



(86) Date de dépôt PCT/PCT Filing Date: 2002/02/13
(87) Date publication PCT/PCT Publication Date: 2002/08/22
(45) Date de délivrance/Issue Date: 2007/11/13
(85) Entrée phase nationale/National Entry: 2003/07/15
(86) N° demande PCT/PCT Application No.: CA 2002/000169
(87) N° publication PCT/PCT Publication No.: 2002/064848
(30) Priorité/Priority: 2001/02/13 (US09/782,797)

(51) Cl.Int./Int.Cl. *C22C 21/00* (2006.01),
B22D 11/00 (2006.01), *C22F 1/04* (2006.01)

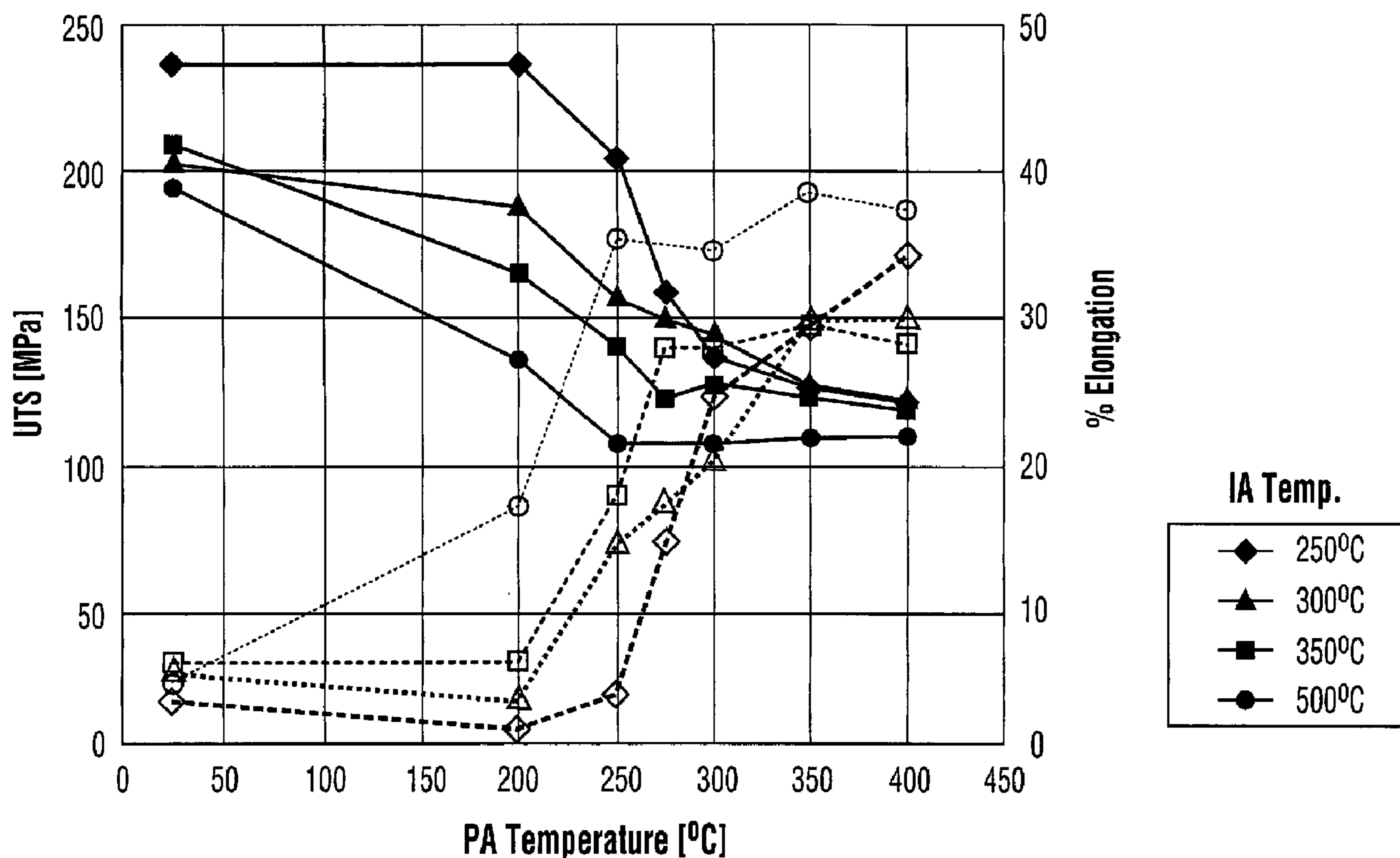
(72) Inventeurs/Inventors:
JIN, ILJOON, CA;
GATENBY, KEVIN MICHAEL, CA;
GABRYEL, CHRISTOPHER, CA

