

Description

Title: Plates, made of aluminum alloy, for vacuum chamber elements

Technical field

The invention relates to products made of an aluminum alloy intended to be used as vacuum
5 chamber elements in particular for the manufacture of integrated electronic circuits based on
semiconductors, flat display screens as well as photovoltaic panels and to a method for
manufacturing the same.

Prior art

Vacuum chamber elements for the manufacture of integrated electronic circuits based on
10 semiconductors, flat display screens and photovoltaic panels, may typically be obtained from
plates made of an aluminum alloy.

Vacuum chamber elements are elements intended for the manufacture of vacuum chamber
structures and internal vacuum chamber components, in particular vacuum chamber bodies,
valve bodies, flanges, connection elements, sealing elements, passages, diffusers, electrodes. In
15 particular, they are obtained by machining and surface treatment of plates made of an
aluminum alloy. An excellent dimensional stability is very important for the plates used to make
these vacuum chamber elements. Thus, it is particularly important to reduce as much as possible
any deformation of the plate during machining thereof, which allows avoiding additional pre-
machining or final refinishing operations.

20 Patent application EP2263811 relates to rolled products, the surface of which is machined and
having a flatness of 0.2 mm or less. According to an embodiment of this patent application, the
alloy contains 0.3 to 1.5% by weight of Mg, 0.2 to 1, 6% by weight of Si, and furthermore one or
more element(s) selected from the group consisting of 0.8% by weight or less of Fe, 1.0% by
weight or less of Cu, 0.6% by weight or less of Mn, 0.5% by weight or less of Cr, 0.4% by weight
25 or less of Zn, and 0.1% by weight or less of Ti, the remainder being Al and unavoidable impurities.

Patent application WO2014/060660 relates to a vacuum chamber element obtained by
machining and surface treatment of a plate having a thickness at least equal to 10 mm made of
an aluminum alloy having the following composition, in % by weight, Si: 0.4 - 0.7; Mg: 0.4 - 0.7;
Ti 0,01 - < 0.15, Fe < 0.25; Cu < 0.04; Mn < 0.4; Cr 0.01 < 0.1; Zn < 0.04; other elements < 0.05
30 each and < 0.15 in total, the remainder being aluminum.

Patent application WO2018/162823 relates to a vacuum chamber element obtained by
machining and surface treatment of a plate having a thickness at least equal to 10 mm made of

an aluminum alloy having the following composition, in % by weight, Si: 0.4 - 0.7; Mg: 0.4 - 1.0; the ratio in % by weight Mg/Si being lower than 1.8; Ti: 0.01 - 0.15, Fe 0.08 - 0.25; Cu < 0.35; Mn < 0.4; Cr: < 0.25; Zn < 0.04; other elements < 0.05 each and < 0.15 in total, the remainder being aluminum, characterized in that the grain size of said plate is such that the average linear
 5 interception length measured in the L/ST plane according to the standard ASTM E112 is at least equal to 350 μm between the surface and the 1/2 thickness.

Patent application US2010018617 discloses an aluminum alloy for anodic oxidation treatment which comprises as alloy elements 0.1 to 2.0% of Mg, 0.1 to 2.0% of Si and 0.1 to 2.0% of Mn, each content of Fe, Cr and Cu being limited to 0.03 % by weight or less, and wherein the
 10 remainder is composed of Al and unavoidable impurities. In particular, this application discloses a homogenization treatment at a temperature higher than 550°C and lower than or equal to 600°C.

Patent application CN108239712 relates to a slab made of the 6082 aluminum alloy for aviation and to a method for manufacturing the latter. The chemical components of the slab made of the
 15 6082 aluminum alloy comprise, in percentage by weight, 1.0% to 1.3% of Si, 0.1% to 0.3% of Fe, 0.05% to 0.10% of Cu, 0.5% to 0.8% of Mn, 0.6% to 0.9% of Mg, 0.06% to 0.12% of Zn, not more than 0.05% of Cr, not more than 0.05% of Ti and the remainder being Al and unavoidable elements.

