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(54) METHOD OF MANUFACTURING AN ALUMINIUM ALLOY PLATE FOR VACUUM CHAMBER ELEMENTS

(57) The invention relates to a method of manufacturing an aluminium alloy plate for vacuum chamber elements, the method comprising the steps of: (a) providing a rolling feedstock material of an Al-Mg-Si aluminium alloy having a composition comprising of, in wt.%, Mg 0.80%-1.05%, Si 0.70%-1.0%, Mn 0.70%-0.90%, Fe up to 0.20%, Zn up to 0.08%, Cu up to 0.05%, Cr up to 0.03%, Ti up to 0.06%, unavoidable impurities and balance aluminium; (b)homogenizing of the rolling feedstock at a temperature in a range of 550-595°C; (c) hot-rolling of the homogenized rolling feedstock in one or more rolling steps to a hot-rolled plate having a thickness of at least 10 mm; (d) solution heat-treatment (SHT") of the hot rolled plate at a temperature in a range of 540-590°C; (e) rapid cooling the SHT plate; (f) stretching of the cooled SHT plate to obtain a permanent elongation from 1-5%; (g) artificial ageing of the stretched plate.

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Description

FIELD OF THE INVENTION

[0001] The invention relates to a method of manufacturing an aluminium alloy plate of an Al-Mg-Si alloy (also known as a 6XXX-series aluminium alloy) for forming elements of the vacuum chambers of apparatuses for manufacturing semiconductor devices and liquid crystal devices, such as CVD systems, PVD systems, ion-implanting systems, sputtering systems and dry etching systems, and those placed in the vacuum chambers. The invention relates also to a method of manufacturing vacuum chamber elements from the Al-Mg-Si alloy plate.

BACKGROUND TO THE INVENTION

[0002] Reactive gases, etching gases, and corrosive gases containing halogen as a cleaning gas are supplied into the vacuum chambers of apparatuses for manufacturing semiconductor devices and liquid crystal devices, such as CVD systems, PVD systems, ion-implanting systems, sputtering systems and dry etching systems. Therefore, the vacuum chambers are required to have corrosion resistance to corrosive gases (hereinafter, referred to as "corrosive gas resistance"). Since a halogen plasma is often produced in the vacuum chamber, resistance to plasmas (hereinafter, referred to as "plasma resistance") is also important. Recently, aluminium and aluminium alloy materials have been used for forming elements of the vacuum chamber because aluminium and aluminium alloy materials are light and excellent in thermal conductivity. Since aluminium and aluminium alloy materials are not satisfactory in corrosive gas resistance and plasma resistance, various surface quality improving techniques for improving those properties have been proposed. However, many of those properties are still unsatisfactory and further improvement of those properties is desired. Coating an aluminium or an aluminium alloy material with a hard anodic oxide film having a high hardness has been found to be effective in improving plasma resistance. The hard anodic oxide film is resistant to the abrasion of a member by a plasma having high physical energy and hence is capable of improving plasma resistance. The vacuum chamber elements require also sufficiently high mechanical strength and elongation, and colour uniformity and a high breakdown voltage after anodisation.

[0003] US patent document US-2012/0325381-A1 discloses a manufacturing process for a block of aluminium at least 250 mm thick designed for manufacture of an element for a vacuum chamber, the method comprises casting a block of a given 6XXX-series aluminium alloy, optionally homogenizing said cast block, performing a solution heat treatment directly on the cast and optionally homogenized block, quenching the block, stress relieving of the quenched block by means of cold compression, followed by artificial ageing to a T652 condition. A key element of the disclosed process is that prior to the solution heat treatment the block has not been hot or cold worked to reduce its thickness. The resultant plate product is a so-called "cast plate". A disadvantage of cast plate is that the unavoidable phases resulting from the combination and precipitation at grain boundaries of elements like iron, manganese, magnesium, and silicon, often in an eutectic form after solidification, cannot be fully dissolved in the subsequent processing steps like homogenization and solution heat treatment and remain as sites for crack initiation, thus significantly lowering the mechanical properties (e.g., ultimate tensile strength, elongation, toughness, and others), or as initiators of local corrosion (e.g. pitting corrosion) and are harmful also for final treatments like anodization which is of particular relevance for vacuum chamber elements. Any oxide layer present within the cast alloy will also remain in its original shape therefore also lowering the mechanical properties. Although cast plate products might be produced more cost effective, because substantially the as-cast microstructure is maintained, and strongly depends on the local cooling speed during the casting operation, there is much more variation in mechanical properties as function of the testing location as compared to rolled plate products, rendering cast plates less suitable for many critical applications.

