



EUROPEAN PATENT APPLICATION
published in accordance with Art. 153(4) EPC

(43) Date of publication:

06.12.2023 Bulletin 2023/49

(21) Application number: **22771135.5**

(22) Date of filing: **03.03.2022**

(51) International Patent Classification (IPC):

C22C 21/10 ^(2006.01) **C22F 1/00** ^(2006.01)
C22F 1/053 ^(2006.01) **B21C 23/00** ^(2006.01)

(52) Cooperative Patent Classification (CPC):

B21C 23/00; C22C 21/10; C22F 1/00; C22F 1/053

(86) International application number:

PCT/JP2022/009204

(87) International publication number:

WO 2022/196381 (22.09.2022 Gazette 2022/38)

(84) Designated Contracting States:

**AL AT BE BG CH CY CZ DE DK EE ES FI FR GB
GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO
PL PT RO RS SE SI SK SM TR**

Designated Extension States:

BA ME

Designated Validation States:

KH MA MD TN

(30) Priority: **15.03.2021 JP 2021041235**

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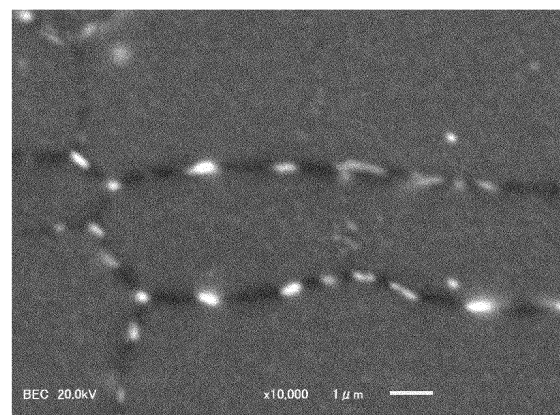
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(54) **HIGH-STRENGTH ALUMINUM ALLOY EXTRUDED MATERIAL AND MANUFACTURING METHOD THEREFOR**

(57) Disclosed is an aluminum alloy extruded material including: Zn: 7.5 to 9.2% by mass; Mg: 1.3 to 2.0% by mass; Cu: 0.1 to 0.7% by mass; one or more elements selected from the group consisting of Mn: 0.30% by mass or less, Cr: 0.25% by mass or less, and Zr: 0.25% by mass or less: totaling 0.1 to 0.5% by mass; and Ti: 0.005 to 0.20% by mass, with the balance being Al and inevitable impurities, wherein an average spacing of grain boundary precipitates is 0.8 to 1.4 μm , and an average particle length of the grain boundary precipitates is 0.3 to 0.5 μm , and a proof stress is 440 N/mm² or more.

Fig. 1



Description

Technical Field

5 **[0001]** The present disclosure relates to a high-strength aluminum alloy extruded material, and a manufacturing method therefor.

Background Art

10 **[0002]** High-strength 7000 series aluminum alloys have a problem of cracks occurring at an area where tensile stress is continuously applied under corrosive environment, namely, stress corrosion cracking (SCC). SCC is strongly avoided because the crack progresses quickly once it occurs, leading to fracture in a short time. In general, see is more likely to occur in higher-strength material. Due to the problem of SCC, the use of 7000 series aluminum alloys is sometimes deferred.

15 **[0003]** For this reason, attempts have been conventionally made to improve SCC resistance.

[0004] Patent Document 1 discloses a 7000 series aluminum alloy extruded material in which when a Zn content expressed in % by mass is [Zn] and a Mg content is [Mg], $5 \leq [\text{Zn}] \leq 7$ and $[\text{Zn}] + 4.7[\text{Mg}] \leq 14$ are satisfied and the Mg content is in excess of the stoichiometric ratio of MgZn_2 . In addition to Zn and Mg in the above ranges, this aluminum alloy extruded material includes Cu: 0.1 to 0.6% by mass, Ti: 0.005 to 0.05% by mass, and also one or more of Mn: 0.1 to 0.3% by mass, Cr: 0.05 to 0.2% by mass, and Zr: 0.05 to 0.2% by mass. This aluminum alloy extruded material is manufactured by die quenching with air cooling (online forced cooling of the extruded material using a die immediately after extrusion, also called press quenching). It exhibits high strength and excellent SCC resistance after being subjected to aging treatment, and thus can be suitably used as a material for automotive components such as door beams and bumper reinforcements.

25 **[0005]** Patent Document 2 mentions a 7000 series aluminum alloy extruded material, including Zn: 5.5 to 9.0% by mass, Mg: 1.0 to 2.0% by mass, Cu: 0.1 to 1.0% by mass, Ti: 0.005 to 0.2% by mass, and 0.1 to 0.5% by mass of one or more of Zr, Cr, Mn, etc. This aluminum alloy extruded material is regulated such that an average length of grain boundary precipitates (MgZn_2) is $5 \mu\text{m}$ or less, and that the number of grain boundary precipitates of more than $5 \mu\text{m}$ in length is 3 or less per $100 \mu\text{m}$ of the length of the grain boundary. Such an aluminum alloy extruded material is manufactured by die quenching with water cooling. It has high strength and excellent energy absorption properties after aging treatment and thus can be suitably used as a material for automotive components such as door beams and bumper reinforcements. Patent Document 2 also mentions that Zr, Cr, Mn, etc. serve to form the crystalline microstructure of the 7000 series aluminum alloy extruded material into a fibrous microstructure and to improve see resistance.

