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(54) **Highly corrosion-resistant and high strength aluminum alloys.**

(57) A highly corrosion-resistant and high-strength aluminum alloy prepared by solidification through rapid quenching is disclosed. This novel aluminum alloy can be obtained by rapid solidification of a melt containing no less than 0.2 atomic% and no more than 15 atomic% of at least one element selected from the group consisting of Si, Ti, Zr, Nb, Ni, Cu and Mn, with the balance being substantially composed of aluminum.

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HIGHLY CORROSION-RESISTANT AND HIGH STRENGTH ALUMINUM ALLOYS

The present invention relates to highly corrosion-resistant and high strength aluminum alloys produced by solidification through rapid quenching.

Industrially aluminum is an important metal material which is light and inexpensive, and is easily processed and extensively used both in the elemental form and after being produced and fabricated by a variety of methods as an alloy. However, aluminum and its alloys are highly susceptible to pitting corrosion under exposure to chloride-containing environments. Therefore, aluminum alloys, typically used as light alloys or sometimes as high strength light alloys depending on the composition, are not suitable for use in applications that require high corrosion resistance. The low resistance to corrosion of aluminum alloys may be explained by the accelerated corrosion that occurs due to the inclusion and/or the secondary phases formed by impurity elements present in the alloys. The addition of alloying elements effective in enhancing the corrosion resistance is impractical because they generally have narrow ranges for the formation of solid solutions.

The present inventors found that rapid solidification of molten alloys extended their solid solubility and sometimes led to the formation of amorphous alloys containing greater amounts of various elements than alloys produced by conventional methods, and that these amorphous alloys possessed extremely high corrosion resistance due to the chemically homogeneous nature of amorphous alloys and the addition of effective elements enhancing the corrosion resistance. The present inventors found by further investigations that vitrification of aluminum alloys is difficult but rapid solidification of molten alloys containing prescribed amounts of appropriate elements results in the formation of highly corrosion-resistant, high strength alloys having high hardness and very high pitting potential which are characteristics of supersaturated solid solutions of very fine grains.

The present invention has been accomplished on the basis of this finding.

The present invention provides a highly corrosion-resistant and high strength aluminum alloy produced by rapid solidification of a melt that contains no less than 0.2 atomic% and no more than 15 atomic% of at least one element selected from the group consisting of Si, Ti, Zr, Nb, Ni, Cu and Mn and the balance of which is substantially composed of Al.

An aluminum melt having the composition specified above may be solidified by any of the rapid quenching techniques commonly employed in the production of amorphous alloys, which include the rotating wheel method wherein a molten metal is injected on to the outer surface of a rotating wheel, the centrifugal quenching method wherein the melt is impinged against the inner surface of the rotating cylinder, the double-roll method wherein a liquid metal is quenched by passage between two closely positioned rolls, the gas method for making rapidly solidified flakes, the piston-anvil method, injecting a molten metal into a revolving chill medium for making a powder by rapid quenching, and other methods such as spraying and cavitation.

The aluminum alloys of the present invention that are produced by rapid solidification of the compositions specified above have appreciably higher pitting potentials than the conventionally processed aluminum alloys. In addition to this high corrosion-resistance, the aluminum alloys of the present invention exhibit high mechanical strength resulting from the formation of supersaturated solid solutions.

Fig. 1 is a diagrammatic view of the layout of the apparatus that may be used for the production of the rapidly solidified alloy of the present invention.

A suitable preparation method of rapidly solidified alloys of the present invention so called the rotating wheel method is as follows:

The rapidly solidified alloys with compositions mentioned above can be prepared by rapid quenching from the liquid state at a cooling rate of higher than 1,000°C/sec. If the cooling rate is slower than 1,000°C/sec., it is difficult to form supersaturated alloys. In principle, the rapidly solidified alloys of the present invention can be produced by any suitable apparatus providing a cooling rate higher than 1,000°C/sec.

One embodiment of apparatus for preparing the rapidly solidified alloys of the present invention is shown in the accompanying drawing. The apparatus is placed in a vacuum chamber indicated by the dotted rectangle. In the drawing, a quartz tube (2) has a nozzle (3) at its lower end in the vertical direction, and raw materials (4) and an inert gas for a jet of the raw materials melted are fed from the inlet (1). A heater (5) is placed around the quartz tube (2) so as to heat the raw materials (4). A high speed wheel (7) of 300 mm diameter is placed below the nozzle (3) and is rotated by a motor (6).

