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(54) **METHOD OF MANUFACTURING AN AL-MG-MN ALLOY PLATE PRODUCT**

(57) The invention relates to a method of manufacturing a rolled Al-Mg-Mn alloy plate product comprising the steps of: (a) providing a rolling feedstock material of an Al-Mg-Mn alloy having a composition comprising of, in wt.%, Mg 4.8-6.0%, Mn 0.3-1.25%, Zn up to 0.9%, Fe up to 0.4%, Si up to 0.3%, Cu up to 0.2%, Cr up to 0.25%, Zr up to 0.25%, Ti up to 0.25%, unavoidable impurities and balance aluminium; (b) heating the rolling feedstock to a temperature in a range of 480-550°C; (c) hot-rolling of the heated rolling feedstock to a hot-rolled plate having

a final gauge in a range of 3-15 mm, and wherein the hot-mill entry temperature is in a range of 400-550°C, and the hot-mill exit temperature is in a range of 130-285°C; and wherein the hot rolling of the rolling feedstock to final gauge is without cold rolling the rolling feedstock prior to the final gauge; (d) annealing of the hot-rolled plate at final gauge at an annealing temperature in a range of 300-550°C; and (e) cooling to ambient temperature.

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Description

FIELD OF THE INVENTION

5 **[0001]** The invention relates to a method of manufacturing an Al-Mg-Mn plate product. The plate material can be used amongst others for civil engineering purposes like shipbuilding, truck trailers, and silo construction.

BACKGROUND OF THE INVENTION

10 **[0002]** For civil engineering purposes, for example shipbuilding, silo construction, and pressure vessels, aluminium alloy plate materials of the AA5083-series are one of the most widely applied aluminium alloys. This aluminium alloy provides a reasonable balance of mechanical strength, good corrosion resistance, and weldability.

[0003] One of the preferred tempers is the H111 and involves hot rolling of the rolling feedstock, optionally cold rolling to final gauge, annealing and moderate strain-hardening by stretching or levelling.

15 **[0004]** There is a demand for Al-Mg-Mn plate material suitable for civil engineering purposes that offers the possibility for down-gauging of the applied aluminium plate material. This requires an increased strength of the plate material while maintaining a good formability by reference to elongation and bendability, corrosion resistance, and weldability.

[0005] It is an object of the invention to provide a method of manufacturing an Al-Mg-Mn alloy plate product having a good balance of, in particular, strength, elongation and bendability.

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DESCRIPTION OF THE INVENTION

[0006] As will be appreciated herein below, except as otherwise indicated, aluminium alloy and temper designations refer to the Aluminium Association designations in Aluminum Standards and Data and the Registration Records, as published by the Aluminium Association in 2018 and are well known to the persons skilled in the art.

25 **[0007]** For any description of alloy compositions or preferred alloy compositions, all references to percentages are by weight percent unless otherwise indicated.

[0008] 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 0.1% Cu may include an alloy having no Cu.

30 **[0009]** As used herein, the term "about" when used to describe a compositional range or amount of an alloying addition means that the actual amount of the alloying addition may vary from the nominal intended amount due to factors such as standard processing variations as understood by those skilled in the art.

[0010] This and other objects and further advantages are met or exceeded by the present invention providing a method of manufacturing a hot-rolled Al-Mg-Mn alloy product of 3 to 15 mm final gauge, the method comprising the steps, in that order, of:

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

40	Mg	4.80% to 6.0%,
	Mn	0.30% to 1.25%,
	Zn	up to about 0.9%, preferably 0.30% to 0.9%,
	Fe	up to about 0.40%, preferably up to about 0.30%,
	Si	up to about 0.30%, preferably up to about 0.20%,
45	Cu	up to about 0.20%, preferably up to about 0.10%,
	Cr	up to about 0.25%,
	Zr	up to about 0.25%,
	Ti	up to about 0.25%, preferably about 0.005% to 0.10%,