DESCRIPTION OF THE INVENTION

[0004] As will be appreciated herein below, except as otherwise indicated, aluminium alloy designations and temper designations refer to the Aluminium Association designations in Aluminium Standards and Data and the Registration Records, as published by the Aluminium Association in 2019 and are well known to the person skilled in the art. The temper designations are laid down also in European standard EN515.

[0005] For any description of alloy compositions or preferred alloy compositions, all references to percentages are by weight percent unless otherwise indicated.

[0006] The term "up to" and "up to about", as employed herein, explicitly includes, but is not limited to, the possibility of zero weight-percent of the particular alloying component to which it refers. For example, up to 0.08% Zn may include an aluminium alloy having no Zn.

[0007] It is an object of the invention to provide a method of manufacturing an aluminium alloy plate of an Al-Mg-Si aluminium alloy or 6XXX-series aluminium alloy for forming vacuum chamber elements. It is another object of the invention to provide a method of manufacturing vacuum chamber elements from an Al-Mg-Si aluminium alloy plate.

[0008] These and other objects and further advantages are met or exceeded by the present invention and providing a method of manufacturing an aluminium alloy plate for vacuum chamber elements, the method comprising the steps of, in this order:

(a) providing a rolling feedstock material of an Al-Mg-Si aluminium alloy having a composition comprising of, in wt.%,

Mg	0.80% to 1.05%;
Si	0.70% to 1.0%;
Mn	0.70% to 0.90%;
Fe	up to 0.20%;
Zn	up to 0.08%, preferably up to 0.05%;
Cu	up to 0.05%, preferably up to 0.03%;
Cr	up to 0.03%, preferably up to 0.02%;
Ti	up to 0.06%, preferably 0.01% to 0.06%;

unavoidable impurities each <0.03%, total <0.10%, balance aluminium;

(b) homogenizing of the rolling feedstock at a temperature in a range of 550°C to 595°C;

(c) hot-rolling of the homogenized rolling feedstock in one or more rolling steps to a hot-rolled plate having a thickness of at least 10 mm;

(d) solution heat-treatment (SHT") of the hot rolled plate at a temperature in a range of 540°C to 590°C;

(e) rapid cooling or quenching of the SHT plate, preferably by one of spray quenching or immersion quenching in water or other quenching media;

(f) stretching of the cooled SHT plate to obtain a permanent elongation from 1% to 5%;

(g) artificial ageing of the stretched plate, preferably to a T6 condition or T7 condition.

[0009] By the careful control of narrow compositional ranges of the Al-Mg-Si alloy in combination with the thermo-mechanical processing the resultant aluminium alloy plate is ideally suitable for manufacturing vacuum chamber elements. It is available in a wide range of thicknesses and is very good anodisable with a hard anodic coating. The aluminium plate material has high mechanical properties providing good shape stability of the vacuum chamber element. Several properties of an anodised element depend on the plate material's microstructure and composition. The plate product has a microstructure having a uniform distribution of phases within the plate leading to a less affected anodic layer concerning e.g. plate thickness and uniformity at the surface after anodisation. The resultant plate product according to this invention provides a high corrosive gas resistance, e.g. as tested in a bubble test using 5% HCl; and has a high breakdown voltage (AC, DC) measured according to ISO-2376(2010).

[0010] In an embodiment the Al-Mg-Si alloy plate at thickness 55 mm in T651 condition has a tensile yield strength (YS) of at least 250 MPa, and even of at least 265 MPa, in the LT-direction in accordance with the applicable norm ISO 6892-1 B.

[0011] In an embodiment the Al-Mg-Si alloy plate at thickness 55 mm in T651 condition has a tensile strength (UTS) of at least 300 MPa, and even of at least 310 MPa, in the LT-direction in accordance with the applicable norm ISO 6892-1 B.

[0012] In an embodiment the Al-Mg-Si alloy plate at thickness 55 mm in T651 condition has an elongation (A_{50mm}) at least 8%, and even of at least 10%, in the LT-direction in accordance with the applicable norm ISO 6892-1 B.