Patent application CN108239713 relates to a slab made of an aluminum alloy for an electronic
 20 product and to a method for manufacturing the aluminum alloy slab. The chemical components of the aluminum alloy slab for the apparition of the electronic product comprise, in percentage by weight, 0.3% to 0.4% of Si, not more than 0.10% of Fe, not more than 0.05% of Cu, not more than 0.05% of Mn, 0.45% to 0.55% of Mg, not more than 0.05% of Zn, not more than 0.05% of Cr, not more than 0.05% of Ti and the remainder being Al and unavoidable elements.

Patent application WO2021/064320 relates to plates having a thickness comprised between 8
 25 and 50 mm made of an aluminum alloy having the following composition, in % by weight, Si: 0.7 – 1.3; Mg: 0.6 – 1.2; Mn: 0.65 – 1.0; Fe: 0.05-0.35; at least one element selected from among Cr: 0.1 – 0.3 and Zr: 0.06-0.15; Ti < 0.15; Cu < 0.4; Zn < 0.1; other elements < 0.05 each and < 0.15 in total, the remainder being aluminum and to the method for manufacturing the same. In
 30 particular, the addition of Cr and/or Zr allows obtaining an improved dimensional stability, in particular during the machining steps, while having sufficient static mechanical properties, and an excellent anodizing ability. However, the addition of Cr and/or Zr might prove to be unfavorable for some applications related to vacuum chambers.

Patent application WO2021/250545 relates to a method for manufacturing a slab made of an aluminum alloy for vacuum chamber elements, valves or complete assemblies, the method comprising the steps of: (a) providing a feedstock material for rolling an Al-Mg-Si aluminum alloy having a composition comprising, in % by weight, 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 the remainder being aluminum; (b) homogenizing the rolling feedstock at a temperature within a range from 550 to 595°C; (c) hot rolling the homogenized rolling feedstock in one or more rolling step(s) into a hot-rolled slab having a thickness of at least 10 mm; (d) solution heat treatment of the hot-rolled plate at a temperature within a range from 540 to 590°C; (e) rapid cooling of the solution heat-treated slab; (f) controlled stretching by a permanent elongation of 1 to 5% of the solution heat-treated and cooled slab; (g) artificial aging of the stretched slab.

There is a need for products made of an aluminum alloy intended for making vacuum chamber elements, having sufficient mechanical characteristics, an improved dimensional stability for the machining steps as well as an optimum ability to create oxide layers with high chemical resistance.

Disclosure of the invention

A first object of the invention is a method for manufacturing a plate having a final thickness comprised between 6 and 50 mm wherein

- a) a rolling slab is cast in an aluminum alloy having the following composition, in weight %, Si: 0.6 – 1.3; Mg: 0.6 – 1.2; Mn: 0.65 – 1.0; Fe: 0.04 – 0.20; Cr ≤ 0.05; Ti ≤ 0.15; Cu ≤ 0.1; other elements < 0.05 each and < 0.15 in total, the remainder being aluminum,
- b) said rolling slab is homogenized,
- c) said rolling slab is rolled with an entry temperature of at least 500°C to obtain a plate with a thickness at least equal to 8 mm,
- d) optionally, out an annealing is carried out,
- e) the hot-rolled and optionally annealed plate is cold rolled with a reduction of at least 20%,
- f) the cold-rolled plate is solution heat treated and quenched,
- g) said solution heat-treated and quenched plate is stress relieved by controlled stretching with a permanent elongation of 1 to 5%,
- h) said stretched plate carrying is artificially aged,

i) optionally said aged plate is machined.

Another object of the invention is a plate obtainable by the method according to the invention.

Still another object of the invention is the use of a plate according to the invention to make a vacuum chamber element.

5 **Figures**

[Fig. 1] Figure 1 illustrates the measurement of residual stresses.

Detailed description of the invention

The designation of the alloys is done in accordance with the regulations of The Aluminum Association (AA), known to a person skilled in the art. The definitions of the metallurgical
10 tempers are indicated in the European standard EN 515. Unless stated otherwise, the definitions of the standard EN12258-1 apply.

Unless stated otherwise, the compositions are expressed in % by weight.