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unavoidable impurities each <0.05%, total <0.2%, balance aluminium;

(b) heating the rolling feedstock to a temperature in a range of about 480°C to 550°C;

(c) hot-rolling of the heated rolling feedstock in one or more rolling steps to a hot-rolled plate having a final gauge in a range of 3 mm to 15 mm, and wherein the hot-mill entry temperature is in a range of about 400°C to 550°C, and the hot-mill exit temperature is in a range of about 130°C to 285°C; and wherein the hot rolling of the rolling feedstock to final gauge is without cold rolling the rolling feedstock prior to the final gauge;

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(d) annealing of the hot-rolled plate at final gauge at an annealing temperature in a range of about 300°C to 550°C; and

(e) cooling of the annealed hot-rolled plate at final gauge from annealing temperature to ambient temperature. Next the

cooled feedstock at final gauge is suitable for finishing operations such as levelling or stretching to improve product flatness, edge-trimming and slitting, and cut-to-length.

[0011] The method according to this invention allows for the production of Al-Mg-Mn-(Zn) plate products having a tensile yield strength of at least 150 MPa, an ultimate tensile strength of at least 310 MPa, and an elongation at fracture (A50) of at least 18%, and with improved values are herein described and claimed. In addition, the method allows for the production of Al-Mg-Mn-(Zn) plate products having a very good bendability. In particular, it allows bending angles of 180° at bending radii of 4 times, and preferably 3 times, and in the best example, 2 times the material thickness. The bendability is an important parameter as it allows the shaping or forming of products using the Al-Mg-Mn-(Zn) plate product into particular shapes. The mechanical properties have been measured in accordance with DIN-EN-ISO 6892-1 (2016), and the bendability has been measured in accordance with DIN-EN-ISO 7438 (2016). The plate products have a good corrosion resistance and are fusion weldable by means of various fusion welding techniques known in the art.

[0012] These properties are achieved in a more efficient manufacturing process as there is no need of any cold rolling operation of the rolling feedstock to final gauge. Down-gauging of the plate product is possible, offering weight saving opportunities in a civil construction of a storage vessel, such as the hull of silos in a trailer, truck, or container.

[0013] The method of the present invention can be operated more economically to provide a plate product having better mechanical properties than AA5083-H111.

[0014] The Al-Mg-Mn-(Zn) alloy can be provided as an ingot or slab for fabrication into rolling feedstock using casting techniques regular in the art for cast products, e.g., DC-casting, EMC-casting, 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. In another embodiment, thin gauge slabs resulting from continuous casting, e.g., belt casters or roll casters, also may be used, and having a thickness of up to about 40 mm. After casting the rolling feedstock, the thick as-cast ingot is commonly scalped to remove segregation zones near the cast surface of the ingot.

[0015] The heating, e.g., by homogenization and/or pre-heating, prior to hot rolling is carried out at a temperature in the range of about 480°C to 550°C. In either case, it decreases the segregation of alloying elements in the material as cast. In multiple steps, the Zr, Cr and Mn can be intentionally precipitated to control the microstructure of the hot mill exit feedstock. If the treatment is carried out below about 480°C, the resultant homogenisation effect is inadequate. It is preferred to have a temperature of more than 500°C. If the temperature is above about 550°C, eutectic melting might occur resulting in undesirable pore formation. It has been found that a higher temperature results in an increased elongation in the final plate product at a small trade-off of the tensile yield strength. The preferred time of the above treatment is between 1 and 30 hours, for example, 8 hours or 18 hours.

[0016] In an embodiment, a separate homogenisation treatment is performed prior to pre-heating or heating the rolling feedstock. The homogenisation treatment is performed in a temperature range of 480°C to 550°C. The soaking time at the homogenisation temperature is preferably between 1 and 30 hours.