[0013] Mg in combination with Si are the main alloying elements in the aluminium alloy to provide strength by the formation of Mg_2Si phases. The Mg should be in a range of 0.80% to 1.05%, and preferably in a range of 0.85% to 1.05%. A preferred upper-limit for the Mg content is 1.0%. A too high Mg content may lead to lead to the formation of coarse Mg_2Si phases having an adverse effect of the quality of a subsequently applied anodisation coating. A too low Mg content has an adverse effect on the tensile properties of the aluminium plate.

[0014] The Si should be in a range of 0.70% to 1.0%. In an embodiment the Si content is at least 0.75%, preferably at least 0.80%, and most preferably at least 0.84%. In an embodiment the upper-limit for the Si-content is 0.95%.

[0015] In an embodiment the ratio of Mg/Si, in wt.% is more than 0.9, and preferably more than 1.0, and most preferably more than 1.05. Reducing the amount of free Si in the aluminium alloy favours an increased elongation in the aluminium plate after SHT at relative high temperatures as done in accordance with the invention.

[0016] Another important alloying element is Mn and should be in a range of 0.70% to 0.90% to increase the strength in the aluminium plate and to control the grain structure and leads recrystallisation after solution heat treatment and quenching. A preferred lower limit is 0.75%. A preferred upper-limit is 0.85%.

[0017] Fe is an impurity element which should not exceed 0.20%. To control grain size and to achieve high mechanical strength and good corrosion resistance after anodisation the Fe level is preferably up to 0.12%. However, it is preferred that at least 0.03% is present, and more preferably at least 0.04%. A too low Fe content may lead to undesirable recrystallized grain coarsening and makes the aluminium alloy too expensive. A too high Fe content results in reduced tensile properties and has an adverse effect on for example the breakdown voltage after anodisation due to the formation of amongst others AlFeSi phases and has also an adverse effect on the corrosive gas resistance.

[0018] Zn up to about 0.08%, Cu up to about 0.05%, and Cr up to about 0.03% are tolerable impurities and have an adverse effect on the quality of a subsequently applied anodisation coating, e.g. reduced corrosive gas resistance. In an embodiment the Zn is up to about 0.05%, and preferably up to about 0.03%. In an embodiment the Cu is up to about 0.03%, and preferably up to about 0.02%. In an embodiment the Cr is up to about 0.02%.

[0019] Ti up to 0.06% is added as a grain refiner of the as-cast microstructure. In an embodiment it is present in a range of about 0.01% to 0.06%, and preferably in a range of about 0.01% to 0.04%.

[0020] Balance is made by aluminium and unavoidable impurities. Impurities are present up to 0.03% each and up to 0.10% total.

[0021] In an embodiment the Al-Mg-Si aluminium alloy has a composition consisting of, in wt.%, Mg 0.80% to 1.05%, Si 0.70% to 1.0%, Mn 0.70% to 0.90%, Fe up to 0.20%, Zn up to 0.08%, Cu up to 0.05%, Cr up to 0.03%, Ti up to 0.06%, unavoidable impurities each up to 0.03%, total up to 0.10%, balance aluminium, and with preferred narrower ranges as herein described and claimed.

[0022] The Al-Mg-Si-Mn aluminium alloy is provided as an ingot or slab for fabrication into a hot rolled plate product by casting techniques regular in the art for cast products, e.g. Direct-Chill (DC)-casting, Electro-Magnetic-Casting (EMC)-casting, Electro-Magnetic-Stirring (EMS)-casting, and preferably having an ingot thickness in a range of about 220 mm or more, e.g. 400 mm, 500 mm or 600 mm. After casting the rolling feedstock, the as-cast ingot is commonly scalped to remove segregation zones near the cast surface of the ingot. Grain refiners such as those containing titanium and boron, or titanium and carbon, are used as is well-known in the art to obtain a fine as-cast grain structure.

[0023] The purpose of a homogenisation heat treatment has at least the following objectives: (i) to dissolve as much as possible coarse soluble phases formed during solidification, and (ii) to reduce concentration gradients to facilitate the dissolution step. A preheat treatment achieves also some of these objectives. The homogenisation process is done a temperature range of 550°C to 595°C. In an embodiment the homogenization temperature is at least 555°C, and more preferably at least 565°C. The soaking time at the homogenisation temperature is in the range of about 1 to 20 hours, and preferably does not exceed about 15 hours, and is more preferably in a range of about 5 to 15 hours. The heat-up rates that can be applied are those which are regular in the art.