The apparatus is previously evacuated down to about 10^{-5} torr and then exposed to an inert gas atmosphere such as argon or nitrogen. The raw materials (4) having the specific compositions required are melted by the heater (5) in the quartz tube under an inert gas atmosphere. The molten alloys impinge under the pressure of the inert gas of 0.4 -2 kg/cm² onto the outer surface of the wheel (7) which is rotated at a speed of 500 to 10,000 rpm whereby the rapidly solidified alloys are formed as long thin plates, which may, for example, have thicknesses of 0.01 -0.1 mm, widths of 1 -10 mm and lengths of several to several tens of meters.

The criticality of each of the components in the aluminum alloy in accordance with the present invention is hereunder described. The aforementioned elements Si, Ti, Zr, Nb, Ni, Cu and Mn are alloying elements which provide high corrosion resistance and mechanical strength by supersaturatedly dissolving in the α -Al phase which is the softest phase having the lowest corrosion resistance among phases in the aluminum alloys.

If the amount of at least one element selected from the group consisting of Si, Ti, Zr, Nb, Ni, Cu and Mn is less than 0.2 atomic%, the corrosion resistance of the aluminum alloy prepared by rapid solidification is not sufficiently high and is not much different from that of rapidly solidified aluminum metal. If, on the other hand, the amount of at least one element selected from among Si, Ti, Zr, Nb, Ni, Cu and Mn exceeds 15 atomic%, the rapidly solidified alloy is too brittle to be used for practical purposes, except for the case where only Mn is added.

Therefore, in order to attain the objects of the present invention, the amount of at least one element selected from among Si, Ti, Zr, Nb, Ni, Cu and Mn must be within the range of 0.2 -15 atomic%.

In order to attain better results, it is desirable to add at least one of these elements in an amount of 0.5 -15 at.%.

The rapidly solidified aluminum alloy described above may contain no more than 4 atomic% of another element such as Mg, V, Cr, Fe, Co or Zn without sacrificing the objects of the present invention.

In accordance with the present invention, the elements added will be finely distributed along the boundaries of fine grains formed within the alloy as a result of rapid solidification and, hence, will prevent growth of these grains in subsequent heat treatments. Therefore, the aluminum alloy of the present invention may, after rapid solidification, be processed into a desired shape by extrusion, compression, press-forming or sintering under the processing conditions selected properly so that the objects of the present invention will not be impaired.

When a liquid alloy having one of the compositions described above is rapidly solidified, the alloying elements will dissolve in the α -Al matrix to form a supersaturated solid solution, and impart remarkably high corrosion resistance and strength to the matrix. Additionally, all other phases that will result from the addition of the alloying elements have high corrosion resistance and strength, thereby producing an aluminum alloy that exhibits very high corrosion resistance and strength features.

On the other hand, most of the alloying elements in conventionally processed aluminum alloys will not dissolve in the α -Al matrix and a variety of impurity elements will form heterogeneous phases which are deleterious to the purpose of attaining high corrosion resistance. On the other hand, the added elements in the present invention will dissolve in a molten aluminum alloy no matter how complex their compositions are. Therefore, when a molten metal having one of the compositions specified by the present invention is rapidly solidified, a variety of elements that are

effective in the enhancement of passivation will dissolve in the most corrosion-susceptible α -Al phase to form supersaturated solid solutions. The so solidified alloy forms a highly protective passive film having a high pitting corrosion resistance and hence exhibits high corrosion resistance and high mechanical strength.

The following examples are provided for the purpose of further illustrating the present invention but are by no means intended as limiting.