35 Conventional Art Document

Patent Document

[0006]

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Patent Document 1: JP 2011-144396 A

Patent Document 2: JP 2015-221924 A

Disclosure of the Invention

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Problems to be Solved by the Invention

[0007] The SCC resistance requirements for aluminum alloy extruded materials have been more stringent in recent years. Therefore, even the 7000 series aluminum alloy extruded materials mentioned in Cited References 1 and 2 may not meet the SCC resistance requirements, and a high-strength 7000 series aluminum alloy extruded material with higher SCC resistance is required.

[0008] The present disclosure has been made in order to meet such requirements, and an object thereof is to provide a 7000 series aluminum alloy extruded material with high strength and high SCC resistance as well as a manufacturing method therefor.

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Means for Solving the Problems

[0009] According to a first aspect of the present invention, there is provided an aluminum alloy extruded material

including:

Zn: 7.5 to 9.2% by mass;
Mg: 1.3 to 2.0% by mass;
Cu: 0.1 to 0.7% by mass;

one or more elements selected from the group consisting of Mn: 0.30% by mass or less, Cr: 0.25% by mass or less, and Zr: 0.25% by mass or less: totaling 0.1 to 0.5% by mass; and Ti: 0.005 to 0.20% by mass, with the balance being Al and inevitable impurities, wherein an average spacing of grain boundary precipitates is 0.8 to 1.4 μm , and an average particle length of the grain boundary precipitates is 0.3 to 0.5 μm , and a proof stress is 440 N/mm² or more.

[0010] In a second aspect of the present invention, there is provided the aluminum alloy extruded material according to the first aspect, wherein the average spacing of the grain boundary precipitates is 1.2 μm or less.

[0011] According to a third aspect of the present invention, there is provided a method for manufacturing an aluminum alloy extruded material, the method including the steps of: soaking an aluminum alloy including:

Zn: 7.5 to 9.2% by mass,
Mg: 1.3 to 2.0% by mass,
Cu: 0.1 to 0.7% by mass,

one or more elements selected from the group consisting of Mn: 0.30% by mass or less, Cr: 0.25% by mass or less, and Zr: 0.25% by mass or less: totaling 0.1 to 0.5% by mass, and Ti: 0.005 to 0.20% by mass, with the balance being Al and inevitable impurities; hot-extruding the aluminum alloy after the soaking; cooling the aluminum alloy between 400°C and 300°C at an average cooling rate of 100°C/minute or more and 600°C/minute or less during cooling after the extrusion; and applying artificial aging treatment to the aluminum alloy after the cooling.

[0012] In a fourth aspect of the present invention, there is provided the method for manufacturing an aluminum alloy extruded material according to the third aspect, wherein the cooling after the extrusion is performed by die quenching.

Effects of the Invention

[0013] In one embodiment of the present invention, it is possible to provide a 7000 series aluminum alloy extruded material with high strength and high SCC resistance, and a manufacturing method therefor.

Brief Description of the Drawings

[0014] FIG. 1 is an SEM (scanning electron microscope) photograph showing an example of the observation results of grain boundary precipitates in a 7000 series aluminum alloy extruded material according to the embodiment of the present invention.

Mode for Carrying Out the Invention

[0015] The present inventors have studied from various angles. As a result, they have found that a 7000 series aluminum alloy extruded material with the predetermined compositions is capable of obtaining high SCC resistance even when the proof stress is as high as 440 N/mm² or more by setting an average spacing of grain boundary precipitates to 0.8 to 1.4 μm and an average particle length of the grain boundary precipitates to 0.3 to 0.5 μm .

[0016] They have also found that such an aluminum alloy extruded material can be manufactured using an aluminum alloy having a predetermined composition by a method, including: (a) soaking an aluminum alloy; (b) hot-extruding the aluminum alloy after the soaking; (c) cooling the aluminum alloy between 400°C and 300°C at an average cooling rate of 100°C/minute or more and 600°C/minute or less during cooling after the extrusion; and (d) applying artificial aging treatment to the aluminum alloy after the cooling.

[0017] Hereinafter, embodiments of the present invention will be described in detail.

<1. Aluminum alloy composition>

[0018] A 7000 series aluminum alloy extruded material according to the embodiment of the present invention includes Zn: 7.5 to 9.2% by mass, Mg: 1.3 to 2.0% by mass, Cu: 0.1 to 0.7% by mass, one or more elements selected from the group consisting of Mn: 0.30% by mass or less, Cr: 0.25% by mass or less, and Zr: 0.25% by mass or less: totaling 0.1 to 0.5% by mass, and Ti: 0.005 to 0.20% by mass.

[0019] Hereinafter, each element will be described in detail.

(Zn: 7.5 to 9.2% by mass)

[0020] Zn forms $MgZn_2$ together with Mg and improves the strength of a 7000 series aluminum alloy extruded material. In order to increase the strength, as typified by proof stress (0.2% proof stress), of the 7000 series aluminum alloy extruded material after aging treatment (artificial aging treatment), the Zn content needs to be 7.5% by mass or more. On the other hand, if the Zn content exceeds 9.2% by mass, the material strength is improved, but the average spacing of grain boundary precipitates ($MgZn_2$) tends to become smaller, which may degrade SCC resistance.