(73) Propriétaire/Owner:
NOVELIS, INC., CA

(74) Agent: KIRBY EADES GALE BAKER

(54) Titre : PRODUCTION DE FEUILLES D'ALLIAGE EN ALUMINIUM A HAUTE RESISTANCE

(54) Title: PRODUCTION OF HIGH STRENGTH ALUMINUM ALLOY FOILS



(57) Abrégé/Abstract:

An aluminum alloy foil is formed from an alloy containing about 1.2 to 1.7% by weight iron, about 0.4 to 0.8% by weight silicon and about 0.07 to 0.20% by weight manganese, with the balance aluminum and incidental impurities. The alloy is continuously strip cast, e.g. on a belt caster, to form a strip having a thickness of less than about 25 mm, which is then cold rolled to interanneal gauge followed by interannealing at a temperature of about 280 to 350°C. The interanneal strip is cold rolled to final gauge and further annealed to form the final foil product, having high strength and excellent quality.

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
22 August 2002 (22.08.2002)

PCT

(10) International Publication Number
WO 02/064848 A1

(51) International Patent Classification⁷: **C22C 21/00**,
B22D 11/00, C22F 1/04

(74) Agents: **EADES, Norris, M.** et al.; Kirby Eades Gale
Baker, Box 3432, Station D, Ottawa, Ontario K1P 6N9
(CA).

(21) International Application Number: PCT/CA02/00169

(22) International Filing Date: 13 February 2002 (13.02.2002)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
09/782,797 13 February 2001 (13.02.2001) US

(63) Related by continuation (CON) or continuation-in-part
(CIP) to earlier application:
US 09/782,797 (CIP)
Filed on 13 February 2001 (13.02.2001)

(71) Applicant (*for all designated States except US*): **ALCAN
INTERNATIONAL LIMITED** [CA/CA]; 1188 Sher-
brooke Street West, Montreal, Québec H3A 3G2 (CA).

(72) Inventors; and

(75) Inventors/Applicants (*for US only*): **JIN, Iljoon**
[CA/CA]; 696 Sussex Boulevard, Kingston, Ontario K7M
5B1 (CA). **GATENBY, Kevin** [GB/CA]; 1084 Old Colony
Road, Kingston, Ontario K7P 1M5 (CA). **GABRYEL,
Christopher** [CA/CA]; 2625 McIvor Road, Kingston,
Ontario K7L 4V4 (CA).

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU,
AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU,
CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC,
LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW,
MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG,
SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ,
VN, YU, ZA, ZM, ZW.

(84) Designated States (*regional*): ARIPO patent (GH, GM,
KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW),
Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM),
European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR,
GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent
(BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR,
NE, SN, TD, TG).

Published:

- with international search report
- before the expiration of the time limit for amending the
claims and to be republished in the event of receipt of
amendments

*For two-letter codes and other abbreviations, refer to the "Guid-
ance Notes on Codes and Abbreviations" appearing at the begin-
ning of each regular issue of the PCT Gazette.*

(54) Title: PRODUCTION OF HIGH STRENGTH ALUMINUM ALLOY FOILS

(57) Abstract: An aluminum alloy foil is formed from an alloy containing about 1.2 to 1.7% by weight iron, about 0.4 to 0.8% by weight silicon and about 0.07 to 0.20% by weight manganese, with the balance aluminum and incidental impurities. The alloy is continuously strip cast, e.g. on a belt caster, to form a strip having a thickness of less than about 25 mm, which is then cold rolled to interanneal gauge followed by interannealing at a temperature of about 280 to 350°C. The interanneal strip is cold rolled to final gauge and further annealed to form the final foil product, having high strength and excellent quality.