Example 1

Thirty alloys having the compositions listed in Table 1 were cast by argon arc melting of commercial metals. The cast alloys were remelted under an argon atmosphere and rapidly solidified by the rotating wheel method after remelting under an argon atmosphere, producing thin sheets of rapidly solidified alloys ranging 0.01 -0.05 mm in thickness, 1 -3 mm in width, and 3 -20 m in length. Analysis by X-ray diffractor revealed that the added alloying elements dissolved in large amounts to form supersaturated solid solutions of the α -Al phase since the eutectoid-forming Si and the compound phase consisting of Al and alloying elements had lower diffraction intensities than conventionally processed alloys having the same compositions. Optical microscopy and scanning-electron microscopy showed that each of the alloy samples prepared in accordance with the present invention had fine-grained structures comprising particles no larger than 0.5 μ m.

Table 1 : Alloy Composition

(at.%)

Sample No.	Al	Si	Ti	Zr	Nb
1	99.5	0.5	0	0	0
2	97	3	0	0	0
3	95	5	0	0	0
4	90	10	0	0	0
5	85	15	0	0	0
6	99.7	0	0.3	0	0
7	98	0	2	0	0
8	94	0	6	0	0
9	90	0	10	0	0
10	99.8	0	0	0.2	0
11	94	0	0	6	0
12	90	0	0	10	0
13	99.8	0	0	0	0.2
14	98	0	0	0	2
15	95	0	0	0	5
16	90	0	0	0	10
17	93	5	2	0	0
18	91	5	4	0	0
19	89	5	6	0	0
20	85	5	10	0	0
21	93	5	0	2	0
22	91	5	0	4	0

23	89	5	0	6	0
24	85	5	0	10	0
25	93	5	0	0	2
26	91	5	0	0	4
27	89	5	0	0	6
28	85	5	0	0	10
29	91	3	2	2	2
30	89	0	3	3	5

Example 2

Two samples of 99.999% pure aluminum were prepared by rapid solidification and conventional processing, and two samples of an Al-5 at.% Si alloy were also prepared by conventional processing and rapid solidification in accordance with the present invention using the rotating wheel method. The conventionally processed Al-5 at.% Si alloy was comprised of an α -Al phase and a eutectoid of α -Al and cubic Si. In the rapidly solidified Al-5 at.% Si alloy,

the amount of the cubic Si in the eutectoid was reduced to about one third of the amount in the conventionally processed counterpart and the resulting α -Al phase had at least 3 at.% of Si dissolved therein. The structure of the rapidly solidified alloy was uniform and comprised of fine grains not larger than 0.5 μ m in size.

The pitting potential of the four solidified samples were measured in deaerated 0.5N NaCl at 30°C. Their Vickers hardness values were also measured. The results are shown in Table 2.

Table 2 : Pitting Potentials and Hardnesses of Rapidly Solidified and Conventionally Processed Metals and Alloys

Samples	Pitting potential V (SCE)	Hardness Hv
Conventionally processed Al	-0.750	26.6
Conventionally processed Al-5% Si	-0.650	α -phase, 38 eutectoid, 51.5
Rapidly solidified Al	-0.675	51.5
Rapidly solidified Al-5% Si (sample of the present invention)	-0.575	72

As Table 2 shows, the rapidly solidified Al-5 at.% Si alloy sample processed in accordance with the present invention had a pitting corrosion potential 175 mV higher than that of conventionally processed 99.999% pure aluminum, and 75 mV higher than that of the conventionally

processed Al-5 at.% Si alloy sample. In addition to this high pitting corrosion resistance, the alloy of the present invention had a very high hardness that contributed to its high strength property.

Example 3

Three different samples of aluminum alloys, viz., Al-6 at.% Ti, Al-2 at.% Ti-5 at.% Si, and Al-6 at.% Ti-5 at.% Si, were prepared by rapid solidification in accordance with the present invention using the rotating wheel method.

The Al-Ti-Si alloys were composed of three phases, i.e., α -Al, Al_3Ti , and a eutectoid consisting of α -Al and cubic Si, and rapid solidification reduced the amount of the Al_3Ti phase to about one fifth and the cubic Si in the eutectoid to about one third as a result of dissolution of substantial

amounts of Ti and Si in the α -Al phase. For example, the α -Al phase in the Al-6% Ti-5% Si alloy sample prepared by rapid solidification in accordance with the present invention contained about 5 at.% of Ti and about 3 at.% of Si. Each of the alloys prepared by the present invention had a uniform structure comprising fine grains no larger than 0.5 μ m. Their pitting potentials in deaerated 0.5N NaCl solution and Vickers hardness were measured. The results are shown in Table 3 for comparison with the data for conventionally processed samples.

