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(54) **ALUMINUM ALLOY SHEET EXCELLENT IN HIGH-SPEED SUPERPLASTIC FORMABILITY AND PROCESS OF FORMING THE SAME**

(57) An alloy containing 3.0 to 8.0 wt.% of Mg, 0.001 to 0.1 wt.% of Ti, and amounts of Fe and Si (as impurities) as small as 0.06 wt.% or less, the balance being Al and unavoidable impurities, wherein the number per square millimeter of grains of an Al-Fe-Si compound having a diameter of 1 μm or more is 2000 or less, the mean crystal grain diameter is 25 to 200 μm and the elongation is more than 380% as worked at 350 to 550°C and a strain rate of 10⁻² to 10⁰/s. This alloy may further contain a small amount of Cu, Mn and/or Cr and is formed at 350 to 550°C and a strain rate of 10⁻² to 10⁰/s. An aluminum alloy sheet with excellent high strain rate superplastic formability and therefore capable of being formed at high temperature and high speed is obtained and the use of this sheet shortens the forming time to improve productivity.

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Description**FIELD OF THE INVENTION**

5 This invention relates to an aluminum alloy sheet which has excellent high-speed superplastic formability, and more specifically, to an Al-Mg alloy sheet which enables superplastic forming at high strain rate of 10^{-2} to $10^0/s$; and also to a process for forming the same.

BACKGROUND OF THE INVENTION

10 Based on Al-Mg alloy systems, by using a technique of regulating recrystallization to obtain finer crystal grains, superplastic alloys having an elongation of several hundred percent in high temperature ranges such as that between 500 to 550°C have been developed and are being used in various applications. However, conventional Al-Mg superplastic alloys demonstrate the best elongation at a forming speed (i.e. strain rate) between 10^{-4} to $10^{-3}/s$, at which it
 15 takes 30 to 100 minutes, for example, to form an ordinary utensil. This is an unacceptably low productivity for a commercial manufacturing process. Superplastic alloys that can be formed at a much higher forming speed are therefore required.

For example, an aluminum alloy sheet containing 2.0 to 6.0% of Mg, 0.0001 to 0.01% of Be, and 0.001 to 0.15% of Ti, with Fe and Si as impurities being controlled each at 0.2% or less and the largest grain diameter of impurity-based
 20 intermetallic compounds limited to 10 μm or less is proposed in Japanese Patent Application Laid-Open No. 72030/1992. While such a product does show an elongation of 350% or more at a strain rate of $10^{-3}/s$ under a high-temperature deformation condition of 400°C, the elongation decreases as the forming speed increases and becomes insufficient at strain rates of $10^{-2}/s$ or higher.

Another aluminum alloy sheet, proposed in Japanese Patent Application Laid-Open No. 318145/1992, contains 2
 25 to 5% of Mg, 0.04 to 0.10% of Cu, as well as optional small quantities of certain transition elements, Cr, Zr, or Mn; with Si and Fe as impurities being controlled at 0.1% or less, and at 0.15% or less, respectively; while controlling the crystal grain diameter at 20 μm or less and maintaining the grain diameter and the cubic ratio of transition metal-based intermetallic compounds Within certain specific ranges. Such an alloy sheet also has a limited application range of strain rates in the order of $10^{-4}/s$, and is not suitable for high strain rate superplastic forming at a higher strain rate.

SUMMARY OF THE INVENTION

The present invention has been achieved as a result of diverse examination and exhaustive experiments concern-
 35 ing the relationships of superplastic formability with various alloy constituents and their quantitative combinations, in addition to those with impurity content and their distribution, as well as with crystal grain diameters of impurity-based intermetallic compounds, made in an attempt to overcome the aforementioned shortcomings of the Al-Mg superplastic aluminum alloy. In particular, the object of the present invention is to provide, by identifying a particular distribution and crystal grain diameter range for Al-Fe-Si compounds to be controlled based on restriction of Fe and Si as impurities, an aluminum alloy sheet that has excellent high strain rate superplastic formability with sufficient elongation in a forming
 40 process with a high forming speed such as at a strain rate ranging from 10^{-2} to $10^0/s$, as well as to provide a processing method for forming such an aluminum alloy sheet.