WO 02/064848 A1

PRODUCTION OF HIGH STRENGTH ALUMINUM ALLOY FOILS

Technical Field

This invention relates to the production of high strength aluminum alloy foil products. Specifically, it
5 relates to a process for manufacturing a new aluminum alloy foil using a continuous belt casting process.

Background Art

Thin gauge foils are generally prepared by casting an ingot of an aluminum alloy in a process known as DC
10 or direct chill casting. The ingots are generally heated to a high temperature, hot rolled to a re-roll gauge thickness of between 1 and 10 mm, then cold rolled to a "foil-stock" gauge typically 0.2 to 0.4 mm thick. The strip is often subjected to an interanneal step
15 during the cold rolling process. The "foil-stock" may be subject to further cold rolling operations, to produce a final foil thickness of about 5 to 150 microns.

There is a cost advantage to using continuous strip
20 casting as the starting point in manufacture of such foils since homogenization prior to hot rolling is not required, and the amount of hot reduction to form re-roll gauges is greatly reduced. Where high volume continuous casting is required, twin belt casting is the
25 preferred method of continuous casting. However, continuous strip casting processes apply different cooling conditions during solidification from those in DC casting, and there is an absence of a high temperature homogenization step prior to hot rolling.

Consequently when continuous strip casting processes are used with alloys normally prepared by DC casting and homogenization, this results in the formation of different intermetallic species. In continuous strip casting, the cooling rate of the strip during casting is generally higher (in some cases much higher) than the cooling rate in large DC ingots. Thus, such alloys processed in a continuous strip casting process also result in foil stock which has a higher supersaturation of solute elements, and therefore has undesirable hardening and softening properties, resulting in difficulties in rolling the foil stock to the final gauge thickness and in controlling the properties of the final gauge produced.

There is a particularly strong interest in producing what are referred to as "ultra high strength foils", i.e. a class of foils having an ultimate tensile strength (UTS) level of 130 MPa or higher. This strength is much higher than the strength of common AA1xxx alloy foils (60-90 MPa) or that of higher strength AA8021-type alloy foils (90-120 MPa). In one method of production of ultra high strength foils, AA8006-type alloys are cast on a twin roll caster and the roll cast materials are processed following specifically tailored processing routes. An AA8006-type alloy has the nominal composition of less than 0.4% by weight silicon, 1.2 to 2.0% by weight percent iron and 0.3 to 1.0% by weight manganese, with the balance aluminum and usual impurities. When the same AA8006 alloy is cast on a belt caster, the resulting strip does not have the same microstructure as that of the twin

roll cast strip. For instance, severe shell distortion occurs generating a wide variety of intermetallic sizes and concentrations that negatively affect microstructure control. Therefore, the final anneal cannot produce the
5 desired structure. Thus, it has not been possible to produce ultra high strength foils using the belt casting route.

A process for producing high strength aluminum foil using twin roll casting is described in Furukawa Alum
10 Japanese Patent JP1034548. That process used an aluminum alloy containing 0.8 to 2 wt.% Fe, 0.1 to 1 wt.% Si, 0.01 to 0.5 wt.% Cu, 0.01 to 0.5 wt.% Mg and 0.01 to 1 wt.% Mn. Ti and B were also included at grain refining levels. The alloy was twin roll cast to a
15 thickness of 0.5 to 3 mm and rolled to foil. A heat treatment at 200 to 450°C was also included.

In Mitsubishi, Japanese Patent Publication H3-153835 a fin material is described that was made from an Al-Fe-Si-Mn alloy. The alloy was cast to a thickness of
20 30 mm, hot rolled and cold rolled with interanneal, but with no final anneal.

Alcoa, U.S. Patent 5,380,379 describes the production of a foil from an aluminum alloy containing about 1.35 to 1.6 wt.% iron, about 0.3 to 0.6 wt.%
25 manganese, about 0.1 to 0.4 wt.% copper, about 0.05 to 0.1 wt.% titanium, about 0.01 to 0.02 wt.% boron, up to about: 0.2 wt.% silicon, 0.02 wt.% chromium, 0.005 wt.% magnesium and 0.05 wt.% zinc using a twin roll caster. The alloy was cast and then heat treated at a
30 temperature of about 460 to 500°C before cold rolling.

