' storage.

The test sample which was quenched in oil and preliminarily aged for 1 minute had an original hardness of 41 kg./mm.² which increased to 53 kg./mm.² after 1 hour storage at room temperature.

The test sample which was preliminarily aged for 10 minutes had an original hardness of 58 kg./mm.² which did not increase until after about 9 hours' storage at room temperature and increased to about 61 kg./mm.² after 15½ hours' storage at room temperature.

The test sample which was preliminarily aged for 15 minutes had an original hardness of about 65 kg./mm.² which did not increase until after about 40 hours' storage at room temperatures and had a hardness of about 65 kg./mm.² after 41 hours' storage at room temperature.

The test sample which was preliminarily aged for 20 minutes had an original hardness of about 70 kg./mm.² which did not increase during storage at room temperatures for 84 hours.

The four other test samples which were quenched in water were artificially aged at various periods of time after the quenching and the hardness obtained compared with the test samples which had been preliminarily aged

in accordance with the invention. The artificial aging treatment was for 15 hours at 152° C. The following chart shows the Brinell hardness obtained.

| Treatment | Storage at Room Temperature in Hours | | | | |
|---|---|---|---|---|---|
| | 0 | 1 | 15½ | 41 | 84 |
| Quenched in water 20° C. | 114.5 | 95.5 | 89.0 | ------ | 88.0 |
| Quenched in oil 150° C. for 1 minute. | ------ | 100 | ------ | ------ | ------ |
| Quenched in oil 150° C. for 10 minutes. | ------ | ------ | 110 | ------ | ------ |
| Quenched in oil 150° C. for 15 minutes. | ------ | ------ | ------ | 116 | ------ |
| Quenched in oil 150° C. for 20 minutes. | ------ | ------ | ------ | ------ | 117 |

This chart clearly shows the improvements obtained in accordance with the invention when solution heat treated alloys must be stored at room temperatures before the artificial aging treatment. Even those samples which were subjected to artificial aging after a period of storage at room temperatures sufficiently long that the hardness of the sample increased during such storage show a considerably greater hardness than those which were stored after quenching to room temperature.

The test sample shown in the above chart which was quenched in water and immediately artificially aged possesses a tensile strength of 36.2 kg./mm.² and a yield strength of 31.0 kg./mm.² whereas the sample which was quenched in water and stored at room temperature for one hour before artificial aging only had a tensile strength of 33.3 kg./mm.² and a yield strength of 22.0 kg./mm.² and the water quenched sample which was stored for 84 hours before artificial aging had a tensile strength of 32.6 kg./mm.² and a yield strength of 20.0 kg./mm.². In comparison the test sample which had been preliminarily aged for 20 minutes in accordance with the invention and which received its final artificial aging 84 hours after such preliminary aging had a tensile strength of 38.6 kg./mm.² and a yield strength of 31.0 kg./mm.² In other words the tensile strength obtained was 107% of that obtained in the water quenched sample which was artificially aged immediately after quenching and the yield strength equal to that obtained in the sample which was aged artificially immediately after quenching. The decided advantage of the process in accordance with the invention is clearly apparent for mechanical values may be obtained in alloys which are artificially aged long after the solution heat treatment and quenching operations which are identical with or superior to those obtained when the alloys are quenched in water and immediately artificially aged under laboratory conditions which are almost impossible to fulfill in operations upon a commercial scale.

The length of time required on preliminary artificial aging treatment in accordance with the invention depends upon the time the preliminarily aged alloy is to be stored at room temperature before the final artificial aging and also upon the temperature used for the preliminary aging, the higher the temperature the less time will be required.