To achieve this object, the aluminum alloy sheet with excellent high strain rate superplastic formability in the present invention comprises 3.0 to 8.0% (% by weight, hereinafter the same) Mg, 0.001 to 0.1% Ti, small amounts of Fe and Si
 45 (as impurities), each 0.06% or less, the balance being Al and unavoidable impurities, wherein the number per square millimeter of grains of an Al-Fe-Si compound existing in the matrix structure of said alloy and having a diameter of 1 μm or above is 2000 or less, the mean crystal grain diameter is 25 to 200 μm and the elongation is 350% or more as worked at 350 to 550°C and a strain rate of 10^{-2} to $10^0/s$, all of the foregoing constituting the basic features of the invention.

As the second and the third features constituting the invention, this alloy may further comprise 0.05 to 0.50% of Cu
 50 in addition to Mg and Ti as described above; or may comprise either one or both of Mn or Cr not exceeding 0.10% each in addition to Mg and Ti as described above, or alternatively, in addition to Mg, Ti, and Cu as described herein.

The processing for fabricating the aluminum alloy sheet with excellent high-speed superplastic formability in the present invention is characterized by working an aluminum alloy sheet prepared in accordance with the invention at 350
 to 550°C and a strain rate of 10^{-2} to $10^0/s$.

DETAILED DESCRIPTION OF THE INVENTION AND PREFERRED EMBODIMENTS

Referring to the significance of the alloy constituents described in this invention and the basis of their stated limits,
 first of all, Mg acts to recrystallize the alloy during the high-temperature deformation. The preferred content range is

between 3.0 and 8.0%, below which the effect on promoting the recrystallization is insufficient while a content in excess of 8.0% acts to reduce hot workability of the material. Cu on the other hand acts to improve the superplastic elongation of the Al-Mg alloy system. The preferred content range is between 0.05 to 0.50%, where a content below 0.05% fails to give sufficient elongation while a content in excess of 0.50% acts to reduce the hot workability.

5 Ti acts to turn the ingot crystals into finer grains and to provide the alloy with a better superplastic formability. The preferred content range is between 0.001 to 0.1%, where a content below 0.001% will fail to give the expected effect and a content in excess of 0.1% will yield coarse compounds that hinder workability as well as ductility. Further, Mn and Cr act to make recrystallized grains finer in the alloy recrystallization process that occurs during high-temperature deformation. The preferred content range is below 0.10% for each, where a content in excess of 0.10% will act to increase
10 a constituent particle whose grain diameter is 1 μm or above to decrease the superplastic formability of the alloy.

In the present invention, it is essential to limit Fe and Si as impurities each at 0.06% or less. These impurities form an Al-Fe-Si compound that is insoluble and prone to precipitate along the grain boundary, increasing cavities and thereby impairing the superplastic elongation. Preferably, the Fe and Si should each be controlled at 0.05% or less. It is also noted here that up to 50 ppm of Be maybe added to prevent oxidation of the molten metal, just as in the case of
15 ordinary Al-Mg alloys.

Referring further to the alloy structure of the present invention, since the Al-Fe-Si compound present in the alloy matrix gives rise to the above mentioned problem, it is better to allow as little of such a compound as possible, and, in particular, the limit in terms of number per square millimeter of an Al-Fe-Si compound having a grain diameter of 1 μm or more should be 2000 or less, since particles in excess of 2000 per square millimeter will increase cavities and
20 thereby impair the superplastic elongation.

It is essential to regulate the original mean crystal grain diameter of the aluminum alloy sheet within a range of 25 to 200 μm . If the original mean crystal grain diameter is below 25 μm , the original crystal grains will be recreated when recrystallization occurs during high temperature deformation, making it difficult to obtain a recrystallized structure with clean crystal grains as a result of a recrystallization process to obliterate the grain boundary with precipitation of the
25 aforementioned insoluble compounds. If the original mean crystal grain diameter exceeds 200 μm , the shearing deformation within the crystal grains becomes more prominent with increasing deformation rate, causing the crystal grains to rupture more easily, thus suppressing the superplastic elongation.

