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(54) **ALUMINIUM FOIL**

Aluminiumfolie

FEUILLE EN ALUMINIUM

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EP-A- 0 394 818 GB-A- 1 479 429
US-A- 4 737 198

- **PATENT ABSTRACTS OF JAPAN vol. 013 no. 214 (M-827) ,18 May 1989 & JP,A,01 034548 (FURUKAWA ALUM CO LTD;OTHERS: 01) 6 February 1989,**
- **PATENT ABSTRACTS OF JAPAN vol. 012 no. 230 (C-508) ,29 June 1988 & JP,A,63 026340 (KOBELITE LTD) 3 February 1988,**
- **PATENT ABSTRACTS OF JAPAN vol. 015 no. 385 (C-0871) ,27 September 1991 & JP,A,03 153836 (MITSUBISHI ALUM CO LTD) 1 July 1991,**
- **HUFNAGEL W. 'Aluminium-Taschenbuch' 1983, ALUMINIUM-VERLAG, DÜSSELDORF, DE ***
Alloy X8006 * see page 1018 - page 1019

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Description

This invention is concerned with aluminium foil having improved strength. In the current Al-Fe-Mn based foil alloys, such as AA 8006 and AA 8014, the good balance of strength and formability of thin gauge foil is obtained by achieving a combination of fine grain size after final annealing and dispersion strengthening. This invention describes the use of an additional strengthening mechanism to achieve increased strength; namely solid solution strengthening, and specifies the range within which the solute level must be controlled in order to avoid loss of other beneficial properties associated with the solute-free versions of these alloys.

British Patent Specification 1 479 429 described dispersion-strengthened aluminium alloys based on the Al-Fe-Mn system, such as AA 8006 and AA 8014. (from Registration record of international alloy designations and chemical composition limits for wrought Al and wrought Al alloys, AA Inc. May 1987). The as-cast ingot comprised unaligned intermetallic rods. These were broken up during working to provide a wrought aluminium alloy product containing dispersed intermetallic particles. The invention was applicable to the production of rolled sheet, which was to some extent anisotropic. It was possible to reduce the relative proportions of the anisotropy by introducing small proportions of Cu and/or Mg which remained in solid solution in the Al phase and had known strength providing properties. The loss of anisotropy implies discontinuous recrystallisation and loss of grain size control, which changes would have been acceptable in the sheet products mainly envisaged and exemplified.

JP-A- 1 034 548 described the production of high strength aluminium foil by twin-roller casting and having the composition 0.8-2wt% Fe, 0.1-1% Si, 0.01-0.5% Cu, 0.01-0.5% Mg, 0.01-1% Mn, one or more among $\leq 0.1\%$ Ti and $\leq 0.05\%$ B and the balance Aluminium. Final annealing is performed at 400°C.

The successful production of aluminium foil having useful properties depends on several critical parameters. The metal to be rolled must not be too hard, otherwise rolling down to the very low thicknesses below 100 μm required is not commercially viable. After rolling, the foil has to be heated, to a temperature sufficient to remove rolling lubricant but not so high that adjacent sheets of foil stick together. This temperature window is quite narrow, generally 220 - 300°C, and results in a final annealing treatment of the foil. During this annealing treatment, recrystallisation takes place, and it is necessary that this be continuous recrystallisation, which retains a desired small grain size, rather than discontinuous recrystallisation, which results in grain growth. If large grains are present, the foil has reduced mechanical properties. While these critical parameters have long been achieved using Al-Fe-Mn alloys, it was not apparent that they could be achieved in combination with solid solution hardening. And indeed, as the inventors have discovered, the nature and amount of solute that can be added is critically circumscribed.

In one aspect this invention provides aluminium foil composed of an alloy of composition by weight %:

Fe	1.2 - 2.0%
Mn	0.2 - 1.0%
Mg and/or Cu	0.1 - 0.5%
Si	up to 0.4%
Zn	up to 0.1%
Ti	up to 0.1%
balance Al of at least commercial purity	

which foil has an average grain size below 5 μm after final annealing.

In another aspect, the invention provides aluminium foil of the stated composition, wherein at least 50% by volume of the as-rolled texture is retained after final anneal.

In another aspect, the invention provides aluminium foil of the stated composition, wherein the crystallographic texture of the final annealed product is a retained rolling texture.

The aluminium foil preferably has a thickness below 100 μm , particularly in the range 5 - 40 μm e.g. 10 - 20 μm . The improved strength of foil according to this invention should enable thinner gauges to be marketed.