[0021] Therefore, the Zn content is set within the range of 7.5 to 9.2% by mass to obtain the predetermined strength while ensuring SCC resistance. The lower limit of the Zn content is preferably 7.7% by mass, more preferably 8.0% by mass, and still more preferably 8.1% by mass, while the upper limit thereof is preferably 9.0% by mass, and more preferably 8.8% by mass.

(Mg: 1.3 to 2.0% by mass)

[0022] Mg forms $MgZn_2$ together with Zn and improves the strength of a 7000 series aluminum alloy extruded material. In order to increase the strength, as typified by proof stress, of the 7000 series aluminum alloy extruded material after aging treatment (artificial aging treatment), the Mg content needs to be 1.3% by mass or more. On the other hand, if the Mg content exceeds 2.0% by mass, the average spacing of grain boundary precipitates ($MgZn_2$) tends to become smaller, which may degrade SCC resistance. Furthermore, deformation resistance increases, also degrading the extrudability of the aluminum alloy. Therefore, the Mg content is set within the range of 1.3 to 2.0% by mass. The lower limit of the Mg content is preferably 1.4% by mass, while the upper limit thereof is preferably 1.8% by mass.

(Cu: 0.1 to 0.7% by mass)

[0023] Cu is solid soluble in grain boundary precipitates ($MgZn_2$) to reduce a potential difference between the grain boundary precipitates and PFZs (precipitate-free-zones) and suppresses preferential dissolution of the grain boundary precipitates under corrosive environment, thereby improving see resistance of the 7000 series aluminum alloy extruded material. However, that effect is small if the Cu content is less than 0.1% by mass. On the other hand, if the Cu content exceeds 0.7% by mass, the deformation resistance increases, degrading the extrudability and also causing the weld-crack resistance of the extruded material to deteriorate. Therefore, the Cu content is set at 0.1 to 0.7% by mass. The lower limit of the Cu content is preferably 0.2% by mass, while the upper limit thereof is preferably 0.5% by mass.

(One or more elements selected from the group consisting of Mn: 0.30% by mass or less, Cr: 0.25% by mass or less, and Zr: 0.25% by mass or less: totaling 0.1 to 0.5% by mass)

[0024] Mn, Cr, and Zr precipitate finely in an aluminum alloy during soaking and serve to pin the grain boundaries to suppress recrystallization, thereby refining the crystal grains of the 7000 series aluminum alloy extruded material to form them into a fibrous microstructure. Further, these elements have the effect of improving SCC resistance of the 7000 series aluminum alloy extruded material by refining the crystal grains. It is thought that one or more of Mn, Cr, and Zr include (1) only one of the three elements, (2) a combination of two of the three elements (Mn and Cr, Mn and Zr or Cr and Zr), or (3) all three elements, and any of the above (1) through (3) may be selected.

[0025] However, if the contents of Mn, Cr, and Zr exceed 0.3% by mass, 0.25% by mass, and 0.25% by mass, respectively, or if the total content thereof exceeds 0.5% by mass, the extrudability deteriorates and the hardening sensitivity of the extruded material is enhanced. On the other hand, if the total content of Mn, Cr, and Zr is less than 0.1% by mass, the desired effect may not be obtained. Therefore, the contents of Mn, Cr, and Zr are set within the range of 0.3% by mass or less for Mn, 0.25% by mass or less for Cr, and 0.25% by mass or less for Zr, respectively, and the total content of Mn, Cr, and Zr are set within the range of 0.1 to 0.5% by mass.

[0026] Among these, Zr is preferentially added over Mn and Cr in the range of 0.1 to 0.25% by mass because Zr has

less effect on enhancing the hardening sensitivity of the 7000 series aluminum alloy extruded material compared to Mn and Cr, and one or both of Mn and Cr are preferably added supplementarily as needed. The preferred lower limit of the Zr content is 0.12% by mass, and the more preferred lower limit thereof is 0.14% by mass. The preferred upper limit thereof is 0.23% by mass, and the more preferred upper limit thereof is 0.20% by mass. The preferred upper limit of the Cr content is 0.1% by mass, and the more preferred upper limit thereof is 0.06% by mass. The preferred upper limit of the Mn content is 0.1% by mass, and the more preferred upper limit thereof is 0.06% by mass.

(Ti: 0.005 to 0.20% by mass)

[0027] Ti has the effect of forming Al_3Ti in the molten metal and refining crystal grains of an ingot. However, the effect is small if the Ti content is less than 0.005% by mass. On the other hand, if the Ti content exceeds 0.20% by mass, coarse crystallites are formed in the ingot, reducing the toughness of the 7000 series aluminum alloy extruded material. Therefore, the Ti content is set at 0.005 to 0.20% by mass. Preferably, the lower limit of the Ti content is 0.01% by mass, and the upper limit thereof is 0.05% by mass.

[0028] The basic components are as mentioned above, and in one of the preferred embodiments of the present invention, the balance is Al and inevitable impurities.