The advantages of the preliminary artificial aging treatment are also apparent from the following tests which were carried out with specimens of an alloy compost of 0.79% Mg, 0.79% Mn, 0.93% Si, 0.49% Fe and 0.02% Cu. The specimens were all given a solution heat treatment for one and one-half hours at 550° C. Specimens of such alloy which were quenched to room temperature were found to possess a Brinell hardness of 45 kg./mm.² which increased to about 67 kg./mm.² after storage at room temperature for 21 days. Specimens that were quenched in oil at 180° C. and maintained in such oil for five minutes had an initial Brinell hardness of about 57 kg./mm.² which increased to about 67 kg./mm.² after 21 days' storage. Specimens which were quenched in oil at 180° C. and maintained at such temperature for 10 minutes had an initial Brinell hardness of about 67 kg./mm.² which did not substantially increase during 21 days' storage. The following table gives a comparison of the tensile strengths and yield strengths obtained in the test specimens which were treated in various ways after the solution heat treatment.



| Specimen Number | Treatment after solution heat treatment | Treatment after 21 days | Tensile Strength, kg./mm.² | Yield Strength, 0.2% Offset, kg./mm.² |
|---|---|---|---|---|
| 22 | $H_2O$ at 20° C. | ------ | 31.7 | 25.4 |
| 24 | ------do------ | 2 hrs. at 180° C. | 32.9 | 26.1 |
| 34 | Oil at 180° C., 5 minutes. | ------ | 28.2 | 17.9 |
| 31 | ------do------ | 2 hrs. at 180° C. | 33.4 | 28.0 |
| 29 | Oil at 180° C., 10 minutes. | ------ | 28.9 | 21.6 |
| 25 | ------do------ | 2 hrs. at 180° C. | 34.6 | 33.6 |
| 32 | Oil at 180° C., 2 hours. | ------ | 35.9 | 34.2 |

As may be seen from this table, preliminary treatment for 5 minutes at 180° C. is insufficient to produce the optimum results, for the yield strength obtained after the subsequent artificial aging is substantially below those obtainable by artificial aging immediately after quenching in oil at 180° C. The preliminary treatment for 10 minutes at 180° C. on the other hand renders it possible to obtain both tensile strengths and yield strengths approaching those obtained with an artificial aging immediately after quenching at 180° C.

The preliminary aging treatment in accordance with the invention not only has the advantage that the preliminarily aged alloys may be stored at room temperatures between a solution heat treatment and the artificial aging thereof without substantially affecting the mechanical values obtainable by artificial aging but it also provides other advantages in the processing of Al-Mg-Si alloys.

Such preliminary aging treatment may, for example, be used for articles which are to be anodized and coated with an artificial resin containing lacquer which must be burned in. In this instance the anodizing and coating with the lacquer may be carried out after the preliminary aging treatment and the articles may then be subjected to the final artificial aging treatment at a temperature and period suitable for burning in the lacquer. As a consequence, it is not only possible to reduce the heat requirements for producing the finished product and also to avoid possible reduction in the mechanical properties which may occur when a lacquer coating is burned in on a fully artificially aged alloy.

Also, the preliminary aging treatment in accordance with the invention provides advantages when the article is to be formed by one or two draws after the solution heat treatment. The yield strength of the alloy after preliminary aging treatment is substantially less than that of the alloy which is aged at room temperatures and consequently less power is required for the forming operations. If two drawing operations are to succeed the solution heat treatment it has been found desirable to combine the first draw with the preliminary aging treatment. This may be easily done by taking the article to be drawn directly from the solution heat treatment and drawing it with a punch and die which are cooled to the extent that the article is quenched to the artificial aging temperature of, for example, 150° C. while it is drawn and transferring it to an oven to complete the preliminary aging treatment and then after such preliminary aging treatment it may be subjected to the second drawing operation before it is subjected to the final artificial aging treatment. Obviously it is also possible to combine a drawing operation with the process in accordance with the invention wherein the article is quenched to the artificial aging temperature and is maintained at such temperature for the full desired artificial aging treatment.

The preliminary aging treatment in accordance with the invention is especially well suited for the production of semifinished products for persons who do not have the necessary equipment to carry out a solution heat treatment. For example, Al-Mg-Si alloys containing Mg and Si in the quantities specified which are prepared from aluminum of the highest purity and contain other elements only as traces are admirably suited for dental work because of their corrosion resistance and lightness. However, in fully artificially age hardened condition they are not easily formed and therefore to obtain the most desirable strengths the dentist would have to subject his products to a solution heat treatment at his office. This requires more complicated apparatus than a dentist nor-

mally has at his disposal and consequently his use of such alloys is comparatively limited. Alloys preliminarily aged in accordance with the invention may, on the other hand, easily be used by the dentist for the necessary final artificial aging may easily be carried out with simple equipment such as, for example, an oil bath heated to 150° to 180° C.