Fe and Mn are present to provide dispersion strengthening properties, as described in the aforesaid GB 1 479 429. Preferably the Fe content is 1.4 - 1.8%; the Mn content is 0.3 - 0.6%; and the Fe + Mn content is 1.8 - 2.15%.

If the Fe + Mn concentration in the melt exceeds this value of 2.15 then coarse primary intermetallic particles (typically up to 100 μm length) can form during solidification as a consequence of nucleation of these phases on the cooler parts of the molten metal distribution system. These coarse particles will break-up somewhat during subsequent processing but will still persist as relatively coarse non-deformable particles in the final product. For the case of sheet products this will not cause significant problems, but in the case of foil products will give rise to problems with pin-hole formation in the rolled strip and give rise to excessive strip breaks during processing. It is thus preferred to cast a composition where primary intermetallic particles cannot form, and this imposes an upper limit on the Fe and Mn levels

for use of this invention for foil products.

Mg and/or Cu is added to provide solution strengthening, in a concentration of 0.1 - 0.5% preferably 0.15 - 0.35%. At the lower end of these ranges, little strengthening is observed. At the upper end of these ranges, there is a risk that the solute will encourage discontinuous recrystallisation and will result in undesired grain growth. This risk is particularly apparent at relatively high annealing temperatures. As shown in the examples, Mg provides a better solution strengthening effect than Cu at equivalent concentrations and is accordingly preferred.

The inventors have tried other solution strengthening elements, but have found that they tend to encourage discontinuous recrystallisation during final anneal or are otherwise unsatisfactory. It is therefore believed that Mg and Cu are the only two usable solution strengthening additives.

Si and Zn are included in the AA specifications of AA 8006 and AA 8014. But they are preferably not deliberately included here. It is an advantage of the invention that recycled scrap metal can be used to make the foil.

The foil is specified as having an average (or mean) grain size below 5 μm , preferably below 3 μm . The grain size is preferably substantially uniform, and is achieved as a result of continuous recrystallisation during final anneal. Alternatively a non-uniform grain size may be acceptable provided that gross discontinuous recrystallisation during final anneal is avoided. For example, the majority of grains may have a size of 2-3 μm with a minor proportion of grains of 10-30 μm . This duplex grain size structure may reduce the ductility of the foil, but the overall properties may nevertheless be satisfactory.

Grain size may be determined by the mean linear intercept method. On a micrograph of a section of the alloy under test, a line (e.g. a straight line or a circle) of known length is drawn, and a count is made of the number of intercepts of that line with grain boundaries. The mean linear intercept grain size (mean grain size) is the length of the line divided by the number of intercepts.

The foil is generally anisotropic. Cold-rolling develops an as-rolled texture typical of dilute Al alloys. Texture is conventionally measured from an orientation distribution function in terms of six parameters (cube, goss, copper, S, brass and random). Conventionally, these are measured as a volume fraction of crystals orientated over a $\pm 15^\circ$ spread about the appropriate Miller indices which are {001}<100>, {110}<001>, {112}<111>, {123}<634>, {011}<211> respectively, the random component being the remaining volume fraction. The copper, S and brass components are generated by cold rolling. Discontinuous recrystallisation would tend to destroy the as-rolled texture and favour the formation of cube and/or goss and/or random. In the foil of this invention, at least 50% by volume, preferably 75% of this as-rolled texture as represented by the copper, S and brass components is retained after final anneal. Preferably the crystallographic texture of the final annealed product is substantially the same as the as-rolled product with no significant levels of recrystallisation texture components.

It has surprisingly been found that the foil of this invention may have a surface roughness greater than that of its solute-free counterpart. This increase in roughness was confirmed by optical profilometry (Perthometer) measurements, giving an R_a of 0.38 for foil of this invention (Example 2) compared with an R_a of 0.24 for a commercial foil of corresponding composition without Mg. The rougher surface improves the matt appearance of the foil.

In making the aluminium foil of this invention, a molten aluminium alloy of desired composition is cast, e.g. by direct chill (D.C.) casting, or alternatively by roll casting or belt casting or other known casting techniques. The cast metal is rolled by successive rolling steps in conventional manner down to the required foil thickness. These steps typically involve hot rolling followed by cold rolling, possibly with one or more interannealing steps. Finally, the foil is heated to a temperature sufficient to remove the rolling lubricant. The heating rate is preferably $1^\circ\text{C} - 100^\circ\text{C}$ per hour. As noted above, this temperature is typically in the range $220 - 300^\circ\text{C}$, preferably $230 - 280^\circ\text{C}$, more preferably $230 - 250^\circ\text{C}$, and also effects continuous recrystallisation of the foil. The aluminium foil of this invention is preferably substantially free of surface contamination by rolling lubricant.