Another process for producing aluminum foil is described in Showa, Japanese Patent JP62250144. Here an aluminum alloy was used containing 0.7-1.8 wt.% Fe, 0.2 to 0.5 wt.% Si and 0.1 to 1.5 wt.% Mn. The procedure
5 involved direct chill casting, homogenization and hot rolling prior to the cold roll step.

In Swiss Aluminum, U.S. Patent 4,671,985 an aluminum foil is described containing 0 to 0.5 wt.% Si, 0.8 to 1.5 wt.% Fe and 0 to 0.5 wt.% Mn. After being
10 strip cast it was hot rolled, followed by cold rolling without interanneal.

WO 98 45492 describes an aluminum foil made from an aluminum alloy containing 0.2 to 0.5 wt.% Si, 0.4 to 0.8 wt.% Fe, 0.1 to 0.3 wt.% Cu and 0.05 to 0.3 wt.% Mn.
15 The alloy was continuously cast, cold rolled, interannealed at a temperature of 250 to 450°C, cold rolled to final gauge and final annealed at about 330°C.

It an object of the present invention to produce using continuous strip casting a novel high strength
20 aluminum foil having properties equivalent to high strength foil produced by direct chill or twin roll casting of AA8006.

It is a further objective to produce a high strength alloy by a continuous casting route capable of
25 high volume production rates.

Disclosure of the Invention

In accordance with the present invention, the problem of producing a high strength aluminum alloy foil using a continuous strip caster has been solved by way
30 of a new alloy composition and a new processing route.

Thus, the alloy that is used is one containing 1.2 to 1.7 wt.% Fe, 0.4 to 0.8 wt.% Si and 0.07 to 0.20 wt.% Mn, with the balance aluminum and incidental impurities. The above alloy is then cast in a continuous strip
5 caster to a strip thickness of less than about 25 mm, preferably about 5 to 25 mm, followed by cold rolling to interanneal gauge. The interannealing is carried out at a temperature in the range of about 280 to 350°C, followed by cold rolling to final gauge and final
10 anneal.

The interanneal is typically continued for about 2 to 8 hours, and the final anneal is preferably at a temperature of about 250 to 300°C for about 1 to 6 hours. The continuous strip casting is preferably
15 conducted on a belt caster and the interanneal gauge is typically about 0.5 to 3.0 mm.

In the above alloy, the Si content was increased and the Mn content was decreased as compared to the traditional AA8006 alloy. This solved local non-uniform
20 cooling problems encountered with AA8006 alloy and a stable recovered structure was obtained by a carefully selected interanneal temperature range. The grain size of the stable recovered structure is typically in the 1 to 7 μm range.

25 Fe in the alloy is a strengthening element, forming intermetallic particles during casting (which typically break down into smaller particles during rolling) and dispersoids during subsequent heat treatments (typically fine particles 0.1 micron or less in size) during the
30 process. These particles stabilize the subgrains in the final anneal process. If Fe is less than 1.2 wt.%, the

effect of Fe is not sufficient to make a strong foil,
and if Fe exceeds 1.7 wt.%, there is a danger of forming
large primary intermetallic particles during casting
which are harmful for rolling and the quality of the
5 foil products.

Si in the alloy improves castability in the casting
stage and the uniformity of the cast structure. It also
accelerates the precipitation of dissolved solute
elements during the annealing stage. If Si is less than
10 0.4 wt.%, casting is difficult and the cast structure
becomes less uniform. If the Si is more than 0.8 wt.%,
the recrystallization temperature is lowered and the
final anneal temperature range becomes too narrow.

Mn in the alloy is required to control the recovery
15 process and hence the grain size of the foil after the
final anneal. If Mn is less than 0.07 wt.%, the effect
of the element is insufficient and a stable recovered
structure cannot be obtained. If the Mn exceeds 0.20
wt.%, the ductility of the material after the final
20 anneal becomes too low.