[0017] Commonly, a pre-heat refers to the heating of an ingot to a set temperature and soaking at this temperature for a set time followed by the start of the hot rolling at that temperature. Homogenisation refers to a heating and cooling cycle applied to a rolling ingot in which the final temperature after homogenisation is ambient temperature.

[0018] In the embodiment of process step (b) where solely a heating or pre-heating is performed without a separate prior homogenisation treatment, then it is preferred that the heating or pre-heating is performed at a temperature in the range of about 480°C to 550°C. Further, it is preferred to have a set temperature of more than 500°C.

[0019] In the embodiment of process step (b) where a homogenization treatment is performed prior to the pre-heat, the pre-heat temperature is then set in a range of 400°C to 550°C. It is preferred that the pre-heat temperature is in a range of 480°C to 550°C, and preferably above 500°C, followed by the start of the hot rolling process at that temperature.

[0020] However, it is possible to pre-heat the already homogenized rolling feedstock to a set temperature in a range of 400°C to 480°C, preferably 430°C to 480°C, followed by the start of the hot rolling process at that temperature. As the rolling feedstock has been homogenized, it has been subjected to at least one process step of heating to a temperature in a range of about 480°C to 550°C, even when the pre-heat temperature is set at a lower temperature followed by the start of the hot rolling process at that temperature.

[0021] The first hot rolling step begins while the heated or pre-heated feedstock is at a temperature in the range of about 400°C to 550°C, preferably about 480°C to 550°C, and is more preferably above about 500°C.

[0022] In an embodiment, in the first hot rolling operation of the preheated feedstock at the defined temperature, it is subjected to breakdown hot rolling in one or more passes using reversing or non-reversing mill stands that serve to reduce the thickness of the feedstock to a gauge range of 15 to 40 mm, and preferably of 15 to 30 mm, and more preferably of 15 to 25 mm. The breakdown rolling starts at about 400°C to 550°C, preferably at about 480°C to 550°C, and more preferably at a temperature of about 500°C or more. Preferably, the hot-mill process temperature should be controlled such that after the last rolling pass the hot-mill exit temperature of the feedstock is in a range of about 370°C to 495°C. A more preferred lower-limit is about 400°C. A more preferred upper-limit is about 465°C.

[0023] Next after breakdown hot-rolling, the feedstock is supplied to a mill for hot finishing rolling in one or more passes to a final gauge in the range of 3 to 15 mm, preferably 3 to 10 mm, for example 4 mm or 5 mm. The hot finishing rolling

operation can be done, for example, using a reverse mill or a tandem mill. The temperature of the hot rolled feedstock when the feedstock is inputted into the mill for hot finishing rolling is maintained preferably at a temperature of about 370°C to 495°C. A more preferred lower-limit is about 400°C. A more preferred upper-limit is about 465°C.

5 **[0024]** Control of the hot-mill exit temperature of the rolled feedstock is important to arrive at the desired balance of metallurgical and mechanical properties, and the hot-mill temperature should be controlled such that after the last rolling pass upon leaving the hot-mill, the hot-mill exit temperature of the rolling feedstock is in a range of about 130°C to 285°C. A preferred lower-limit is about 150°C, and more preferably about 175°C. A preferred upper-limit is about 275°C, and more preferably about 250°C, and more preferably about 235°C. At a too low exit-temperature of the rolling feedstock, the strength and the hardness of the final plate product will be too high. A too low exit-temperature will also adversely affect the coiling behavior of the feedstock following the hot-rolling operation as well as in a subsequent finishing operation. Whereas at too high exit-temperatures, at least the strength and hardness of the feedstock will be too low and provide an unfavorable balance of properties.

10 **[0025]** In an embodiment following the last hot-rolling step, the hot-rolled feedstock at final gauge is cooled to ambient temperature.

15 **[0026]** In a preferred embodiment, the hot-rolled feedstock at final gauge is cooled from hot-mill exit-temperature to ambient temperature by immediately coiling of the hot-rolled feedstock and allowing the coil to cool, preferably by means of air cooling, in an ambient environment to ambient temperature and stored.