The technical basis of the invention, as presently understood by the inventors, is explained in the following paragraphs.

A range of aluminium alloys are known to achieve a fine grain size after final annealing by a gradual coarsening of the cold-rolled substructure, sometimes called continuous recrystallisation, which allows a good combination of strength and formability to be achieved. During the final annealing of aluminium foil products it is important to avoid the occurrence of large recrystallised grains which severely diminish formability, often as a result of strain localisation leading to premature failure during loading. These grains are formed in the classical discontinuous manner whereby individual grains nucleate and grow to a large size. It is known that in these type of alloys this transition from discontinuous to continuous recrystallisation occurs when the level of cold work is increased above a critical level typical of foil rolling.

If there is a sufficient high concentration of non-deformable intermetallic particles, such as the FeAl_6 and/or $(\text{FeMn})\text{Al}_6$ eutectic rods formed during solidification of Al-Fe-Mn alloys such as AA 8006 and AA 8014, then after deformation to high strains these particles must have increased dislocation activity associated with them in order to maintain continuity across the aluminium/particle interface. Under conventional solute-free conditions, these dislocations are capable of rearranging themselves into dislocation walls, or sub-grain boundaries. As deformation proceeds the geometri-

cally necessary dislocations generated during the rolling process continue to migrate to, and recover into, the sub-grain boundaries, increasing their misorientation. Eventually these boundaries will attain high misorientations with their neighbours, i.e. high angle grain boundaries. When these boundaries are then annealed, they can all migrate at similar rates, thus encouraging continuous recrystallisation. In addition this is helped by the ability of the now broken up rod eutectic to pin grain boundaries and prevent excessive rates of grain growth. Dispersoids formed during hot processing of the ingot will also assist this pinning process.

Thus, the conventional (solute-free) AA 8006 achieves a fine grain size after anneal, which imparts the good balance of strength and ductility associated with these alloys. The strength is inversely proportional to the grain (or sub-grain) size, and follows a d^{-1} relationship.

This invention still maintains this strengthening mechanism whilst using the additional strengthening mechanism of solid solution strengthening. If the amount of solute added is too high then the ability to control the grain size during the final anneal is lost, giving rise to a decrease in grain size strengthening and formability. This presumably is because dynamic recovery is prevented during rolling, and so the driving force for discontinuous recrystallisation is increased. This also makes it increasingly difficult to roll the foil to the required thin gauge because of the increased rolled strength, giving a loss of the roll softening normally found in solute-free alloys of this type.

Another aspect of the rearrangement of dislocations into high angle grain boundaries during the rolling process is that the strength of the foil decreases as the rolling strain is increased (roll softening), instead of the usual roll hardening associated with most aluminium alloys. Adding solute to the alloy will hinder the ability of the dislocations to rearrange themselves into low energy configuration in the sub-grain boundaries, and will prevent roll softening from occurring. Thus, if too much solute is added the cold rolled strength of the foil will be significantly increased, losing the ability to roll the material to the thin gauges needed for household foil and packaging applications (within the range 40 μm to 5 μm).

Reference is directed to the accompanying drawings in which :-

Figure 1 shows the effect of annealing temperature on tensile strength of laboratory processed alloys rolled to 140 μm ;

Figure 2 shows the effect of annealing temperature on tensile yield stress of laboratory processed alloys rolled to 140 μm ;

Figure 3 shows the effect of annealing temperature on tensile elongation of laboratory processed alloys rolled to 140 μm ; and

Figures 4a and 4b are pole diagrams of a foil sample before and after annealing.

Example 1

Laboratory Processing

The effect of different levels of copper and magnesium additions have been investigated using laboratory processing of 200 mm x 75 mm cross-section D.C. ingots of 1.6% Fe, 0.40% Mn, 0.15% Si (denoted by 8006 in the figures) and modified alloys containing 0.2% and 0.4% of Cu or Mg. At the cooling rates associated with this ingot cross-section, the addition of the solutes does not prevent the formation of the preferred rod eutectic, with only slight coarsening being observed.

The above ingots have been heated to 525°C, hot rolled to 20 mm, and annealed at 330°C for 3 hours to simulate commercial hot processing. The materials have then been cold rolled to 4.5 mm, interannealed at 360°C, and cold rolled to 145 μm . This reproduces the strain levels achieved during rolling of 14 μm household foil. Table 1 shows the effect of the rolling reduction on the tensile strength of the materials. Adding solid solution strengtheners prevents the usual roll softening associated with AA 8006 from occurring, thus imposing an upper limit on how much solute could be added and still enable thin gauge products to be rolled commercially.