[0029] Fe and Si are major inevitable impurities in the 7000 series aluminum alloy extruded material. If the Fe content is extremely large, various properties such as elongation and fatigue strength of the 7000 series aluminum alloy extruded material are reduced, and thus the Fe content is preferably restricted to, for example, 0.30% by mass or less. If the Si content is extremely large, various properties such as elongation and fatigue strength of the 7000 series aluminum alloy extruded material are reduced, and seizure is more likely to occur in extrusion. Thus, the Si content is preferably restricted to, for example, 0.15% by mass or less.

[0030] Regarding the inevitable impurities other than Fe and Si, for example, the content of each element of these inevitable impurities is restricted to, for example, 0.05% by mass or less, and the total content of the inevitable impurities, other than Fe and Si, is restricted to, for example, 0.15% by mass or less; these restricted contents are allowable ranges of normal inevitable impurities of the 7000 series aluminum alloy extruded material. As for B among the impurities, it is mixed in the aluminum alloy in an amount of about 1/5 of the Ti content along with the addition of Ti, but its content is preferably 0.02% by mass or less, and more preferably 0.01% by mass or less.

(Other selective elements)

[0031] Furthermore, in another preferred embodiment of the present invention, elements other than those mentioned above may be added as necessary to the extent that they do not impair the action according to the embodiment of the present invention. The properties of the aluminum alloy can be further improved according to the components contained.

[0032] Examples of such selective elements include one or more elements selected from the group consisting of Sc, Sr, Sn, Ag, Ca and Mo. The preferred content of each of these elements and the reasons for their contents are mentioned below.

Sc: 0.05 to 0.5% by mass

[0033] The effect of refining crystal grains can be obtained by containing Sc within this range.

Sr: 0.05 to 0.5% by mass

[0034] The effect of improving mechanical properties can be obtained by containing Sr within this range.

Sn: 0.05 to 0.5% by mass

[0035] The effect of improving mechanical properties can be obtained by containing Sn within this range.

Ag: 0.05 to 0.5% by mass

[0036] The effect of improving mechanical properties and SCC resistance can be obtained by containing Ag within this range.

Ca: 0.05 to 0.5% by mass

[0037] The effect of improving mechanical properties and SCC resistance can be obtained by containing Ca within

this range.

Mo: 0.05 to 0.5% by mass

- 5 **[0038]** The effect of improving mechanical properties and SCC resistance can be obtained by containing Mo within this range.

<2. Average spacing and average particle length of grain boundary precipitates>

- 10 **[0039]** When a 7000 series aluminum alloy is hot-extruded and then cooled by die quenching, etc., precipitates (MgZn_2) are generated in the crystal grains and at the grain boundaries during cooling, depending on the Zn and Mg contents and the cooling rate. During cooling, MgZn_2 precipitating at the grain boundaries are larger in size than MgZn_2 precipitates in the crystal grains.

- 15 **[0040]** However, if the cooling rate is sufficiently high (e.g., in water or mist cooling), precipitation during cooling can be suppressed. Subsequently, when the 7000 series aluminum alloy extruded material (temper designation: T1) obtained after the die quenching is subjected to aging treatment (artificial aging treatment) into the aluminum alloy (temper designation: T5), Zn and Mg in solid solution in the aluminum alloy precipitate finely in the form of MgZn_2 in the crystal grains and at the grain boundaries. In this aging treatment, the size and distribution form of MgZn_2 having precipitated during the cooling by the die quenching do not change significantly.

- 20 **[0041]** It is noted that the T1 temper designation means the state of the material subjected to natural aging after the die quenching, and the T5 temper designation means the state of the material subjected to the subsequent aging treatment.

- 25 **[0042]** When the 7000 series aluminum alloy extruded material is placed under corrosive environment and MgZn_2 dissolves, while at the same time continuous tensile stress is present in the material, cracking propagates in the material mainly through the grain boundaries, and this cracking is observed as SCC. This is because fine grain boundary precipitates (MgZn_2) at the level of 1 μm or less in length that have precipitated during the cooling by die quenching and during the aging treatment dissolve to form holes, which serve as transmission paths of cracking.

- [0043]** see is more likely to occur as grain boundary precipitates (MgZn_2) are distributed more densely along the grain boundaries (as the average spacing between the adjacent grain boundary precipitates becomes smaller).

- 30 **[0044]** On the other hand, as the average spacing of the grain boundary precipitates is increased, the size (average particle length) increases, and sufficient strength (proof stress) cannot be obtained even when artificial aging treatment is performed. The inventors of this application have found that both high strength and high SCC resistance can be achieved by setting the average spacing and average particle length of the grain boundary precipitates within the respective appropriate ranges.

- 35 **[0045]** In the embodiment of the present invention, the grain boundary precipitates of the 7000 series aluminum alloy extruded material have an average spacing of 0.8 to 1.4 μm and an average particle length of 0.3 to 0.5 μm . The average spacing of the grain boundary precipitates becomes smaller as the total content of Zn and Mg increases. The average spacing of the grain boundary precipitates becomes smaller as the cooling rate by die quenching, etc. increases. As the average spacing of grain boundary precipitates becomes smaller, the size (average particle length) of the grain boundary precipitates tends to become smaller. Thus, both the average spacing and average particle length of the grain boundary precipitates can be controlled within the appropriate ranges by setting the Zn and Mg contents within the appropriate ranges as mentioned above and controlling the cooling rate after the hot extrusion to be within the appropriate range as mentioned later.