[0027] It is an important aspect of the invention that the hot rolling of the rolling feedstock to final gauge is without cold rolling the rolling feedstock prior to the final gauge.

20 **[0028]** Following the hot-rolling operation, the plate material at final gauge is annealed at a temperature in a range of about 300°C to 550°C, for example about 400°C or 410°C. A preferred lower limit for the annealing temperature is about 360°C and more preferably about 380°C. A preferred upper limit for the annealing temperature is about 450°C, and more preferably about 430°C. The annealing operation results in particular to an increase in the elongation at fracture of the plate product.

25 **[0029]** In an embodiment, the plate material is being annealed coiled condition. Commonly, such an annealing operation is performed by placing one or more coils of ambient temperature in a furnace at a temperature of about 300°C to 550°C. As the heating-up of coiled material is relatively slow, the coiled plate material is placed in the annealing furnace for about 1 to 10 hours soak time, preferably about 1 to 8 hours, and more preferably for about 1 to 6 hours, and subsequently removed from the annealing furnace and allowed to cool in an ambient environment to ambient temperature and stored.

30 **[0030]** In another embodiment, the plate material is being annealed as individual plate material of limited length, for example, 6 or 10 meters. Commonly, such an annealing operation is performed by placing a single or multiple plates of ambient temperature in an annealing furnace at a soak temperature of about 300°C to 550°C. As the heating-up of individual plate material is relatively fast, the plate material is placed in the pre-heated annealing furnace for about 10 to 90 minutes soak time, preferably about 10 to 60 minutes, and subsequently removed from the annealing furnace and allowed to cool in an ambient environment to ambient temperature and stored. The faster heat-up rate in this embodiment is preferred over coil annealing as it provides a desired increase in elongation at fracture of the final plate material.

35 **[0031]** Following the annealing step, the annealed hot-rolled plate at final gauge is cooled from annealing temperature to ambient temperature and stored. Next, the cooled plate material at final gauge is suitable for finishing operations such as levelling in case of coiled plate material or stretching (typically up to about 1.5%) in case of individual plate material to improve product flatness, edge-trimming and slitting, and cut-to-length.

40 **[0032]** The careful control of the hot-rolling process and annealing and cooling to ambient temperature results in an Al-Mg-Mn-(Zn) plate product having a fully recrystallized microstructure and providing the required balance of properties. With fully recrystallized, it is meant that the degree of recrystallization of the microstructure is more than about 75%, preferably more than about 80%, and more preferably not more than 90%.

45 **[0033]** In the aluminium alloy product manufactured in accordance with the method of the invention, the Mg-content should be in a range of about 4.80% to 6.0% and forms the primary strengthening element of the alloy. A preferred lower limit for the Mg-content is about 5.0%, and more preferably about 5.1%, to provide increased strength. A preferred upper limit for the Mg-content is about 5.8%.

50 **[0034]** The Mn-content should be in the range of about 0.30% to 1.25% and is another essential alloying element. A preferred upper-limit for the Mn-content is about 1.1 %, and more preferably about 0.9%, to provide a balance in strength and bendability. A preferred lower-limit for the Mn-content is about 0.5%, and more preferably about 0.55%.

[0035] The Zn-content is up to 0.9%. In a preferred embodiment, the Zn-content should be in the range of 0.30% to 0.9% and is then another essential alloying element to provide the required strength, elongation and corrosion resistance.

55 **[0036]** To control the microstructure of the final product, next to the addition of Mn, it is preferred to have a purposive addition of either Cr or Zr each up to about 0.25% as dispersoid-forming elements, whereby the addition of Zr is preferred. A preferred addition of Zr is in a range of about 0.05% to 0.25%, and more preferably of about 0.05% to 0.20%. When Zr is added purposively, it is then preferred that the Cr level does not exceed 0.1%, and is preferably less than about 0.05%.