[0024] The hot rolling is performed to a hot rolling plate thickness of 10 mm or more. In an embodiment the upper-limit is about 230 mm, preferably about 200 mm and more preferably about 180 mm.

[0025] A next important process step is solution heat treating ("SHT") of the hot rolled plate material. The plate product should be heated to bring as much as possible all or substantially all portions of the soluble alloying elements into solution. The SHT is preferably carried out at a temperature in the temperature range of about 540°C to 590°C. A higher SHT temperature provides more favourable mechanical properties, e.g. an increased R_m. In an embodiment the lower-limit for the SHT temperature is 545°C, preferably it is 550°C. In an embodiment the upper-limit for the SHT temperature is about 580°C, and more preferably about 575°C. A low SHT temperature reduces the strength of the aluminium plate and some large Mg_2Si phases main remain undissolved and may create so called "hot spots" and reducing the corrosion resistance after anodization and reduce the breakdown voltage. It is believed that shorter soaking times are very useful, for example in the range of about 10 to 180 minutes, preferably in a range of 10 to 40 minutes, and more preferably in a range of 10 to 35 minutes, for example for plate thicknesses up to 50 mm. A too long soaking time at a relative high SHT temperature results in the growth of several phases adversely affecting the ductility of the aluminium plate. The SHT is typically carried out in a batch or a continuous furnace. After SHT, it is important that the plate material be cooled with a high cooling rate to a temperature of 100°C or lower, preferably to below 40°C, to prevent or minimise the uncontrolled precipitation of secondary phases. On the other hand cooling rates should preferably not be too high to allow for a sufficient flatness and low level of residual stresses in the plate product. Suitable cooling rates can be achieved with the use of water, e.g. water immersion or water jets.

[0026] The SHT and quenched plate material is further cold worked, preferably by means of stretching in the range of about 1% to 5% of its original length to relieve residual stresses therein and to improve the flatness of the plate product. Preferably the stretching is in the range of about 1.5% to 4%, more preferably of about 2% to 3.5%.

[0027] After cooling the stretched plate material is aged, preferably artificially aged, more preferably to provide a T6 condition, more preferably a T651 condition. In an embodiment the artificial ageing is performed at a temperature in the range of 150°C to 190°C, and preferably for a time of 5 to 60 hours.

[0028] In an embodiment the stretch plate material is aged to an over-aged T7 condition, preferably to a T74 or T76 condition, and more preferably to an T7651 condition.

[0029] In a further aspect of the invention it relates to a method of manufacturing a vacuum chamber element, the method comprising the steps of manufacturing the Al-Mg-Si alloy plate having a thickness of at least 10 mm as herein set forth and claimed, and further comprising the subsequent steps of:

(h) machining said aged plate, e.g. in T6, T651, T7, T74, T76, or T7651 condition, into a vacuum chamber element of predetermined shape and dimensions;

(i) surface treating of the vacuum chamber element, preferably by means of anodisation; preferably to provide an anode layer or anode coating layer thickness of at least 20μm, and preferably a thickness of at least 30μm;

(j) optionally the product thus anodised is hydrated or sealed in deionised water at a temperature of at least 80°C and preferably of at least 98°C, preferably for a duration of at least about 1 hour. In an embodiment the hydration is performed in two steps, a first steps with a duration of at least 10 minutes at a temperature of 30°C to 70°C, and a second step with a duration of at least about 1 hour at a temperature of at least 98°C.

[0030] In an embodiment the anodization is performed using an electrolytic solution comprising at least sulfuric acid at a temperature about 15°C to 30°C and a current density from about 1.0 A/dm^2 to about 2 A/dm^2. The acid concentration in the anodizing bath is typically in a range of about 5 to 20 vol.%. The process takes from about 0.5 to 60 minutes, depending on the desired oxide layer thickness. The sulfuric anodizing generally yields an oxide layer with a thickness from about 8 microns to about 40 microns.

[0031] In an embodiment the anodization is performed in an electrolytic solution comprising at least sulfuric acid at a temperature from about 0°C to about 10°C and a current density from about 3 A/dm^2 to about 4.5 A/dm^2. The process generally takes from about 20 minutes to about 120 minutes. This hardcoat anodizing generally yields an oxide layer with a thickness from about 30 microns to about 80 microns, or even thicker.