- 40 **[0046]** In one preferred embodiment, the upper limit of the average spacing of the grain boundary precipitates is 1.2 μm or less. This allows for higher strength while maintaining high SCC resistance.

- [0047]** The "particle length of the grain boundary precipitates" means the length along the grain boundary of the grain boundary precipitate.

- [0048]** The average spacing and average particle length of the grain boundary precipitate can be determined by SEM observation as described in detail in examples mentioned later.

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<3. Proof stress>

- [0049]** As mentioned above, the average particle length of the grain boundary precipitates can be controlled not to become extremely large, thereby achieving high strength. In the 7000 series aluminum alloy extruded material according to the embodiment of the present invention, the proof stress is 440 N/mm^2 or more.

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<4. Manufacturing method>

[0050] The 7000 series aluminum alloy extruded material according to the embodiment of the present invention can be manufactured using an aluminum alloy having a predetermined composition by a method, including: (a) soaking an aluminum alloy; (b) hot-extruding the aluminum alloy after the soaking; (c) cooling the aluminum alloy between 400°C and 300°C at an average cooling rate of 100°C/minute or more and 600°C/minute or less during cooling after the extrusion; and (d) applying artificial aging treatment to the aluminum alloy after the cooling.

[0051] Hereinafter, the manufacturing method according to the embodiment of the present invention will be described in detail.

(a) Homogenization treatment

[0052] The 7000 series aluminum alloy having the above predetermined composition is soaked. Examples of forms of the 7000 series aluminum alloys which are soaked can include ingots and billets. The conditions for the homogenization treatment may be selected from any conditions that enables hot extrusion.

[0053] In a preferred embodiment that can reduce the average Cu content in a Fe-based crystallite, means for improving see resistance includes subjecting the 7000 series aluminum alloy to the homogenization treatment under conditions such as at high temperature and for a long time. By subjecting the aluminum alloy to the homogenization treatment at a temperature between 490°C and 550°C for 4 hours or more, Cu present in a high concentration in the Fe-based crystallite is diffused into the Al matrix, resulting in a decrease in the Cu content in the Fe-based crystallite. To reduce the average Cu content in the Fe-based crystallite, a higher homogenization treatment temperature is preferable. However, if the homogenization treatment temperature is too high, the pinning action of the recrystallization inhibiting elements (Zr, Cr, Mn) at the grain boundaries may be degraded, resulting in coarsening of the extruded material microstructure. Therefore, a homogenization treatment temperature is preferably within the range of 500 to 540°C, and more preferably within the range of 510 to 530°C. A longer homogenization treatment time is preferable from the viewpoint of reducing the average Cu content in the Fe-based crystallite, but an extremely long time may cause coarsening of the extruded material microstructure. Therefore, a homogenization treatment time is preferably 10 hours or less. To sufficiently reduce the Cu content in the Fe-based crystallite, the higher temperature conditions mentioned above are preferred over the homogenization treatment conditions (470°C × 6 hours) that are commonly used for 7000 series aluminum alloys in the conventional art. Cooling after the homogenization treatment is not particularly limited, but may be performed at a cooling rate in the range of 100 to 200°C/hour, for example.

[0054] The homogenization treatment time mentioned here means a holding time at this temperature.

(b) Hot extrusion

[0055] The 7000 series aluminum alloy after the homogenization treatment, for example, in the form of billets or ingots, is hot-extruded.

[0056] The conditions for the hot extrusion may be any conditions that allows for processing the aluminum alloy into a desired shape.

[0057] Examples of preferred extrusion conditions include a billet temperature (extrusion temperature) of 450 to 510°C and an extrusion speed of 2 to 15 m/min.

[0058] Heating for hot extrusion may be performed by reheating the 7000 series aluminum alloy that has been cooled after the homogenization treatment.

(c) Cooling after extrusion

[0059] The extruded 7000 series aluminum alloy is cooled. The cooling may be performed immediately after the extrusion is performed, or after the aluminum alloy is held at a predetermined temperature (e.g., re-solution treatment or the like) after the extrusion. This cooling may be performed by any method, but is performed between 400°C and 300°C, which is the temperature range at which MgZn₂ is most likely to precipitate, at an average rate of 100°C/minute or more and 600°C/minute or less. Thus, the obtained 7000 series aluminum alloy can have an average spacing of grain boundary precipitates of 0.8 to 1.4 μm and an average particle length of 0.3 to 0.5 μm. If the average cooling rate is less than 100°C/min, the amount of MgZn₂ precipitated during cooling becomes large, and the effect of the subsequent aging treatment becomes insufficient, whereby the proof stress cannot be sufficiently improved. On the other hand, if the average cooling rate exceeds 600°C/min, many fine grain boundary precipitates are formed, and the average spacing of the grain boundary precipitates becomes extremely small, which degrades see resistance.

[0060] The average cooling rate between 400°C and 300°C is preferably 100°C/min to 500°C/min, and more preferably 100°C/min to 400°C/min.

[0061] Preferably, the average cooling rate between 400°C and 200°C is also 100°C/min or more and 600°C/min or less.

[0062] A preferred cooling method includes die quenching. Cooling of a die during die quenching can be any method, such as water cooling, air cooling, or natural cooling. Air cooling is preferred because it can achieve an average cooling rate of 100 to 600°C/minute between 400°C and 300°C with relative ease.