[0037] Fe is a common impurity in aluminium alloys and should not exceed 0.40%. For highly demanding applications,

the content should not exceed 0.30%, and preferably it does not exceed 0.25%.

[0038] In addition, Si is a common impurity in aluminium alloys and should not exceed 0.30%. For highly demanding applications, the content should not exceed 0.25%, and preferably it does not exceed 0.20%.

[0039] Cu may have an adverse effect on the corrosion resistance of the aluminium alloy, and its content should not exceed 0.20%, and preferably, it does not exceed 0.10%.

[0040] Ti is important as a grain refiner during solidification of both ingots and welded joints produced using the hot-rolled aluminium alloy plate product of the invention. Ti levels should not exceed about 0.25%, and the preferred range for Ti is about 0.005% to 0.10%. Ti can be added as a sole element or with either boron or carbon serving as a casting aid for grain size control.

[0041] In an embodiment of the invention the Al-Mg-Mn-Zn alloy product consists of, in wt. %: Mg 4.80% to 6.0%, Mn 0.30% to 1.25%, Zn up to 0.9%, Fe up to 0.40%, Si up to 0.30%, Cu up to 0.20%, Cr up to 0.25%, Zr up to 0.25%, Ti up to 0.25%, unavoidable impurities each <0.05%, total <0.2%, balance aluminium; and with preferred narrower compositional ranges as herein described and claimed.

[0042] The method according to this invention enables the production of Al-Mg-Mn-(Zn) plate material having a composition as herein described and claimed and having in a gauge range of 3 mm to 15 mm, preferably 3 mm to 10 mm, a tensile yield strength in the LT-direction of at least 150 MPa, preferably of at least 160 MPa, and more preferably of at least 170 MPa. The ultimate tensile strength in the LT-direction is at least 310 MPa, and preferably at least 320 MPa, and more preferably at least 330 MPa. The elongation at fracture (A50) is at least 18%, preferably at least 20%, and more preferably at least 22%. In an embodiment, the elongation at fracture (A50) does not exceed 35%. In addition, the method allows for the production of Al-Mg-Mn-(Zn) plate products having a very good bendability. In particular it allows bending angles of 180° at bending radii of 4 times, and preferably 3 times, and in the best examples 2 times the material thickness.

[0043] The plate material at final gauge obtained by the method according to this invention is an ideal candidate for use in civil constructions such as vessels for transporting goods, storage vessels like the hull of a silo in a trailer, truck, or container.

[0044] The invention will now be illustrated with reference to non-limiting embodiments according to the invention.

Example 1.

[0045] On an industrial scale of processing rolling ingots of 600 mm thickness have been DC-cast of an AlMgMn alloy, and subsequently scalped, preheated for 10 hours at about 505°C, hot rolled using an entry temperature of about 505°C and then hot rolled to 4 mm final gauge. Two different hot-mill exit temperature were applied, namely Ingot A was 225°C (invention) and Ingot B was 295°C (comparative). The hot-rolled plates were not cold rolled. Upon leaving the hot-mill the plate materials were immediately coiled and allowed to cool to ambient temperature in an ambient environment. The coils were then cut-to-length and the resultant plate materials were annealed by soaking for about 25 minutes at about 400°C. After cooling to room temperature the plates were stretched and measured for their mechanical properties in accordance with DIN-EN-ISO 6892-1 (2016). The results are listed in Table 1.

[0046] The aluminium alloy consisted of 5.3% Mg, 0.8% Mn, 0.45% Zn, 0.1 % Zr, 0.1 % Fe, 0.08% Si, 0.01% Cu, 0.02% Ti, balance impurities and aluminium.

Table 1. Mechanical properties.