Table 3: Pitting Potentials and Hardnesses of Rapidly Solidified and Conventionally Processed Metals and Alloys

Samples	Rapidly solidified		Conventionally processed	
	Pitting potential V (SCE)	Hardness Hv	Pitting potential V (SCE)	Hardness Hv
Al (comparative sample)	-	-	-0.750	26.6
Al-6% Ti	-0.600	160	-0.750	-
Al-2% Ti-5% Si	-0.500	140	-0.710	-
Al-6% Ti-5% Si	-0.550	170	-0.730	-

The Al-Ti alloy and the Al-Ti-Si alloys rapidly solidified in accordance with the present invention contained sufficiently large amounts of Ti and Si in the α -Al phase to provide a highly protective passive film that could prevent the pitting corrosion by passivity breakdown. Therefore, the rapidly quenched alloys of the present invention had the pitting potentials 150 -250 mV higher than that of the 99.999% pure aluminum processed conventionally. The alloys of the present invention were also characterized by fine grained phases containing the finely dispersed very hard Al_3Ti phase and the α Al matrix supersaturated with alloying elements. As a result, the hardness of the alloys was even higher than the maximum value attainable by the conventional aluminum alloys.

Example 4

Four different samples of aluminum alloys, viz., Al-6 at.% Zr, Al-2 at.% Zr-5 at.% Si, Al-4 at.% Zr-5 at.% Si and Al-6 at.% Zr-5 at.% Si, were prepared by rapid solidification in accordance with the present invention using the

rotating wheel method. Each of the rapidly solidified samples had a fine-grained structure comprising particles no larger than 0.5 μ m. An Al-Zr-Si alloy processed conventionally was composed of three phases, i.e., α -Al, a eutectoid consisting of α -Al and cubic Si, and $Al_{0.45}Zr_{0.33}Si_{0.22}$. The Al-Zr-Si alloys solidified rapidly in accordance with the present invention were comprised of the α -Al phase, Al_3Zr phase and a eutectoid consisting of α -Al and cubic Si, with a smaller content of the Si in the eutectoid than in the conventionally processed counterparts. In solidification by rapid quenching, the high-melting point Al_3Zr phase precipitated before dissolving Si and then the eutectoid and α -Al phases rapidly formed. Therefore, the α -Al phase was supersaturated with Si and Zr. The alloy samples prepared in accordance with the present invention were subjected to the measurements of the pitting potential in deaerated 0.5N NaCl solution and Vickers hardness. The results are shown in Table 4 for comparison with the data for samples solidified conventionally.

Table 4 : Pitting Potentials and Hardnesses of Rapidly Solidified and Conventionally Processed Metals and Alloys

Samples	Rapidly solidified		Conventionally processed	
	Pitting potential V (SCE)	Hardness Hv	Pitting potential V (SCE)	Hardness Hv
Al (comparative sample)	-	-	-0.750	26.6
Al-6% Zr	-0.590	200	-0.750	-
Al-2% Zr-5% Si	-0.550	145	-0.730	-
Al-4% Zr-5% Si	-0.530	180	-0.730	-
Al-6% Zr-5% Si	-0.550	243	-0.750	-

Because the α -Al phase was supersaturated with Zr and Si, the alloys of the present invention formed a highly protective passive film over the α -Al phase that could prevent the pitting corrosion resulting from the passivity breakdown of the α -Al phase. Therefore, the rapidly quenched alloys of the present invention had the pitting potentials 160 -220 mV higher than the value for the 99.999% pure aluminum processed conventionally. In addition to this feature of high corrosion resistance, the alloys had a significantly improved hardness because of the dispersion of the fine-grained Al_3Zr phase.