[0063] The cooling rate may be measured by contacting the aluminum alloy extruded material with a contact-type thermometer such as a thermocouple. As a simple method, a non-contact thermometer may be used to measure the surface temperature of the aluminum alloy extruded material. If it is further difficult to measure the temperature, simulation may be used as appropriate to determine the temperature.

(d) Artificial aging treatment

[0064] The artificial aging treatment is applied to the 7000 series aluminum alloy extruded material that has been cooled after the hot extrusion. This treatment can increase the proof stress to 440 N/mm² or more. The conditions for the artificial aging treatment may be any conditions as long as the proof stress can be increased to 440 N/mm² or more.

[0065] As preferred conditions for the artificial aging treatment, two-step aging treatment can be exemplified, which involves holding at a temperature of 65 to 95°C for 2 to 6 hours, followed by holding at a temperature of 120 to 170°C for 6 to 15 hours.

[0066] While the method for manufacturing a 7000 series aluminum alloy extruded material according to the embodiment of the present invention has been described above, a person skilled in the art who understands the desired properties of the 7000 series aluminum alloy extruded material according to the embodiment of the present invention may have found, by trial and error, another method for manufacturing a 7000 series aluminum alloy extruded material according to the embodiment of the present invention having the desired properties, other than the above manufacturing method.

Examples

[0067] Hereinafter, the present invention will be described more specifically with reference to Examples. The present invention is not limited by the following Examples, but may be implemented with modifications as appropriate to the extent that the modifications can conform to the above-mentioned and following purposes, and all of these modifications are included in the scope of the present invention.

[0068] Samples Nos. 1 to 6 were obtained by a method described below.

[0069] 7000 series aluminum alloy billets of 194 mm in diameter obtained by semi-continuous casting were subjected to homogenization treatment at 520°C for 6 hours and then cooled to room temperature. The cooling method was air cooling with a fan. Table 1 shows alloy compositions of the samples Nos. 1 to 6.

[0070] Subsequently, these billet samples were reheated and hot-extruded at an extrusion temperature of 500°C and an extrusion speed of 5 m/min, followed by air cooling with a fan immediately after the extrusion. The cross-sectional shape of each extruded material is a hollow extruded material with 15 mm high × 120 mm wide × 3 mm thick. Each extruded material sample was held at 470°C for 1 hour for re-solution treatment. Then, by simulating die quenching, the samples were cooled to room temperature. Specifically, the samples Nos. 1 to 4 were cooled by air cooling with a fan, the sample No. 5 by natural cooling, and the sample No. 6 by water cooling (shower cooling).

[0071] Table 1 shows the average cooling rates of the extruded materials between 400°C and 300°C. The cooling rate was measured by a thermocouple inserted into a hole made in the extruded material sample. The time (t minutes) taken for the extruded material sample to reach 300°C from 400°C was determined, and the cooling rate was calculated as (400-300)/t (°C/min). The calculated cooling rate is based on the temperature of the inside of the extruded material, but the temperature difference between the surface and inside of the extruded material is small because of the excellent thermal conductivity of the aluminum alloy, and thus it can be measured at either location.

[Table 1]

Sample No.	Chemical composition (% by mass)										Average cooling rate between 400°C and 300°C (°C/min)
	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Zr	Al	
1	0.05	0.10	0.29	0.00	1.64	0.01	7.63	0.02	0.17	Balance	187
2	0.05	0.11	0.30	0.00	1.60	0.00	8.59	0.02	0.15	Balance	235
3	0.05	0.10	0.31	0.00	1.68	0.01	9.15	0.02	0.17	Balance	184

(continued)

Sample No.	Chemical composition (% by mass)										Average cooling rate between 400°C and 300°C (°C/min)
	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Zr	Al	
4	0.05	0.09	0.28	0.00	1.49	0.00	6.51	0.02	0.15	Balance	114
5	0.05	0.11	0.30	0.00	1.60	0.00	8.59	0.02	0.15	Balance	62
6	0.05	0.11	0.30	0.00	1.60	0.00	8.59	0.02	0.15	Balance	716
* indicates that the requirements specified by the embodiment of the present invention were not satisfied.											

[0072] The two-stage artificial aging treatment (first stage: 90°C × 3 hours → second stage: 140°C × 8 hours) was applied to the extruded material samples after the cooling.

[0073] The extruded material samples after the artificial aging treatment were used to measure the average spacing and average particle length of the grain boundary precipitates using the method detailed below. In addition, as an evaluation of the properties of these samples, the proof stress and SCC critical stress were measured by the method detailed below. The measurement results are shown in Table 2.

[Table 2]

Sample No.	Grain boundary precipitate		Evaluation results of properties	
	Average spacing (μm)	Average particle length (μm)	Proof stress (N/mm ²)	see critical stress (N/mm ²)
1	1.04	0.46	481	120
2	1.11	0.40	487	120
3	0.95	0.45	498	120
4	1.21	0.43	434	140 or more
5	1.94	0.65	409	180 or more
6	0.62*	0.28	510	80
* indicates that the requirements specified by the embodiment of the present invention were not satisfied or that the evaluation result of the properties were not good.				

(Average particle spacing and average particle length of grain boundary precipitate)

[0074] A specimen was cut from the top surface of the extruded material sample, and the precipitation form of precipitates (MgZn₂) present at the grain boundaries was observed by scanning an area at a distance of 100 μm from the extruded material surface on a surface perpendicular to the extrusion direction with an electron microscopy (SEM).