The 140 μm foil has been annealed for 2 hours at a range of temperatures using a simulation of batch annealing, involving heating to temperature at 25°C/hour and longitudinal tensile properties measured. The variation of UTS, 0.2% proof stress, and elongation-to-failure are shown in Figures 1, 2 and 3, respectively, for the five alloys. All alloys containing the solute additions show an improvement in UTS over the solute-free AA 8006, with the improvement being of the order of 20 to 40 MPa after the commercially usable anneal at temperatures in the region of 220 - 260°C. However, after annealing at the highest temperature investigated (300°C) the more concentrated alloys have lower strengths than the 0.2% additions. This is more pronounced in the proof stress data, and indicates that in the more concentrated alloys there is a loss in strength as a consequence of loss of grain size control caused by discontinuous recrystallisation. This is confirmed by the optical metallography of the grain structures after annealing where coarse grained regions are apparent in the solute containing alloys annealed at the highest temperatures. The loss of grain size control is often associated with loss of formability and ductility, although the ductilities do not show any reduction here, possibly

as a consequence of the much thicker gauges examined here (140 μm vs 14 μm) preventing strain localisation. This loss of grain size control at the higher temperatures in the 0.4% containing alloys shows that there will be an upper limit on the amount of solute which can be added for solid solution strengthening without running into problems with loss of strength (in particular yield strength) caused by coarser recrystallised grains.

Example 2

Plant Trials

Based on the wish to achieve a significant strength increase over the standard AA 8006 composition, a full scale processing trial has been performed with AA 8006 plus 0.2 wt.% Mg. This was DC cast as an ingot of 1600 mm x 600 mm cross section. The ingot was then processed, the processing route consisting of hot rolling to 3 mm, cold rolling to 450 μm , and interannealing at 360°C. It was then cold rolled to the final gauge of 14 μm .

Tensile testing of the as-rolled foil showed the yield stress to be significantly higher than the standard Mg-free version (Table 2).

Commercial batch anneal is carried out at a temperature of 220 - 260°C during a heating cycle of at least 8 hours from room temperature to the annealing temperature. Preferably the metal is held in the temperature range for at least 30 minutes. The total cycle time depends on the coil width. Tensile properties of the plant annealed foil are shown in Table 2, showing that a significant strength improvement is achieved over AA 8006. The results of the plant trial clearly demonstrate that there will be an upper limit on the amount of magnesium which can be added to large cross-section D.C. cast ingot and still give the required microstructure for continuous recrystallisation.

The process of rolling is highly anisotropic as a result of crystal plasticity and inevitably leads to a product with preferred orientations or crystallographic texture. In order to describe crystallographic texture a system has been devised that enables reference directions on the sample to be related to the crystallographic directions of a large number of grains on a simple diagram called a pole figure. The techniques for measuring crystallographic texture in metals are well established and an excellent reference is Hatherley and Hutchinson "An Introduction to Textures in Metals" The Institute of Metallurgists, Monograph No 5, 1979.

The crystallographic texture of the foil samples before and after annealing have been determined using x-ray diffraction from a laminate made from the 14 μm foil. Figures 4a and 4b show the pole figures generated from the {111} aluminium planes orientated relative to the rolling direction (vertical), transverse direction (horizontal) and the foil plane normal (into the page). Figure 4a is the as-rolled foil. Figure 4b is the annealed foil. The contour levels are 1.00 1.60 2.20 2.80 3.40 4.00 4.60. This shows that the crystallographic texture is essentially unaltered by the anneal, i.e. the texture is a retained rolling texture. Pole figures corresponding to other aluminium reflections have also been obtained from which the Orientation Distribution Function (ODF) in the rolled and annealed conditions have been generated. The volume fractions of specific texture components have been extracted from the ODF's and these are shown in Table 3.

The grain size of the 14 μm foil has been determined after commercial annealing using the mean linear intercept technique. This has been performed on micrographs obtained in the Transmission Electron Microscope (TEM). A total line length of 1mm has been examined and the mean linear intercept grain size determined to be 3.1 μm .