Unless stated otherwise, the static mechanical characteristics, in other words the ultimate tensile strength R_m , the conventional tensile yield strength at 0.2% elongation $R_{p0.2}$ and the
15 elongation at break A%, are determined by a tensile test according to the standard ISO 6892-1, the sampling and the direction of the test being defined by the standard EN 485-1, the direction or orientation L corresponds to the lengthwise direction with respect to the main rolling direction and the direction or orientation LT corresponds to the long transverse direction.

In the context of the present invention, the residual stresses have been determined using the
20 bar method based on the successive removal of layers described in particular in the publication "Development of New Alloy for Distortion Free Machined Aluminum Aircraft Components", F. Heymes, B. Commet, B. Dubost, P. Lassince, P. Lequeu, GM. Raynaud, in 1st International Non-Ferrous Processing & Technology Conference, 10-12 March 1997 – Adams's Mark Hotel, St Louis, Missouri and in patent applications WO2004/053180 or WO2006/064113.

This method applies especially to substantially stretched plates, in which the stress state could be considered to be homogeneous in the sampling area of the bars and biaxial; the two main components being located in the directions L and LT, and there is therefore no component in the direction ST. This method is based on the determination of the deflections of two rectangular bars, cut across the thickness of the plate in the direction parallel to the directions L and LT.
25
30 These bars are machined in the direction ST step-by-step. After each step, the deformation and/or the detection is measured and the thickness of the bar is reported. In the context of the

present invention, the deflection has been measured using a displacement sensor. Thus, it has been possible to calculate the residual stress profiles in the directions L and LT.

A measurement is performed after each machining pass. Typically, between 18 and 25 passes are considered to obtain a sufficient number of points to calculate the stress profile. The machining depth should not be smaller than 1 mm, in order to obtain good cutting quality and should be smaller than 4 mm (for very thick plates, not all passes are measured). It is also possible to use chemical machining to remove a very small thickness of metal. The machining step should be the same for the two samples (*i.e.* in the direction L and in the direction LT).

After each machining pass, the bar is detached from the vise, and the temperature is allowed to stabilize before measuring the deformation. At each step i , the thickness $h(i)$ and the deflection $f(i)$ are reported. The diagram of Figure 1 shows how these data are collected.

These data allow calculating the stress profile in each bar in the form of a curve $u(i)_L$, which corresponds to the average stress in the layer removed during the machining step i , given by the following equations:

For $i = 1$ to $N-1$

$$u(i)_L = -\frac{4}{3} \frac{E}{lf^2} [f(i+1)_L - f(i)_L] \frac{h(i+1)^3}{h(i)(h(i)-h(i+1))} - S(i)_L$$

$$S(i)_L = \frac{4E}{lf^2} \sum_{k=1}^{i-1} [f(k+1)_L - f(k)_L] \left[-(h(i) + h(i+1)) + \frac{h(k+1)(3h(k) - h(k+1))}{3h(k)} \right]$$

where E is the Young's modulus of the thick plate and lf the length between the support points during the measurement of the deflection. Thus, two profiles are obtained: $u(i)_L$ et $u(i)_{LT}$ which correspond to bars with a rectangular cross-section in the directions L and LT.

The stress profiles in the plate are obtained by the following equations:

For $i = 1$ to $N-1$

$$\sigma(i)_L = \frac{u(i)_L + \nu u(i)_{LT}}{1 - \nu^2}$$

$$\sigma(i)_{LT} = \frac{u(i)_{LT} + \nu u(i)_L}{1 - \nu^2}$$

where ν is the Poisson's ratio of the plate.

Afterwards, the elastic energy stored in the plate (W_L , W_{LT} et W_{tot} in KJ/m³) could be calculated based on the equations:

$$W_L = \frac{500}{Eh} \sum_{i=1}^{N-1} \sigma(i)_L [\sigma(i)_L - \nu \sigma(i)_{LT}] dh(i)$$

$$W_{LT} = \frac{500}{Eh} \sum_{i=1}^{N-1} \sigma(i)_{LT} [\sigma(i)_{LT} - \nu \sigma(i)_L] dh(i)$$

$$W_{tot} = W_L + W_{LT}$$

where W_{tot} is the total elastic energy in the plate (expressed in kJ/m³) and h is the total thickness of the plate.