Ingot	Hot-mill entry	Hot-mill exit	YS [MPa]	UTS [MPa]	A50 [%]
A	505°C	225°C	177	333	24.5
B	505°C	295°C	174	335	17.0

[0047] From the results of Table 1, it can be seen that a careful control of the hot-mill exit temperature in accordance with the invention (Ingot A) while keeping the other processing parameters the same results in a hot-rolled plate material having in the annealed condition a significantly increased elongation.

[0048] 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. A method of manufacturing a rolled aluminium-magnesium-manganese alloy plate product comprising the steps of:

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

Mg	4.80% to 6.0%,
Mn	0.30% to 1.25%,
Zn	up to 0.9%,
Fe	up to 0.40%,
Si	up to 0.30%,
Cu	up to 0.20%,
Cr	up to 0.25%,
Zr	up to 0.25%,
Ti	up to 0.25%,

unavoidable impurities each <0.05%, total <0.2%, balance aluminium;

(b) heating the rolling feedstock to a temperature in a range of 480°C to 550°C;

(c) hot-rolling of the heated rolling feedstock in one or more rolling steps to a hot-rolled plate having a final gauge in a range of 3 mm to 15 mm, and wherein the hot-mill entry temperature is in a range of 400°C to 550°C, and the hot-mill exit temperature is in a range of 130°C to 285°C; and wherein the hot rolling of the rolling feedstock to final gauge is without cold rolling the rolling feedstock prior to the final gauge;

(d) annealing of the hot-rolled plate at final gauge at an annealing temperature in a range of 300°C to 550°C; and

(e) cooling of the annealed hot-rolled plate at final gauge from annealing temperature to ambient temperature.

2. Method according to claim 1, wherein the hot-rolled plate at final gauge is coiled upon exiting the hot-mill.
3. Method according to claim 1, wherein the hot-rolled plate at final gauge is coiled upon exiting the hot-mill and cooled to ambient temperature prior to the annealing step.
4. Method according to any one of claims 1 to 3, wherein during step (c) the hot-mill exit-temperature is in a range of 130°C to 250°C, preferably in a range of 175°C to 250°C.
5. Method according to any one of claims 1 to 4, wherein the annealing is by annealing coiled hot-rolled plate for 1 to 8 hours, preferably 1 to 6 hours, at temperature in a range of 300°C to 550°C.
6. Method according to any one of claims 1 to 4, wherein the annealing is by annealing the hot-rolled plate for 10 to 90 minutes at a temperature in a range of 300°C to 550°C.
7. Method according to any one of claims 1 to 6, wherein the annealing temperature is in a range of 300°C to 450°C.
8. Method according to any one of claims 1 to 7, wherein the aluminium alloy has a Zn-content in a range of 0.30% to 0.9%.
9. Method according to any one of claims 1 to 8, wherein the aluminium alloy has a Mn-content of at most 1.1 %, and preferably of at most 0.90%.
10. Method according to any one of claims 1 to 9, wherein the aluminium alloy has a Mg-content of at least 5.0%, and preferably of at least 5.10%.
11. Method according to any one of claims 1 to 10, wherein the aluminium alloy has a Zr-content in a range of 0.05% to 0.25%.
12. Method according to any one of claims 1 to 11, wherein the hot-rolled and annealed plate at final gauge has an elongation at fracture (A50) of at least 20%, and preferably of at least 22%.
13. Method according to any one of claims 1 to 12, wherein the hot-rolled and annealed plate at final gauge has a tensile yield strength of at least 150 MPa, and preferably at least 160 MPa.
14. Method according to any one of claims 1 to 13, wherein the hot-rolled feedstock and annealed plate at final gauge

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has an ultimate tensile strength of at least 310 MPa, and preferably at least 320 MPa.

15. Use of an aluminium alloy plate obtained by the method according to any one of claims 1 to 14 in a storage vessel, preferably the hull of a silo.

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EUROPEAN SEARCH REPORT

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Place of search Munich		Date of completion of the search 20 June 2018	Examiner Radeck, Stephanie
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**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

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