Table 1 -

Effect of solute additions on the as-rolled strength of laboratory processed alloys rolled to give the equivalent strain as commercially rolled housefoil.			
Alloy	0.2% Proof Stress (MPa)	UTS (MPa)	Elongation (%)
8006	159	210	6.1
8006 + 0.2% Cu	217	268	5.9
8006 + 0.4% Cu	249	309	3.3
8006 + 0.2% Mg	259	311	2.2
8006 + 0.4% Cu	274	331	2.4

Table 2 -

Tensile properties of commercially produced 14 µm foil.				
Alloy	condition	0.2% Proof Stress (MPa)	UTS (MPa)	Elongation (%)
AA8006	As-rolled	165	190	1.0
AA8006	Plant annealed	98	115	2.7
8006 + 0.2 Mg	As-rolled	231	255	0.6
8006 + 0.2 Mg	Plant annealed	102	123	1.9

Table 3 -

14 µm Foil		
Compound	Volume % ± 15%	
	As-rolled	Annealed
Cube {001}<100>	2.2	2.8
Goss (110)<001>	3.2	2.4
Copper {112}<111>	20.2)	25.3)
S {123}<634>	35.2) 72.5	39.5) 79.2
Brass {011}<211>	17.1)	14.4)
Random	22.1	15.6

Claims

1. Aluminium foil composed of an alloy of composition in wt %:

Fe	1.2-2.0%
Mn	0.2 - 1.0%
Mg and/or Cu	0.1 - 0.5%
Si	up to 0.4%
Zn	up to 0.1%
Ti	up to 0.1%
balance Al of at least commercial purity	

which foil has an average grain size below 5 µm after final annealing.

2. Aluminium foil composed of an alloy of composition in wt %:

Fe	1.2-2.0%
Mn	0.2 - 1.0%
Mg and/or Cu	0.1 - 0.5%
Si	up to 0.4%
Zn	up to 0.1%
Ti	up to 0.1%
balance Al of at least commercial purity,	

produced by rolling followed by final anneal, wherein at least 50% by volume of the as-rolled texture is retained after the final anneal.

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3. Aluminium foil composed of an alloy of composition in wt %:

Fe	1.2-2.0%
Mn	0.2 - 1.0%
Mg and/or Cu	0.1 - 0.5%
Si	up to 0.4%
Zn	up to 0.1%
Ti	up to 0.1%
balance Al of at least commercial purity,	

wherein the crystallographic texture of the final annealed product is a retained rolling structure.

4. Aluminium foil as claimed in any one of claims 1 to 3, wherein the foil thickness is 40 µm or less.

5. Aluminium foil as claimed in any one of claims 1 to 4, wherein the alloy composition is

Fe	1.4-1.8%
Mn	0.3 - 0.6%
Fe+Mn	1.8-2.15%
Mg	0.15-0.35%
Si	up to 0.4%
balance Al of at least commercial purity.	

6. A method of making the aluminium foil of any one of claims 1 to 5, which method comprises providing a billet of required composition, converting the billet to foil, and final annealing at a temperature of 220°C-300°C.

Patentansprüche

1. Aluminiumfolie, bestehend aus einer Legierung der Zusammensetzung in Gew.-%:

Fe	1,2 - 2,0 %
Mn	0,2 - 1,0 %
Mg und/oder Cu	0,1 - 0,5 %
Si	bis zu 0,4 %
Zn	bis zu 0,1 %
Ti	bis zu 0,1 %
Restanteil Al von wenigstens handelsüblicher Reinheit,	

wobei die Folie eine mittlere Korngröße von unterhalb 5 µm nach dem Fertigglühen aufweist.

2. Aluminiumfolie, bestehend aus einer Legierung der Zusammensetzung in Gew.-%:

Fe	1,2 - 2,0 %
Mn	0,2 - 1,0 %
Mg und/oder Cu	0,1 - 0,5 %
Si	bis zu 0,4 %
Zn	bis zu 0,1 %
Ti	bis zu 0,1 %
Restanteil Al von wenigstens handelsüblicher Reinheit,	

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hergestellt durch Walzen, gefolgt von Fertigglühen, wobei wenigstens 50 Vol.-% der Textur im Walzzustand nach dem Fertigglühen beibehalten ist.

3. Aluminiumfolie, bestehend aus einer Legierung der Zusammensetzung in Gew.-%:

Fe	1,2 - 2,0 %
Mn	0,2 - 1,0 %
Mg und/oder Cu	0,1 - 0,5 %
Si	bis zu 0,4 %
Zn	bis zu 0,1 %
Ti	bis zu 0,1 %
Restanteil Al von wenigstens handelsüblicher Reinheit,	

wobei die kristallographische Textur des fertiggeglühten Produktes eine beibehaltene Walzstruktur ist.