[0075] More specifically, each sample was observed, and a grain boundary area that was considered representative was selected. The particle lengths of the grain boundary precipitates that existed at the grain boundaries observed in the field of view of that area (field of view: 12.7 μm × 9.6 μm) were measured. The sum of the particle lengths of the measured grain boundary precipitates (length in the direction along the grain boundary) was divided by the number of grain boundary precipitates, and the resulting value was defined as the average particle length of the grain boundary precipitates. A value obtained by subtracting the total particle length from the length of the grain boundaries (total extension of the grain boundaries present in the measured range) was divided by the number of grain boundary precipitates. The resulting value was determined as the average spacing of the grain boundary precipitates.

[0076] FIG. 1 is an SEM photograph of the sample No. 3, which was an example of the observation results of the grain boundary precipitates. It can be seen that white grain boundary precipitates (MgZn₂) are formed along the grain boundaries.

(Proof stress)

[0077] A test piece in conformity with JIS13B was taken from the top surface of each extruded material sample in a

direction parallel to the extrusion direction by machining. Two test pieces were taken from each extruded material sample. The test specimens were subjected to a tensile test in conformity with the specifications of JISZ2241 to thereby measure the proof stress (0.2% proof stress). The crosshead speed was set at 5.0 mm/min until the proof stress value was reached, and then at 10.0 mm/min. The proof stress value for each of Nos. 1 to 6 listed in Table 1 is the average value of the proof stress values measured on two test pieces. The proof stress value of 440 N/mm² or more was evaluated as acceptable.

(SCC critical stress)

[0078] SCC testing was performed using a chromic acid promotion method. An SCC test piece of 10 mm wide × 50 mm long was taken from the top surface of the extruded material sample in a direction perpendicular to the extrusion direction by machining while avoiding a weld zone. For each sample, two test pieces were taken at each loading stress. The SCC testing was performed adopting the three-point loading method of the plate bending test (JISH8711:2001) to apply various tensile stresses. The applied load was given by a constant strain method (three-point support beam method). In more detail, tensile stress was generated on the outer surface of the test piece by tightening bolts of three-point bending jigs, and the tensile stress value was measured by a strain gauge bonded to the outer surface of the test piece.

[0079] A corrosion solution used for the SCC testing was a Cr acid solution (NaCl: 3 g, K₂Cr₂O₇: 30 g, and CrO₃: 36 g per liter of distilled water), and the temperature was maintained at 90°C or higher during the testing in order to promote SCC. The test pieces (two pieces for each applied stress) were immersed in the corrosion solution while the stress was being applied thereto, taken out therefrom every 2 hours, and visually observed regarding the presence or absence of occurrence of cracks. The test piece without occurrence of cracks was re-immersed in the solution. This procedure was repeatedly performed for 16 hours after the start of the SCC testing. The maximum applied stress at which both test pieces remained free of cracks until the end of the testing was evaluated as the SCC critical stress of the test pieces. The SCC critical stress of 100 N/mm² or more was evaluated as acceptable.

[0080] As shown in Tables 1 and 2, samples Nos. 1 to 3, which had the composition specified by the embodiment of the present invention and in which the average cooling rate of the extruded material between 400°C and 300°C was in the range of 100 to 600°C/min, had an average spacing of the grain boundary precipitates in the range of 0.8 to 1.4 μm and an average particle length in the range of 0.3 to 0.5 μm. Furthermore, the samples Nos. 1 to 3 had a proof stress of 440 N/mm² or more and an SCC critical stress of 100 N/mm² or more.

[0081] In contrast, samples Nos. 4 to 6, which fell outside the composition range specified by the embodiment of the present invention or in which the average cooling rate after hot extrusion was outside the range of 100 to 600°C/min, had a proof stress of less than 440 N/mm² or a SCC critical stress of less than 100 N/mm².

[0082] More specifically, the sample No. 4 had a low proof stress due to insufficient Zn content.

[0083] The sample No. 5 had a higher SCC critical stress because the average spacing and average particle length of grain boundary precipitates were larger than the range specified by the embodiment of the present invention, but it had a low proof stress because the cooling rate was too low.

[0084] The sample No. 6 had a too high average cooling rate, thereby making the average spacing of the grain boundary precipitates too small and also the average particle length thereof too small, resulting in a low SCC critical stress.

[0085] This application claims priority based on Japanese Patent Application No. 2021-41235 filed on March 15, 2021, the disclosure of which is incorporated by reference herein.

Claims

1. An aluminum alloy extruded material comprising:

Zn:	7.5 to 9.2% by mass;
Mg:	1.3 to 2.0% by mass;
Cu:	0.1 to 0.7% by mass;

one or more elements selected from the group consisting of Mn: 0.30% by mass or less, Cr: 0.25% by mass or less, and Zr: 0.25% by mass or less: totaling 0.1 to 0.5% by mass; and Ti: 0.005 to 0.20% by mass, with the balance being Al and inevitable impurities, wherein an average spacing of grain boundary precipitates is 0.8 to 1.4 μm, and an average particle length of the grain boundary precipitates is 0.3 to 0.5 μm, and a proof stress is 440 N/mm² or more.