It is preferable to carry out a forming process for the aluminum alloy sheet of the present invention at a temperature between 350 to 550°C. At a temperature below 350°C, Al-Mg or Al-Mg-Cu compounds are prone to precipitate along
30 the grain boundary to lower the elongation. Conversely, at a forming temperature exceeding 550°C, the crystal grains tend to become coarse, adversely affecting the elongation. The preferred range of the strain rate during the forming process is between 10^{-2} to $10^0/\text{s}$, where a rate below $10^{-2}/\text{s}$ will cause the crystal grains to become coarser, reducing elongation, while a strain rate exceeding $10^0/\text{s}$ creates a shearing deformation within the crystal grains causing cracks, or forms precipitation along the grain boundary, reducing elongation.

35 As a procedure for preparing the aluminum alloy sheet in the present invention, an aluminum alloy material with the above mentioned composition is melted, cast, and homogenized according to a conventional method. It is preferable to carry out the homogenizing process at a temperature between 450 to 550°C. At temperatures below 450°C, Mg or Cu that are formed along the grain boundary or the cell boundary of the ingot by segregation will not be fully dissolved and may contribute to cracks in a subsequent hot rolling step. Conversely, at temperatures exceeding 550°C, the Al-Mg or
40 Al-Mg-Cu crystallization products will cause a eutectic fusion thereby giving rise to cracks during the hot rolling process.

After the homogenization process, the ingot is hot-rolled to obtain a structure suitable as a forming material. While the required starting temperature for hot rolling is between 250 to 500°C, it is preferable to start just under 400°C. If the hot rolling process is started at a temperature below 250°C, the deformation resistance is too high, making proper rolling difficult. If the rolling temperature is too high, this could alter the distribution form of the precipitation, thereby making
45 it difficult to obtain the required crystal grain structure as well as proper distribution of precipitated compounds.

Following the hot rolling process, a cold rolling is provided. In addition, an intermediate annealing may be provided as necessary. The final annealing of the cold rolled stock should be provided at a temperature between 350 to 550°C. If the annealing is performed at a temperature below 350°C, the isotropy created during the cold rolling process may not completely disappear; if higher than 550°C, a local melting may occur at the recrystallization boundary. As such, it is preferred to conduct the final annealing in a rapid annealing process such as continuous annealing.
50

In the present invention, by restricting content of Fe and Si as impurities in an Al-Mg alloy system and adjusting the combination of manufacturing conditions to the combination of the alloy constituents as described above, the Al-Fe-Si compounds present in the matrix are controlled within certain specific distribution while maintaining the crystal grain diameter within a certain specific range, resulting in such alloy structure and characteristics to produce cleaner grain
55 boundaries with less compounds formed along these boundaries to suppress cavity formation. Recrystallized grains having an average diameter of 20 μm or less are formed during a high-temperature deformation, thereby achieving an excellent elongation of 380% or greater in high speed forming at a strain rate of 10^{-2} to $10^0/\text{s}$ in a temperature range of 350 to 550°C.

EXAMPLES

The following describes examples of practical applications and comparative experiments pertaining to the present invention.

Example 1, Comparative Example 1

Al-Mg based aluminum alloys having compositions as listed in Table-1 below were melted and cast into ingots via a DC casting method. The resultant ingots were homogenized at 530°C for 10 hours to a thickness of 30 mm, and then hot rolled at 390°C to a thickness of 4 mm. The sheets were subsequently cold rolled to a thickness of 2 mm and then rapidly annealed by heating rapidly to 480°C and holding at this temperature for 5 minutes. Specimens prepared from the test materials produced in the above process were evaluated by a tensile test at a strain rate of 10⁻²/s at 480°C. Table 1 lists the average crystal grain diameter for each specimen (as measured at the sheet surface), the number per square millimeter of grains of the AL-Fe-Si compound having a diameter of 1 μm or above, and the elongation measurement results. Note here that the grain count of the compound was made using image processing.