According to the invention, improved plates made of an aluminum alloy of the 6XXX series, in particular plates intended for making vacuum chamber elements, are obtained by a method comprising steps of casting, homogenization, hot rolling, optionally annealing, cold rolling, solution heat treatment, quenching, stress-relieving, aging and optionally machining.

5 In a first step, a rolling slab made of an aluminum alloy having the composition according to the invention is cast.

In particular, the composition according to the invention allows obtaining an advantageous balance between the mechanical properties, the dimensional stability during machining steps and the properties of use in construction and use of vacuum chamber elements, in particular
10 the ability to create oxide layers with high chemical resistance.

In particular, the present Inventors have noticed that, compared to a standard composition of the AA6082 alloy, the presence of a large amount of Mn allows improving the properties, in particular by favoring a fibrous microstructure.

By "fibrous microstructure", it should be understood a microstructure, the grains of which have,
15 in a L/ST section, an aspect ratio between the directions L and ST of at least 4, preferably at least 8 and preferably at least 12.

Thus, the Mn content is from 0.65 to 1.0% by weight. Preferably, the minimum Mn content is 0.70%, advantageously 0.75% and preferably 0.80% and even 0.85%. In an embodiment of the invention, the Mn content is from 0.8 to 1.0% by weight.

20 The addition of Fe is also necessary. Thus, the Fe content is from 0.04 to 0.20% by weight. Preferably, the minimum Fe content is 0.06%, advantageously 0.07% and preferably 0.08%. Preferably, the maximum Fe content is 0.18%, which could contribute in particular to obtaining the desired granular structure. In an embodiment of the invention, the Fe content is from 0.08 to 0.18% by weight.

Mg and Si are added to reach the desired mechanical characteristics thanks to the formation of Mg_2Si .

The Mg content is from 0.6 to 1.2% by weight. Preferably, the minimum Mg content is 0.63%, advantageously 0.66% and preferably 0.70%. Preferably, the maximum Mg content is 1.1%,
 5 advantageously 1.0% and preferably 0.9% and even 0.85%. In an embodiment of the invention, the Mg content is from 0.70 to 0.85% by weight.

The Si content is from 0.6 to 1.3% by weight. Preferably, the minimum Si content is 0.63%, advantageously 0.66% and preferably 0.70%. Preferably, the maximum Si content is 1.2% or 1.1%, advantageously 1.0% and preferably 0.9% and even 0.85%. In an embodiment of the
 10 invention, the Si content is from 0.70 to 0.85% by weight.

The Mg and Si contents are balanced, indeed an excess of Si might impact the anodization performance and affect the performance in use of vacuum chamber components. Preferably, the Mg content and the Si content differ by more than 0.05% by weight.

The Ti content is at most 0.15% by weight. It may be advantageous to add Ti, in particular for
 15 controlling the grain size during casting. In an embodiment of the invention, the Ti content is from 0.01 to 0.05% by weight.

The Cu content is at most 0.1% by weight. Advantageously, the Cu content is at most 0.07% by weight and preferably at most 0.07% by weight in particular so as not to degrade the ability to create layers of anodic oxides with high chemical resistance.

20 The Cr content is at most 0.03% by weight. Advantageously, the Cr content is at most 0.02% by weight and preferably at most 0.01% by weight, in particular so as to avoid the formation of AlCr or Al(Fe,Cr) , the formation of which might affect the performance in use of the anode layers created on vacuum chamber components.

The other elements are present as unavoidable impurities with a content lower than 0.05% by weight each and lower than 0.15% by weight in total, the remainder being aluminum.
 25 Advantageously, the Zr content is at most 0.03% by weight and preferably at most 0.02% by weight, in particular to avoid the formation of Al_3Zr or Al(Fe,Zr) , the formation of which might affect the performance in use of anode layers created on vacuum chamber components.