4. Aluminiumfolie gemäß einem der Ansprüche 1 bis 3, wobei die Foliendicke 40 µm oder weniger beträgt.

5. Aluminiumfolie gemäß einem der Ansprüche 1 bis 4, wobei die Legierungszusammensetzung ist:

Fe	1,4 - 1,8 %
Mn	0,3 - 0,6 %
Fe + Mn	1,8 - 2,15 %
Mg	0,15 - 0,35 %
Si	bis zu 0,4 %
Restanteil Al von wenigstens handelsüblicher Reinheit.	

6. Verfahren zur Herstellung der Aluminiumfolie gemäß einem der Ansprüche 1 bis 5, wobei das Verfahren das Bereitstellen eines Barrens der erforderlichen Zusammensetzung, die Konvertierung des Barrens zu Folie und das Fertigglühen bei einer Temperatur von 220 bis 300°C umfaßt.

Revendications

1. Feuille d'aluminium comprenant un alliage de composition, en pour cent en poids :

Fe	1,2 - 2,0 %
Mn	0,2 - 1,0 %
Mg et/ou Cu	0,1 - 0,5 %
Si	jusqu'à 0,4 %
Zn	jusqu'à 0,1 %
Ti	jusqu'à 0,1 %

le reste d'Al au moins de pureté commerciale, cette feuille ayant une taille moyenne de grain inférieure à 5 µm après recuit final.

2. Feuille d'aluminium comprenant un alliage de composition, en pour cent en poids :

Fe	1,2 - 2,0 %
Mn	0,2 - 1,0 %
Mg et/ou Cu	0,1 - 0,5 %
Si	jusqu'à 0,4 %
Zn	jusqu'à 0,1 %

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(suite)

Ti	jusqu'à 0,1 %
----	---------------

5 le reste d'Al au moins de pureté commerciale, produite par laminage puis recuit final, au moins 50 % en volume de texture telle que laminée, étant conservés après le recuit final.

3. Feuille d'aluminium comprenant un alliage de composition, en pour cent en poids :

10	Fe	1,2 - 2,0 %
	Mn	0,2 - 1,0 %
	Mg et/ou Cu	0,1 - 0,5 %
	Si	jusqu'à 0,4 %
15	Zn	jusqu'à 0,1 %
	Ti	jusqu'à 0,1 %

le reste d'Al d'au moins de pureté commerciale, la texture cristallographique du produit recuit final étant une structure de laminage conservée.

20 4. Feuille d'aluminium selon l'une quelconque des revendications 1 à 3, dans laquelle l'épaisseur de la feuille est de 40 µm ou moins.

5. Feuille d'aluminium selon l'une quelconque des revendications 1 à 3, dans laquelle la composition de l'alliage est :

25	Fe	1,4 - 1,8 %
	Mn	0,3 - 0,6 %
	Fe + Mn	1,8 - 2,15 %
	Mg	0,15 - 0,35 %
30	Si	jusqu'à 0,4 %

le reste d'Al d'au moins une pureté commerciale.

35 6. Procédé de fabrication de la feuille d'aluminium selon l'une quelconque des revendications 1 à 5, selon lequel on prépare une billette de composition requise, on convertit la billette en feuille, et on effectue un recuit final à une température de 220 °C à 300 °C.

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50

55

Fig.1.