Table 1

Material	Composition (wt%)					Average Crystal Grain Diameter (μm)	Al-Fe-Si Compound, φ 1 μm or larger (Nos./mm ²)	Elongation (%)
	Mg	Cu	Ti	Fe	Si			
1	5.4	-	0.02	0.05	0.05	60	1220	455
2	7.3	-	0.02	0.04	0.04	55	1360	420
3	5.4	-	0.01	0.01	0.01	130	250	480
4	5.0	0.45	0.02	0.06	0.06	45	1460	520
5	5.4	0.3	0.02	0.04	0.05	55	1270	560
6	6.5	0.6	0.05	0.07	0.06	-	-	-
7	8.5	-	0.05	0.03	0.04	-	-	-
8	5.2	-	0.04	0.12	0.15	15	3550	220
9	2.8	-	0.02	0.05	0.05	45	1180	280

As shown in Table 1, all of Materials No. 1 through 5 which comply with the present invention demonstrated elongation exceeding 400%. On the other hand, both Material No. 6 with an excessive Cu content, and Material No. 7 with excessive Mg developed cracks during the hot rolling process and failed to produce specimens. Further, Material No. 8 shows inferior elongation due to the excessive amount of impurities Fe and Si and the resultant number of large compound grains. Finally, Material No. 9, containing insufficient amount of Mg, also shows poor elongation due to lack of recrystallization during the stretching deformation.

Example 2, Comparative Example 2

Al-Mg based aluminum alloys having compositions as listed in Table 2 were melted and cast into ingots in the same manner as in Examples 1, and made into 2-mm thick test materials using the same process as in Examples 1. Specimens were then evaluated in the same tensile test under the same conditions. Table 2 lists the average crystal grain diameter, the number per square millimeter of grains of the AL-Fe-Si compound having a diameter of 1 μm or above, and the elongation measurement results.

Table 2

Material	Composition (wt%)							Average Crystal Grain Diameter (μm)	Al-Fe-Si Compound, ϕ 1 μm or larger (Nos./ mm^2)	Elongation (%)
	Mg	Cu	Ti	Mn	Cr	Fe	Si			
10	5.5	-	0.02	0.04	0.04	0.05	0.05	35	1330	410
11	5.4	0.3	0.02	0.05	0.01	0.04	0.04	25	1280	390
12	5.5	-	0.01	0.01	0.05	0.01	0.01	40	550	500
13	5.3	-	0.02	0.12	0.01	0.06	0.06	15	2730	280
14	5.4	-	0.03	0.15	0.10	0.04	0.05	11	3570	210
15	5.5	0.25	0.02	0.08	0.12	0.07	0.06	11	3240	240

As shown in Table 2, all the Materials No. 10 through 12 which comply with the present invention demonstrated elongation exceeding 380%. However, both Materials No. 13 and No. 14, with their excessive Mn content, as well as Material No. 15 with its excessive Cr content all showed inferior elongation due to excessive distribution of the Al-Fe-Si compound grains equal to or larger than 1 μm in diameter.

Examples 3, Comparative Example 3

An aluminum alloy having the same composition as Material No. 5 in Examples 1 was melted and cast in the same manner as in the Examples 1, and the resultant ingot was homogenized at 520°C for 8 hours to a thickness of 30 mm, then hot-rolled starting at 390°C to a thickness of 4 mm. The sheet was subsequently cold-rolled to a thickness of 2 mm and then rapidly annealed by heating rapidly to 480°C and holding there for 5 minutes. Specimens prepared from the test material produced in the above process were evaluated in a tensile test with varying strain rates and forming temperatures as indicated in Table 3. The elongation measurement results are as shown in Table 3. For guidance, the average crystal grain diameter (as measured at the sheet surface) for all of these specimens was in the range of 50 to 60 μm , and the number per square millimeter of grains of the AL-Fe-Si compound having a diameter of 1 μm or above, likewise, was below 2000.

Table 3

Material	Tensile Test Temperature ($^{\circ}\text{C}$)	Strain Rate (/s)	Elongation (%)
16	450	10^{-2}	480
17	180	10^{-2}	540
18	480	10^{-1}	410
19	520	10^{-2}	450
20	350	5×10^{-3}	380
21	580	10^{-2}	30
22	480	5×10^{-4}	280
23	480	2×10^0	80

As shown in Table 3, all the Materials No. 16 through 19 which comply with the present invention demonstrated elongation equal to or greater than 400%. However, the Material No. 21 showed a diminished elongation result due to its high tensile test temperature which resulted in coarse crystal grains. The Materials No. 20 and No. 22, on the other hand, showed poor elongation due to coarse crystal grains formed during deformation because of too small a strain

rate. Finally, the Material No. 23 with too high a strain rate employed also showed inferior elongation.