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2. The aluminum alloy extruded material according to claim 1, wherein the average spacing of the grain boundary precipitates is 1.2 μm or less.

3. A method for manufacturing an aluminum alloy extruded material, the method comprising the steps of:
soaking an aluminum alloy comprising:

Zn: 7.5 to 9.2% by mass,

Mg: 1.3 to 2.0% by mass,

Cu: 0.1 to 0.7% by mass,

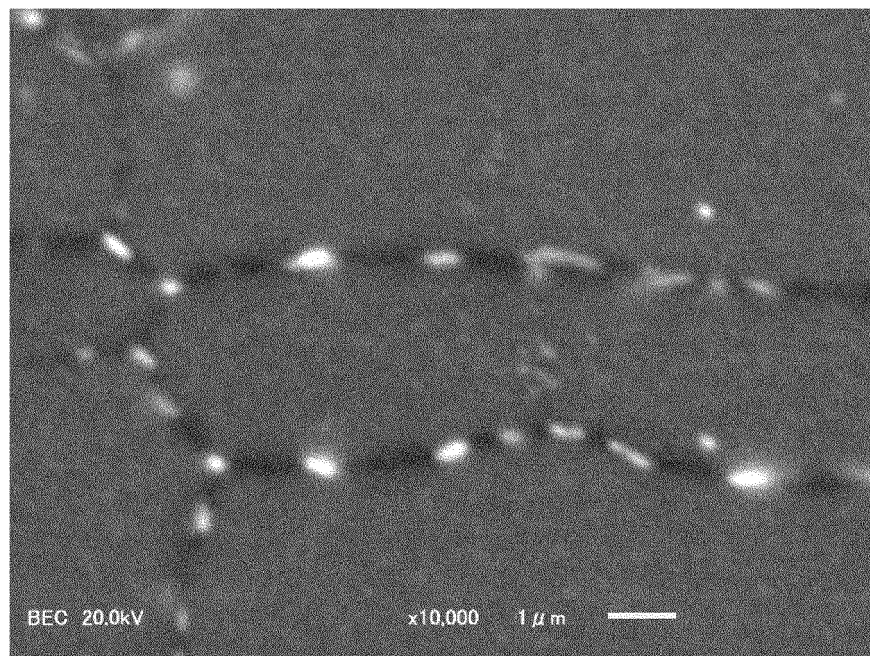
one or more elements selected from the group consisting of Mn: 0.30% by mass or less, Cr: 0.25% by mass or less, and Zr: 0.25% by mass or less: totaling 0.1 to 0.5% by mass,

and Ti: 0.005 to 0.20% by mass, with the balance being Al and inevitable impurities;

hot-extruding the aluminum alloy after the soaking; cooling the aluminum alloy between 400°C and 300°C at an average cooling rate of 100°C/minute or more and 600°C/minute or less during cooling after the extrusion; and applying artificial aging treatment to the aluminum alloy after the cooling.

4. The method for manufacturing an aluminum alloy extruded material according to claim 3, wherein the cooling after the extrusion is performed by die quenching.

Fig. 1



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2022/009204

A. CLASSIFICATION OF SUBJECT MATTER

C22C 21/10(2006.01)i; **C22F 1/00**(2006.01)i; **C22F 1/053**(2006.01)i; **B21C 23/00**(2006.01)i
 FI: C22C21/10; C22F1/053; B21C23/00 A; C22F1/00 602; C22F1/00 612; C22F1/00 630A; C22F1/00 640A; C22F1/00 682;
 C22F1/00 683; C22F1/00 684C; C22F1/00 692A; C22F1/00 692B; C22F1/00 691B; C22F1/00 691C; C22F1/00 694B;
 C22F1/00 694Z

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C22C21/10; C22F1/00; C22F1/053; B21C23/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Published examined utility model applications of Japan 1922-1996
 Published unexamined utility model applications of Japan 1971-2022
 Registered utility model specifications of Japan 1996-2022
 Published registered utility model applications of Japan 1994-2022

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2020-164980 A (KOBELITE LTD) 08 October 2020 (2020-10-08) paragraphs [0022]-[0027], [0029], table 1, alloy no. 2-3, 6	1-4
A	JP 2013-36107 A (SUMITOMO LIGHT METAL IND LTD) 21 February 2013 (2013-02-21) entire text	1-4

☐ Further documents are listed in the continuation of Box C.
 ☒ See patent family annex.

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E" earlier application or patent but published on or after the international filing date	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&" document member of the same patent family
"O" document referring to an oral disclosure, use, exhibition or other means	
"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search 13 April 2022	Date of mailing of the international search report 10 May 2022
Name and mailing address of the ISA/JP Japan Patent Office (ISA/JP) 3-4-3 Kasumigaseki, Chiyoda-ku, Tokyo 100-8915 Japan	Authorized officer Telephone No.

Form PCT/ISA/210 (second sheet) (January 2015)

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/JP2022/009204

5

Patent document cited in search report	Publication date (day/month/year)	Patent family member(s)	Publication date (day/month/year)
JP 2020-164980 A	08 October 2020	(Family: none)	
JP 2013-36107 A	21 February 2013	(Family: none)	

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REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- JP 2011144396 A [0006]
- JP 2015221924 A [0006]
- JP 2021041235 A [0085]