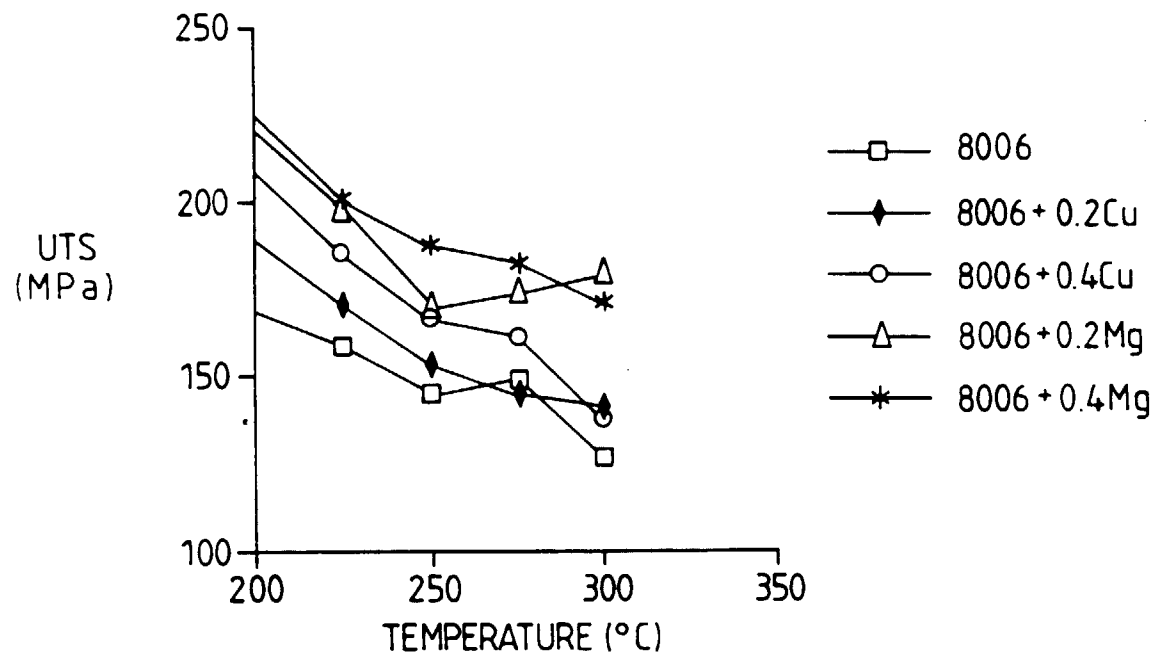


Fig.2.

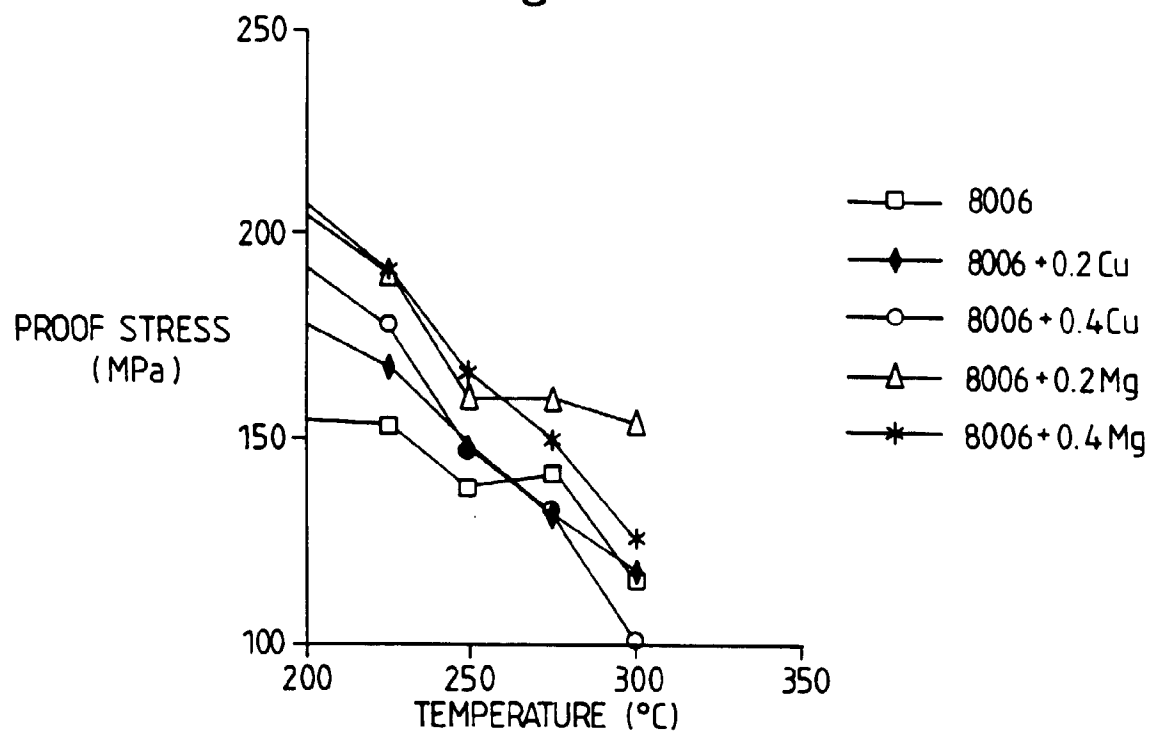


Fig.3.