Example 5

Two molten samples of aluminum alloy, viz., Al-2 at.% Nb-5 at.% Si and Al-6 at.% Nb-5 at.% Si, were solidified by rapid quenching by means of the rotating wheel method. Each of the solidified samples had a fine-grained structure

comprising particles no larger than 0.5 μ m. An Al-Nb-Si alloy processed conventionally was composed of three phases, i.e., α -Al, Al_3Nb and a eutectoid consisting of α -Al and cubic Si. No distinct Si was identified by X-ray diffraction of the Al-Nb-Si alloys solidified by rapid quenching in accordance with the present invention and all the Si present had formed a supersaturated solid solution of the α -Al phase. The alloy samples prepared in accordance with the present invention were subjected to the measurements of pitting potentials (in deaerated 0.5N NaCl solution) and Vickers hardness. The results are shown in Table 5 for comparison with the data for samples solidified by a non-rapid quenching technique.

Table 5 : Pitting Potentials and Hardnesses of Rapidly Solidified and Conventionally Processed Metals and Alloys

Samples	Rapidly solidified		Conventionally processed	
	Pitting potential V (SCE)	Hardness Hv	Pitting potential V (SCE)	Hardness Hv
Al (comparative sample)	-	-	-0.750	26.6
Al-2% Nb-5% Si	-0.530	140	-0.740	-
Al-6% Nb-5% Si	-0.530	241	-0.740	-

In the alloys of the present invention, the Al₃Nb phase having high corrosion resistance and hardness was dispersed as a fine-grained structure, and the solutes formed supersaturated solid solutions of the α -Al phase. Therefore, the alloys had significantly high pitting potentials and improved hardness.

Example 6

Thirty alloys having the compositions listed in Table 6 were cast by argon arc melting of commercial metals. The cast alloys were re-melted under an argon atmosphere and solidified rapidly by the rotating wheel method under an argon atmosphere, producing thin sheets of rapidly solidified alloys ranging 0.01 -0.05 mm in thickness, 1 -3 mm in width, and 3 - 20 m in length. Analysis by X-ray diffractor revealed that the added alloying elements dissolved in large amounts to form supersaturated solid solutions of the α -Al

phase since the eutectoid-forming Si and the compound phase consisting of Al and alloying elements had lower diffraction intensities than conventionally processed alloys having the same compositions. Optical microscopy and scanning-electron microscopy showed that each of the alloy samples prepared in accordance with the present invention had a fine-grained structure comprising particles no larger than 0.5 μ m.

After mechanical polishing with silicon carbide paper up to No. 1,500 in water, the alloy samples were subjected to the measurements of the pitting potential in deaerated 0.5N NaCl solution at 30°C and vickers hardness. The results are shown in Table 6.

As is clear from Table 6, the aluminum alloys prepared by rapid solidification in accordance with the present invention had the pitting potentials 50 -330 mV higher than the value for the 99.999% pure aluminum solidified conventionally, and they also exhibited very high hardness levels.

Table 6 : Alloy Compositions (at.%) and Their Pitting Potentials and Hardnesses

Sample No.	Al	Ni	Cu	Mn	Si	Ti	Zr	Nb	Pitting potential V (SCE)	Hardness Hv
1	99.5	0.5							-0.575	120
2	96	4							-0.505	130
3	94	6							-0.440	140
4	94	4	2						-0.490	140
5	99.7		0.3						-0.600	120
6	94		6						-0.620	130
7	92		6	2					-0.500	135
8	94	2	2	2					-0.400	140
9	99.5			0.5					-0.460	108
10	94			6					-0.420	119
11	90			10					-0.405	130
12	85			15					-0.400	140
13	93	2			5				-0.470	136
14	91	4			5				-0.410	180
15	89	6			5				-0.350	217
16	89	2			5	4			-0.550	170
17	89	2			5		4		-0.530	190
18	89	2			5			4	-0.440	140
19	93		2		5				-0.490	101
20	91		4		5				-0.530	121
21	89		6		5				-0.480	149
22	94		2			4			-0.600	160
23	94		2				4		-0.590	200
24	93			2	5				-0.630	140

25	91			4	5			-0.600	170
26	89			6	5			-0.610	200
27	89			2	5		4	-0.530	241
28	89	2	2	2	5			-0.520	210
29	90	2		4		4		-0.540	170
30	89		2	4	5			-0.520	180
Comparative sample 99.999% Al								-0.680	51.5

As described in the foregoing pages, the aluminum alloys prepared by rapid solidification in accordance with the present invention are characterized in that the elements added in order to enhance the corrosion resistance and strength are dissolved supersaturatedly in the α -Al phase, and form the eutectoid and intermetallic compounds having high corrosion resistance and high mechanical strength. Because of these features, the alloys of the present invention are possessed of the high degree of corrosion resistance and strength that has been unattainable with the prior art techniques.