Although the continuously cast strip may have an
as-cast thickness of up to 25 mm and be hot rolled to a
gauge of about 1 to 5 mm before cold rolling to the
intermediate gauge at which interannealing takes place,
25 according to a preferred procedure, a strip is
continuously cast to a thickness of no more than 10 mm,
most preferably 5 to 10 mm. A strip of this thickness
does not require any hot rolling prior to cold rolling.
The strip is preferably brought to a thickness of about
30 0.5 to 0.8 mm during cold rolling.

It is preferred that the strip be continuously cast in a belt caster. Belt casting is a form of continuous strip casting carried out between moving flexible and cooled belts. Although the belts may exert a force on the strip to ensure adequate cooling, preferably the force is insufficient to compress the strip while it is solidifying. Typically a belt caster will cast strips less than about 25 mm thick and preferably greater than about 5 mm thick. The cooling rate for casting alloys of the present invention generally lies between about 20 and 300°C/sec.

The continuously cast strip must not be homogenized before any subsequent rolling step as this has the effect of lowering the UTS obtainable in the final foil material.

Brief Description of the Drawings

Fig. 1 is a graph relating strength and elongation to partial anneal temperature for an alloy of the invention;

Fig. 2 shows transmission electron micrographs of foils produced from alloys of the invention with variable interanneal temperatures and a final anneal temperature of 300°C;

Fig. 3 shows transmission electron micrographs of foils produced from different alloys of the invention with an interanneal temperature of 300°C and a final anneal temperature of 300°C; and

Fig. 4 shows transmission electron micrographs of foils produced from an alloy of the invention with an

interanneal temperature of 300°C and varying final anneal temperatures.

Best Modes for Carrying Out the Invention

Example 1

- 5 A series of tests were conducted on a laboratory belt caster. The alloys used are shown in Table 1 below:

TABLE 1

Cast No.	Chemical Composition (wt.%)			
	Fe	Si	Mn	Comment
1	1.54	0.47	-	Mn too low
2	1.55	0.46	0.09	Within inventive range
3	1.16	0.48	0.20	Iron too low
4	1.48	0.78	0.10	Within inventive range
5	1.47	0.25	0.10	Si too low
6	0.91	0.45	0.09	Fe too low

- 10 The as-cast strips were nominally 7.3 mm thick, and all casts were free of shell distortion. Casting was done on a twin belts caster with heat fluxes in the range 1.5 to 3.8 MW/m². This corresponds to an average cooling rate through the cast strip of between 150 and 420°C/s.

- 15 Samples of all as-cast strips were taken, cut, polished and anodized in a sulphuric acid solution. The results showed that alloys 1,2,3,4,and 6 were structurally homogeneous, but that alloy 5 showed a non-homogeneous cast structure (different intermetallic particles were formed during solidification from one location to another). This alloy was therefore not
20 processed further.

To examine the effect of interanneal practice and alloy composition on the partial anneal response of the materials in the final annealing step, in particular to see if the materials develop a stable recovery regime in the temperature range 250 to 300°C, all cast strips (except for Cast No. 5) were processed and tensile tested as follows:

As-cast (7.3 mm)
↓
Cold rolled to 3 mm
↓
Interannealed at 250 to 500°C for 4 hours
↓
Cold rolled to 0.3 mm
↓
Final annealed at 250 to 400°C, and tensile tested

A typical example of the test results for Cast No. 2 is given in Figure 1. This shows partial anneal response curves of the alloy which was interannealed at 4 different temperatures. It is seen that the partial anneal response is very dependent on the interanneal practice used. When the interanneal temperature was lower than 250°C or higher than 350°C, the material did not develop any stable recovery regime, i.e., the tensile properties changed rapidly in the recovery temperature range. On the other hand, when the material was interannealed at 300°C, it developed a fairly stable recovery regime in the final anneal stage, i.e. the UTS values in the 250 to 300°C range did not change rapidly.