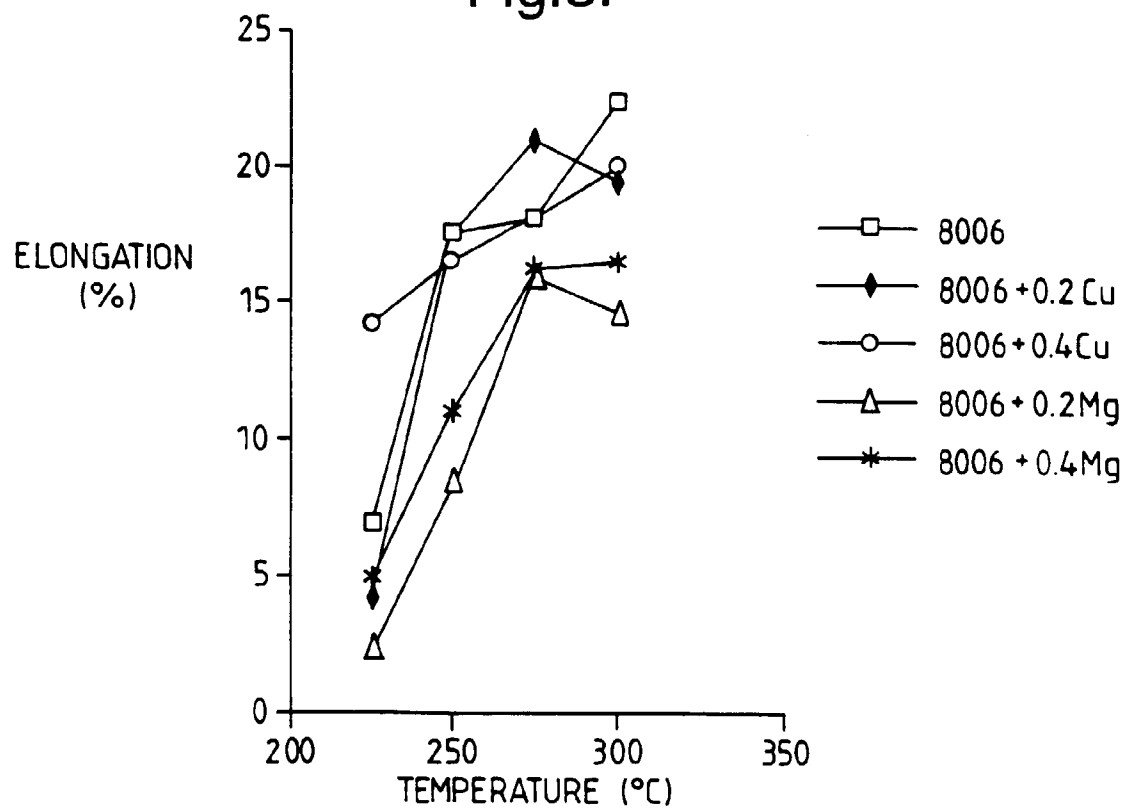


Fig.4B.

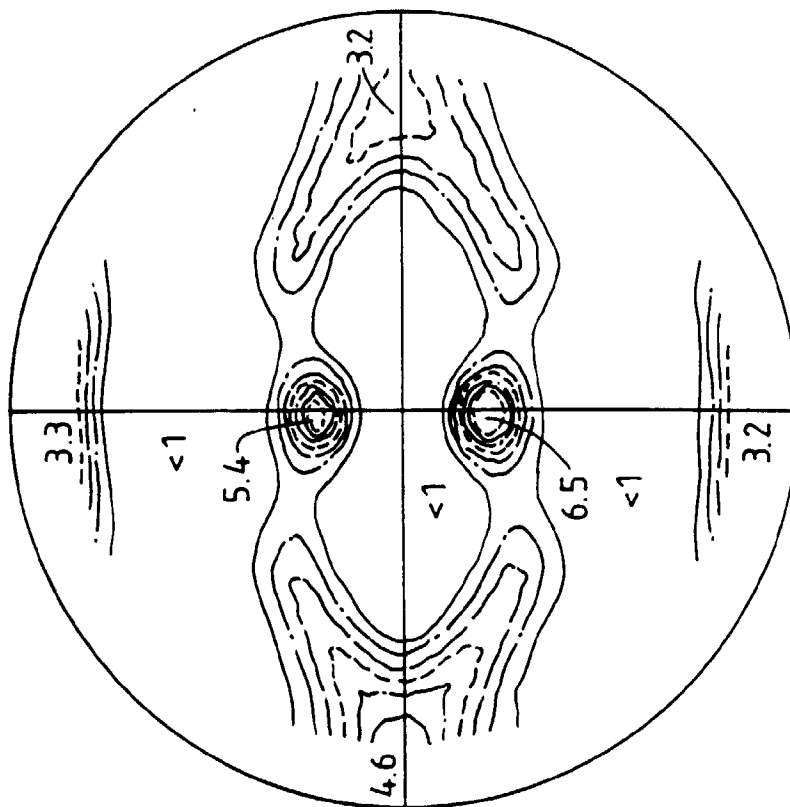


Fig.4A.