Preferably, the alloy according to the invention is cast by vertical direct chill semi-continuous
 30 casting. The slab thus obtained may be scalped, i.e. machined, before the subsequent steps. Afterwards, the rolling slab is homogenized. Preferably, the homogenization temperature is lower than 580°C. In an advantageous embodiment of the invention, the homogenization

temperature is from 520°C to 570°C. Afterwards, the hot rolling is carried out to obtain a plate having a thickness at least equal to 8 mm. The hot rolling entry temperature is at least 500°C, preferably at least 520°C and preferentially at least 530°C.

An intermediate heat treatment, allowing in particular restoring the plate thus hot-rolled may optionally be carried out afterwards, advantageously at a temperature comprised between 300°C and 400°C.

A cold rolling, with a reduction rate of at least 20%, is carried out following the hot rolling or the intermediate heat treatment if it is performed. The present inventors have noticed that the presence of a cold rolling step before solution heat treating and quenching allows improved dimensional stability properties during machining. Advantageously, the reduction rate by cold rolling is from 25% to 35%.

Afterwards, the plate thus cold-rolled undergoes a solution heat treatment followed by quenching. Preferably, the dissolution is performed at a temperature from 510°C to 570°C. The quenching is typically carried out by immersion or spraying with cold water. Afterwards, it is stress-relieved by controlled stretching with a permanent elongation of from 1 to 5%, preferably from 1.5 to 3%, said plate thus solution heat-treated and quenched. Preferably, the stress-relief is performed in the longitudinal and transverse directions.

Finally, an aging is carried out, typically at a temperature comprised between 150°C and 210°C, preferably to obtain a TX51 temper.

Optionally, the plate thus aged is machined down to its final thickness.

The plates obtainable by the method according to the invention have particularly advantageous properties.

The mechanical properties of the plates according to the invention are particularly advantageous. Preferably, the plates according to the invention have a tensile yield strength $R_{p0.2}(LT)$ of at least 230 MPa, preferably at least 240 MPa and preferably at least 250 MPa, and/or an ultimate tensile strength $R_m(LT)$ of at least 255 MPa, preferably at least 265 MPa and preferably at least 275 MPa and/or an elongation at break $A\%$ of at least 10%, preferably at least 12% and preferably at least 14%.

The plates according to the invention have a low level of internal stresses. Thus, the total elastic energy W_{tot} is lower than 0.5 kJ/m³, preferably lower than 0.4 kJ/m³, preferably lower than 0.3 kJ/m³, and preferably lower than 0.2 kJ/m³.

Preferably, the plates according to the invention have a fibrous microstructure.

According to the invention, plates according to the invention are used to make a vacuum chamber element. Thus, the plates according to the invention are machined and anodized to make vacuum chamber elements, such as vacuum chamber bodies, valve bodies, flanges, connection elements, sealing elements, passages, diffusers, electrodes. Indeed, the plates according to the invention have an improved dimensional stability, in particular during machining steps, while having sufficient static mechanical properties, and an optimum ability to create oxide layers with high chemical resistance.

Example

In this example, rolling slabs made of an alloy the composition of which is given in Table 1 have been prepared.

[Table 1]

Alloy	Si	Mg	Mn	Fe	Cr	Ti	Cu	Other elements
I	0.76	0.76	0.80	0.18	0.002	0.013	0.004	< 0.05
II	0.73	0.73	0.82	0.17	0.007	0.019	0.033	< 0.05
III	0.75	0.72	0.83	0.17	0.009	0.022	0.029	< 0.05

Composition of the alloys in percentage by weight

The slabs have been homogenized at 540°C. The slabs have been hot-rolled down to a thickness of from 14 to 32 mm depending on the case. The hot rolling entry temperature was comprised between 530°C and 550°C. In a reference method A, the final thickness has been obtained by hot rolling. In a method B, according to the invention, a 30% cold rolling has been carried out after the hot rolling to obtain the final thickness. The plates thus obtained have been solution heat treated at 540°C, quenched, stress-relieved by controlled stretching in the longitudinal and transverse directions with a permanent elongation of 2% and artificially aged to obtain an overaged temper.