INDUSTRIAL APPLICATION

5 As described in the foregoing, the present invention provides an Al-Mg aluminum alloy sheet having excellent superplastic elongation in high speed forming such as at high strain rate of 10^{-2} to 10^0 /s at a high temperature, and a superplastic forming process using this aluminum alloy sheet shortens the forming time to improve productivity.

Claims

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1. An aluminum alloy sheet with excellent high-speed superplastic formability comprising an alloy containing 3.0 to 8.0% of Mg, 0.001 to 0.1% of Ti, controlled amounts of Fe and Si (as impurities) each at 0.06% or less, and the balance being Al and unavoidable impurities, wherein the number per square millimeter of grains of an Al-Fe-Si compound existing in the matrix structure of said alloy and having a diameter of 1 μ m or more is 2000 or less, the
15 mean crystal grain diameter is 25 to 200 μ m and the elongation is more than 380% as worked at a temperature range of 350 to 550°C and a strain rate of 10^{-2} to 10^0 /s.
 2. The aluminum alloy sheet with excellent high-speed superplastic formability according to claim 1, wherein said alloy further comprises 0.05 to 0.50% of Cu.
 - 20 3. The aluminum alloy sheet with excellent high-speed superplastic formability according to claim 1 or claim 2, wherein said alloy further comprises either one or both of not more than 0.10% of Mn or not more than 0.1% of Cr.
 - 25 4. A method for forming the aluminum alloy sheet with excellent high-speed superplastic formability according to claim 1, claim 2, or claim 3, wherein the aluminum alloy sheet is formed at a strain rate of 10^{-2} to 10^0 /s and at a temperature between 350 to 550°C.
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INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP95/02564

A. CLASSIFICATION OF SUBJECT MATTER		
Int. Cl ⁶ C22C21/06		
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)		
Int. Cl ⁶ C22C21/06		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Jitsuyo Shinan Koho	1926 - 1996	
Kokai Jitsuyo Shinan Koho	1971 - 1995	
Toroku Jitsuyo Shinan Koho	1994 - 1996	
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.
Y	JP, 5-212562, A (Sky Aluminium Co., Ltd.), August 24, 1993 (24. 08. 93), Line 34, column 1 to line 2, column 2, line 43, column 9 to line 8, column 11 (Family: none)	1 - 4
Y	JP, 6-240395, A (Sky Aluminium Co., Ltd.), August 30, 1994 (30. 08. 94), Line 1, column 1 to line 19, column 2, line 28, column 4 to line 1, column 5, line 18, column 5 to table 1, line 47, column 6 (Family: none)	1 - 4
Y	JP, 7-197177, A (Sky Aluminium Co., Ltd.), August 1, 1995 (01. 08. 95), Lines 1 to 17, column 1, lines 2 to 7, column 2, lines 18 to 30, 43 to 49, column 4, lines 2 to 6, 14 to 20, column 5 (Family: none)	1 - 4
Y	JP, 2-285046, A (Sky Aluminium Co., Ltd.), November 22, 1990 (22. 11. 90), Lines 6 to 13, lower left column, page 1, lines 2 to 16, upper left column, page 4	1 - 4
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/> See patent family annex.		
* Special categories of cited documents:		
"A"	document defining the general state of the art which is not considered to be of particular relevance	"T"
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		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
		"&"
		document member of the same patent family
Date of the actual completion of the international search	Date of mailing of the international search report	
March 8, 1996 (08. 03. 96)	March 19, 1996 (19. 03. 96)	
Name and mailing address of the ISA/ Japanese Patent Office	Authorized officer	
Facsimile No.	Telephone No.	

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP95/02564

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	(Family: none) JP, 6-17178, A (Ken Masumoto), January 25, 1994 (25. 01. 94), Lines 26 to 37, column 3, lines 31 to 37, column 4 (Family: none)	1 - 4

Form PCT/ISA/210 (continuation of second sheet) (July 1992)