[0032] The invention relates to a method of manufacturing an aluminium alloy plate for vacuum chamber elements, the method comprising the steps of: (a) providing a rolling feedstock material of an Al-Mg-Si aluminium alloy having a composition comprising of, in wt.%, Mg 0.80%-1.05%, Si 0.70%-1.0%, Mn 0.70%-0.90%, Fe up to 0.20%, Zn up to 0.08%, Cu up to 0.05%, Cr up to 0.03%, Ti up to 0.06%, unavoidable impurities and balance aluminium; (b)homogenizing of the rolling feedstock at a temperature in a range of 550-595°C; (c) hot-rolling of the homogenized rolling feedstock in one or more rolling steps to a hot-rolled plate having a thickness of at least 10 mm; (d) solution heat-treatment (SHT") of the hot rolled plate at a temperature in a range of 540-590°C; (e) rapid cooling the SHT plate; (f) stretching of the cooled SHT plate to obtain a permanent elongation from 1-5%; (g) artificial ageing of the stretched plate.

[0033] The invention is not limited to the embodiments described before, which may be varied widely within the scope of the invention as defined by the appending claims.

Claims

1. Method of manufacturing an aluminium alloy plate for vacuum chamber elements, the method comprising the steps of:

   (a) providing a rolling feedstock material of an Al-Mg-Si aluminium alloy having a composition comprising of, in wt.%,

Mg	0.80% to 1.05%,
Si	0.70% to 1.0%,
Mn	0.70% to 0.90%,

   (continued)

Fe	up to 0.20%,
Zn	up to 0.08%,
Cu	up to 0.05%,
Cr	up to 0.03%,
Ti	up to 0.06%,

   unavoidable impurities each <0.03%, total <0.10%, balance aluminium;
   (b) homogenizing of the rolling feedstock at a temperature in a range of 550°C to 595°C;
   (c) hot-rolling of the homogenized rolling feedstock in one or more rolling steps to a hot-rolled plate having a thickness of at least 10 mm;
   (d) solution heat-treatment (SHT") of the hot rolled plate at a temperature in a range of 540°C to 590°C;
   (e) rapid cooling the SHT plate, preferably by one of spray quenching or immersion quenching in water or other quenching media;
   (f) stretching of the cooled SHT plate to obtain a permanent elongation from 1% to 5%;
   (g) artificial ageing of the stretched plate, preferably to a T6 condition.

2. Method according to claim 1, wherein the hot rolling of the homogenized rolling feedstock is to a plate having a thickness in the range of 10 mm to 230 mm, and preferably in the range of 10 mm to 200 mm.

3. Method according to claim 1 or 2, wherein the Mg-content is in a range of 0.85% to 1.05%.

4. Method according to any one of claims 1 to 3, wherein the Si-content is in a range of 0.70% to 0.95%, and preferably of 0.80% to 0.95%.

5. Method according to any one of claims 1 to 4, wherein the ratio (in wt.%) Mg/Si is more than 0.9, and preferably more than 1.0.

6. Method according to any one of claims 1 to 5, wherein the Mn-content is in a range of 0.75% to 0.85%.

7. Method according to any one of claims 1 to 6, wherein the Fe-content is up to 0.12%, and preferably in a range of 0.03% to 0.12%.

8. Method according to any one of claims 1 to 7, wherein the Ti-content is in a range of 0.01% to 0.06%.

9. Method according to any one of claims 1 to 8, wherein homogenizing of the rolling feedstock is at a temperature in a range of 555°C to 595°C.

10. Method according to any one of claims 1 to 9, where-

in the solution heat treatment of the hot rolled plate is at a temperature in a range of 545°C to 580°C.

11. Method according to any one of claims 1 to 10, wherein the artificial ageing is performed at a temperature in the range of 150°C to 190°C, and preferably for a time of 5 to 60 hours.

12. Method of manufacturing a vacuum chamber element, the method comprising the steps of any one of claims 1 to 11, and further comprising the steps of:

(h) machining said aged plate into a vacuum chamber element;
(i) surface treating of the vacuum chamber element, preferably by means of anodisation.

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EUROPEAN SEARCH REPORT

Application Number
EP 20 17 9258

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The present search report has been drawn up for all claims

1

Place of search	Date of completion of the search	Examiner
The Hague	23 September 2020	Chebeleu, Alice

CATEGORY OF CITED DOCUMENTS

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