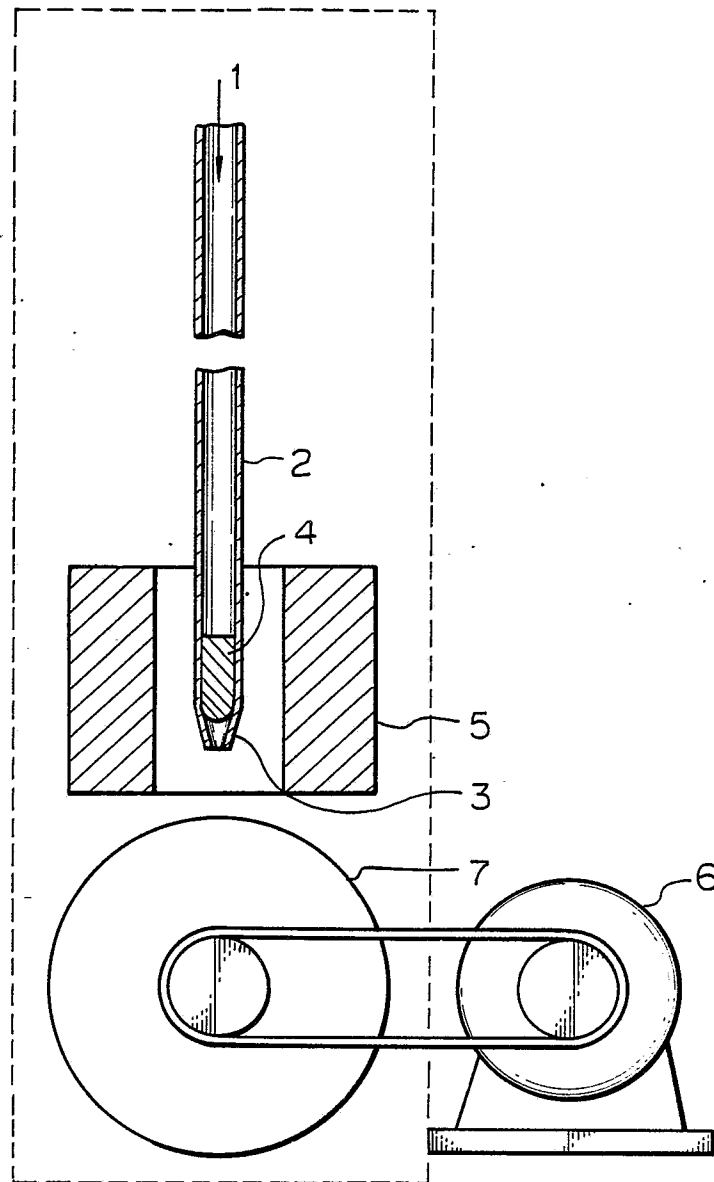
The rapid quenching necessary for producing the alloys of the present invention can be realized by any of the methods that are well established in the art for rapid quenching from the liquid state. Therefore, the alloys of the present invention can be produced without using any special apparatus and, hence, will find great utility in practical applications.

20 Claims

1. A highly corrosion-resistant and high-strength aluminum alloy produced by rapid solidification of a melt containing no less than 0.2 atomic% and no more than 15 atomic% of at least one element selected from the group consisting of Si, Ti, Zr, Nb, Ni, Cu and Mn, with the balance being substantially composed of Al.

2. A highly corrosion-resistant and high-strength aluminum alloy according to Claim 1 wherein said at least one element is present in an amount of 0.5 to 15 atomic%.

Fig. 1





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X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			



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CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			



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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
X, P	EP-A-0 170 963 (ALLIED CORP.) * Claims 1,4; page 13, examples 6,11 and table 1, alloy Al-3Si-8,5Ti * ---	1	
A	EP-A-0 100 287 (CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE) * Claim 1 * --- -----	1	
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 18-06-1986	Examiner LIPPENS M.H.
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