The process in accordance with the invention wherein the Al-Mg-Si alloy is quenched to artificial age hardening temperatures and is maintained at such temperatures is also especially adapted for the treatment of Al-Mg-Si rivets. If the rivets are maintained at artificial aging temperatures for only a very short period of time of the order of 1 minute it is possible to produce rivets which have a lower hardness than rivets which are quenched in water at room temperature and which will have a lower hardness than the water quenched rivets for some time. This renders it possible to drive such rivets using less power than for water quenched rivets during such time. Nevertheless, such driven rivets will age at room temperatures and ultimately will have the same strength and hardness as water quenched rivets.

If the rivets are preliminarily artificially aged at the quenching temperature until they possess a hardness over that of rivets immediately after quenching in water such preliminarily aged rivets will possess a lower hardness than the water quenched rivets after a certain period of time. For example, in some instances rivets preliminarily aged to a hardness equal to that of water quenched rivets which have been stored at room temperature for five hours will maintain the same hardness for about 40 hours' room temperature storage and consequently will have a much lower hardness than the water quenched rivets during the period of storage between 5 and 40 hours after the quenching. Such rivets may be driven and be permitted to age naturally at room temperatures to the same hardness and strength as the water quenched rivets. If the parts which are riveted also have been subjected to a similar preliminary artificial aging treatment, the riveted article may be subjected to a final artificial aging treatment which, if it is carried out before the rivets and the parts riveted begin to increase in hardness, will produce an optimum hardness and strength in the riveted article.

While the process in accordance with the invention has been described with particular reference to Al-Mg-Si alloys containing only relatively minor amounts of Cu and preferably less than 0.1%, improved results may also be obtained with other high strength artificially ageable aluminum alloys which age harden at room temperatures to the detriment of the properties obtainable with subsequent artificial age hardening. The process in accordance with the invention is, however, especially applicable to Al-Mg-Si alloys which contain quantities of Cu to which are insufficient to suppress the detrimental effects of aging at room temperature. While the process in accordance with the invention produces beneficial results in Al-Mg-Si alloys containing up to 0.3% Cu, such as for example 61S, such alloys as contain less than 0.1% Cu, such as A51S and 53S, are especially adapted for the process.

I claim:

1. A process for the artificial aging of an Al-Mg-Si alloy which spontaneously ages at room temperature after quenching to room temperature from a solution heat treatment to the detriment of values obtainable upon subsequent artificial aging which comprises quenching said alloy from a solution heat treatment to an artificial aging temperature between 120° C. and 200° C., maintaining such alloy at such temperature until it has reached a hardness which is greater than that which would be obtained after immediately quenching said alloy in water to room temperature, storing such preliminarily aged alloy at room temperature and subjecting it to an artificial aging treatment at elevated temperatures before the storage at room temperatures causes an increase in the hardness of the alloy.

2. A process for the artificial aging of an Al-Mg-Si alloy containing 0.7%–1.0% of Mg, 0.7%–1.2% of Si, and 0.6%–1.0% of Mn, and containing less than 0.1% of Cu which comprises quenching said alloy from a solution heat treatment to an artificial aging temperature between 140° C. and 180° C., maintaining such alloy at such temperature until it has reached a hardness which is greater than that which would be obtained after immediately quenching said alloy in water to room temperature, storing such preliminarily aged alloy at room temperature and subjecting it to an artificial aging treatment at elevated temperatures before the storage at room temperatures causes an increase in the hardness of the alloy.

3. A process in accordance with claim 1, in which said Al-Mg-Si alloy contains less than 0.1% of Cu.

4. A process in accordance with claim 1, in which said preliminary artificial aging treatment is discontinued before the hardness produced thereby in the alloy is greater than the hardness which would be obtained by quenching said alloy to room temperature from a solution heat treatment and aging such alloy at room temperature.

**References Cited** in the file of this patent

UNITED STATES PATENTS

| Number | Name | Date |
|---|---|---|
| 1,472,739 | Archer et al. | Oct. 23, 1923 |
| 2,239,744 | Smith et al. | Apr. 29, 1941 |