The tensile properties for a variety of alloys after final annealing at 250°C and 300°C, are shown in Table 2 below:

TABLE 2

Cast No.	Interanneal Temp. (°C)	Final Anneal Temp. (°C)	UTS (MPa)	Elong. (%)	UTS drop/MPa
1	250	250	179.9	8.5	60
		300	120.2	35.0	
1	300	250	144.1	19.5	28
		300	116.6	32.2	
1	350	250	117.6	30.7	2
		300	115.6	33.2	
2	250	250	204.3	3.8	69
		300	135.3	24.4	
2	300	250	158.3	14.5	14
		300	144.2	20.2	
2	350	250	139.2	17.7	14
		300	125.5	27.7	
3	250	250	192.6	4.0	36
		300	156.4	14.9	
3	300	250	170.8	10.0	13
		300	158.0	15.9	
3	350	250	162.2	12.9	23
		300	139.1	19.2	
4	250	250	189.2	9.1	46
		300	142.8	24.7	
4	300	250	159.5	16.3	11
		300	148.5	23.4	
4	350	250	152.6	18.1	17
		300	135.9	23.2	
6	300	250	152.4	11.9	20
		300	132.5	19.7	

5 The UTS drop shown in Table 2 is the strength decrease that occurs when the final anneal temperature is increased from 250 to 300°C. This is an indication of the strength stability during the final anneal in the temperature range. A good quality high strength foil
10 requires not only a high strength in the final product form, but also a good ductility and a good strength stability in the final anneal temperature range. Typically the strength after the final anneal should be

higher than 130 MPa, the ductility higher than 13% tensile elongation and the UTS drop less than 25 MPa over the 50°C temperature range.

The data in Table 2 shows that Cast No. 1 (an alloy without Mn) does not meet the good quality foil property criteria under any processing condition primarily because the strength is too low and the material does not possess the stable recovery regime in the final anneal stage, Cast No. 2 (Fe, Si and Mn within the inventive range) makes a good quality foil when the material is annealed at about 300°C, Cast No. 3 (Fe only slightly below the minimum) nearly meets the criteria when the material is interannealed at 300°C, Cast No. 4 meets the criteria with interanneals at both 300°C and 250°C, and Cast No. 6 (low Fe) does not produce good quality foil mainly because of the low ductility.

Thus the examples in Table 2 illustrate that a good quality high strength foil can be produced with the alloy composition and interanneal practice of the present invention.

Claims:

1. A process for producing a high-strength aluminum foil using a continuous strip caster, comprising the steps of:

5 (a) providing an alloy containing in percentage by weight about 1.2 to 1.7% iron, about 0.4 to 0.8% silicon, about 0.07 to 0.20% manganese and the balance being aluminum and incidental impurities,

(b) casting the alloy on a continuous strip caster
10 to form a cast strip having an as-cast thickness of less than about 25 mm,

(c) cold rolling the cast strip to interanneal gauge,

(d) interannealing the strip at a temperature of
15 about 280 to 350°C,

(e) cold rolling the interannealed strip to final gauge, and

(f) subjecting the final gauge strip to a final anneal.

20 2. The process according to claim 1 wherein the continuously cast strip has an as-cast thickness between about 5 to 10 mm.

3. The process according to claim 1 or 2 wherein the continuously cast strip is cast on a belt caster.

25 4. The process according to claim 1, 2 or 3 wherein the continuously cast strip has an as-cast thickness between about 5 to 25 mm and the as-cast strip is hot rolled prior to cold rolling.

5. The process according to any one of claims 1 to 4 wherein the cast strip is cold rolled to a thickness of about 0.5 to 3.0 mm.

6. The process according to claim 5 wherein the
5 cast strip is cold rolled to a thickness of about 0.5 to 0.8 mm.

7. The process according to any one of claims 1 to 6 wherein the interanneal at 280 to 350°C is conducted for about 2 to 8 hours.

10 8. The process according to claim 7 wherein the interanneal is conducted at a temperature of about 300°C.

9. The process according to any one of claims 1 to 8 wherein the final anneal is at a temperature of about 250 to 300°C.

15 10. The process according to claim 9 wherein the final anneal at 250 to 300°C is conducted for about 1 to 6 hours.