The tensile static mechanical characteristics, in other words the ultimate tensile strength R_m , the conventional tensile yield strength at 0.2% elongation $R_{p0.2}$ and the elongation at break A%, have been determined by a tensile test according to the standard NF EN ISO 6892-1 (2016) in the long transverse direction (LT), the sample and the direction of the test being defined by the standard EN 485 (2016). The characterizations have been performed in the long transverse direction.

The results are given in Table 2.

[Table 2]

Test	Method	Alloy	Final thickness (mm)	R _m (LT) MPa	R _{p0.2} (LT) MPa	A %
1	A	I	14	260	234	11
2	A	I	17	270	245	13
3	A	I	22	265	239	11
4	B	II	14	277	250	14
5	B	II	14	283	259	14
6	B	II	17	278	250	14
7	B	III	17	283	257	14
8	B	III	22	284	258	14
9	B	II	22	284	258	16

Static mechanical properties

The elastic energy has been measured according to the bar method already described.

[Table 3]

Method	Test	Final thickness (mm)	Average elastic energy W _{tot}
A	1	14	0.56
A	2	17	0.90
A	3	22	1.37
B	4, 5	14	0.08
B	6, 7	17	0.18
B	8, 9	22	0.15

- 5 **Average elastic energies determined by the bar method** (3 measurements per part, 6 measurements in the case where 2 parts are concerned)

The parts obtained with the method according to the invention have a substantially lower elastic energy than that obtained with the reference method, while having more advantageous mechanical characteristics.

Claims

1. A method for manufacturing a plate having a final thickness comprised between 6 and 50 mm, wherein
 - a) a rolling slab is cast in an aluminum alloy having the following composition, in weight %,5
Si: 0.6 – 1.3; Mg: 0.6 – 1.2; Mn: 0.65 – 1.0; Fe: 0.04 – 0.20; Cr ≤ 0.05; Ti ≤ 0.15; Cu ≤ 0.1; other elements < 0.05 each and < 0.15 in total, the remainder being aluminum,
 - b) said rolling slab is homogenized,
 - c) said rolling slab is rolled with an entry temperature of at least 500°C to obtain a plate with a thickness at least equal to 8 mm,
 - 10 d) optionally, out an annealing is carried out,
 - e) the hot-rolled and optionally annealed plate is cold rolled with a reduction of at least 20%,
 - f) the cold-rolled plate is solution heat treated and quenched,
 - g) said solution heat-treated and quenched plate is stress relieved by controlled stretching15
with a permanent elongation of 1 to 5%,
 - h) said stretched plate carrying is artificially aged,
 - i) optionally said aged plate is machined.
2. The method according to claim 1, wherein the Mn content is from 0.8 to 1.0% by weight.
3. The method according to claim 1 or claim 2, wherein the Fe content is from 0.08 to 0.18%20
by weight.
4. The method according to any one of claims 1 to 3, wherein the reduction rate by cold rolling is from 25% to 35%.
5. A plate obtainable by the method according to any one of claims 1 to 4.
6. The plate according to claim 5, having a tensile yield strength $R_{p0.2}(LT)$ of at least 230 MPa,25
preferably at least 240 MPa and preferably at least 250 MPa, and/or an ultimate tensile strength $R_m(LT)$ of at least 255 MPa, preferably at least 265 MPa and preferably at least 275 MPa and/or an elongation at break A% of at least 10%, preferably at least 12% and preferably at least 14%.

7. The plate according to claim 5 or claim 6, such that the total elastic energy W_{tot} is lower than 0.5 kJ/m^3 , preferably lower than 0.4 kJ/m^3 , preferably lower than 0.3 kJ/m^3 , and preferably lower than 0.2 kJ/m^3 .
8. The plate according to any one of claims 5 to 7, the microstructure of which is fibrous.
- 5 9. A use of a plate according to any one of claims 5 to 8 for making a vacuum chamber element.
10. The use according to claim 9, wherein the plates according to the invention are machined and anodized to make vacuum chamber elements, such as vacuum chamber bodies, valve bodies, flanges, connection elements, sealing elements, passages, diffusers, electrodes.