1/4

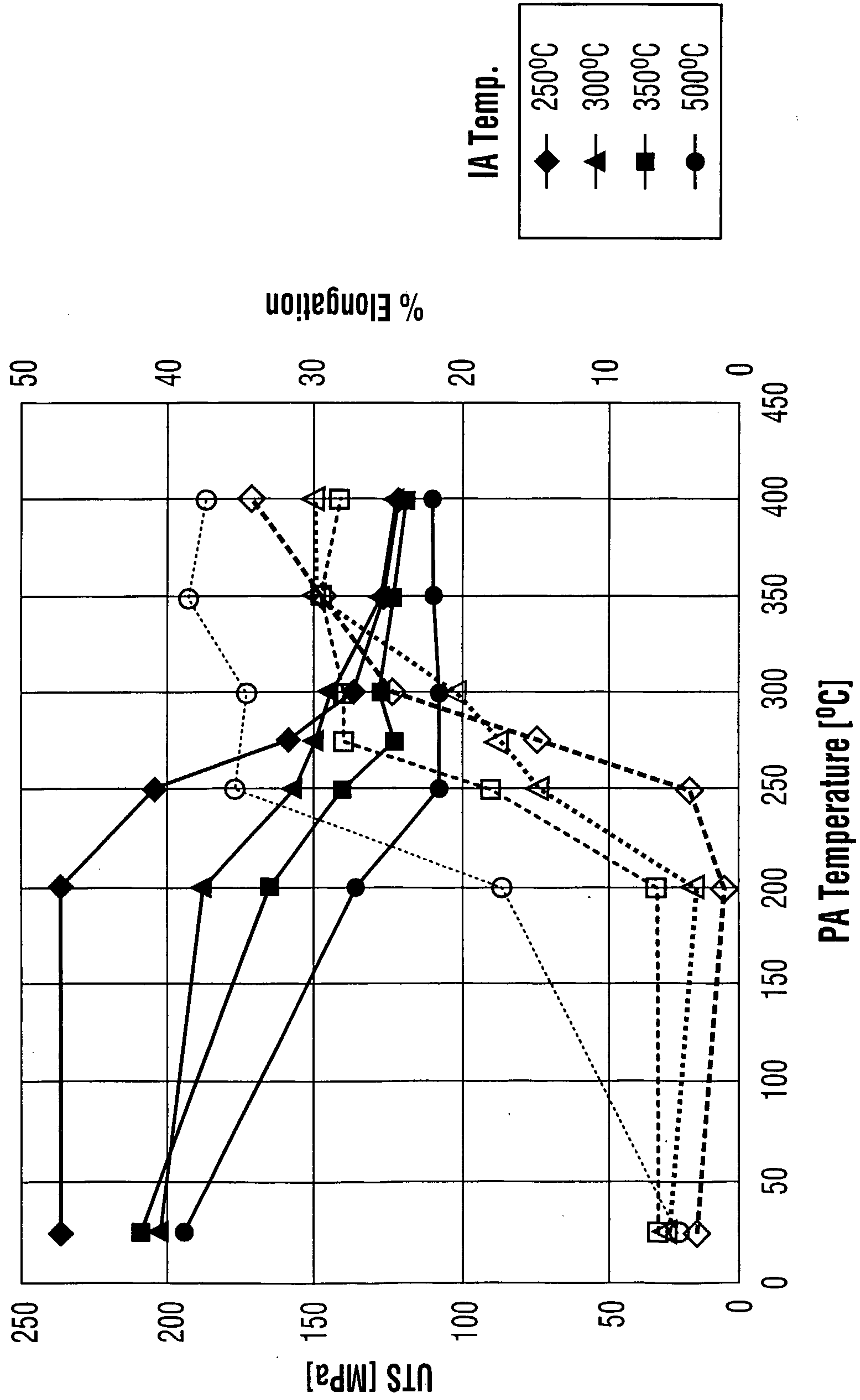


FIG. 1

2/4



IA @ 350°C



IA @ 300°C



IA @ 250°C

FIG. 2

3/4



TB2-436 (1.5 Fe-0.5 Si -0.1 Mn)



TB2-435 (1.5 Fe-0.5 Si)

FIG. 3

4/4



Final Anneal @ 400°C



Final Anneal @ 250°C

FIG. 4

