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(54) Title: PROCESS FOR PREPARING AN ALUMINUM ALLOY SHEET WITH IMPROVED BENDABILITY AND ALUMINUM ALLOY SHEET PRODUCED THEREFROM

(57) Abstract: A process is described for producing an aluminum alloy sheet having excellent bendability for use in forming panels for automobiles. An aluminum alloy is used comprising 0.5 - 0.75% by weight Mg, 0.7 - 0.85% by weight Si, 0.15 - 0.35% by weight Mn 0.1 - 0.3% by weight Fe, optionally 0.2 - 0.4% by weight Cu and the remainder Al and incidental impurities, and this alloy is cast into ingots by semi-continuous casting. The ingot is subjecting to hot rolling and cold rolling, followed by solution heat treatment of the formed sheet. This sheet material is then pre-aged by rapidly cooling from an initial pre-aging temperature of at least 80°C to room temperature at a cooling rate of more than 5°C/hour.

**PROCESS FOR PREPARING AN ALUMINUM ALLOY SHEET WITH  
IMPROVED BENDABILITY AND ALUMINUM ALLOY SHEET  
PRODUCED THEREFROM**

Technical Field

5        The present invention is directed to a process for preparing an aluminum alloy sheet having improved bendability and paint bake response. The invention is also directed to an aluminum alloy sheet obtained by the process.

Background Art

10      There is a continuing and growing need for improved aluminum alloys with improved properties particularly for use in the automotive industry. To be useful to the automotive industry, an aluminum alloy sheet product must possess good forming characteristics in the as-supplied temper so that it can be shaped and bent as desired. At the same time, the alloy product after shaping, painting and baking must have sufficient strength to resist dents and other impacts.

15      Aluminum alloys of the AA (Aluminum Association) 6000 series are desired to have low yield strength in the as-supplied temper and high yield strength in the finished product. The low yield strength in the as-supplied temper is desirable to obtain excellent formability and reduced springback, while high yield strength in the finished product is required for adequate dent 20 resistance at the lowest possible gauge for maximum weight savings.

United States Patent No. 5,266,130 Uchida et al., issued November 30, 1993 describes a process for manufacturing an aluminum alloy sheet material having good shape fixability and bake hardenability by regulating the heat pattern in the step of cooling after the solution heat treatment. The sheet is first 25 rapidly cooled to a quench temperature of 60 to 250°C and then further cooled at a rate based on the specific quench temperature. One aluminum alloy containing 0.8% Si, 0.7% Mg, 0.20% Mn and 0.15% Fe included a pre-aging treatment incorporating a cooling rate of 4°C/min from 150 to 50°C. Another alloy containing 0.8% Si, 0.7% Mg, 0.30% Cu, 0.10% Mn, 0.15% Fe, 0.02% Ti 30 and 20ppm B was subjected to the same pre-aging treatment.

United States Patent No. 5,616,189 Jin et al., issued April 1, 1997 describes an aluminum alloy containing magnesium, silicon and optionally copper in amounts suitable for the preparation of a sheet for use in the automotive industry. The patent also describes a process for preparing an 5 aluminum alloy sheet with suitable properties for use in the automotive industry. Among aluminum alloys tested were an alloy containing 0.30% Cu, 0.50% Mg, 0.70% Si, 0.05% Mn and 0.22% Fe and another alloy containing 0.29% Cu, 0.52% Mg, 0.68% Si, 0.07% Mn and 0.21% Fe. A sheet produced from these 10 alloys was subjected to a 5 hour pre-aging treatment at 85°C. The patent also states that sheet can be coiled at 85°C and allowed to cool slowly to ambient at a rate less than 10°C/hour.

It is an object of the present invention to provide an improved processing technique whereby an aluminum alloy sheet is formed which has excellent bendability and paint bake response.

15 The paint bake response of conventional AA (Aluminum Association) 6000 series aluminum alloys is complex. These alloys in the as-supplied condition contain a large number of fine clusters and zones uniformly distributed throughout the matrix. During a paint cure step, some fine unstable clusters and zones re-dissolve in the matrix, while others grow in size to 20 improve strength during hardening. The exact mechanism explaining how the bendability and paint bake response are improved is not entirely understood. It is believed that the process of the present invention slows the formation of clusters and zones and produces mostly those that do not re-dissolve during the paint cure step. Therefore, a large number of fine clusters and zones become 25 available for nucleation of the hardening particles and hence improve aging response.

#### Disclosure of the Invention

The alloys of the present invention are automotive aluminum alloys of AA6000 series containing (in percentages by weight) 0.50 – 0.75% Mg, 0.7 – 30 0.85% Si, 0.15 – 0.35% Mn, 0.1 – 0.3% Fe and the balance being aluminum and incidental impurities. Preferably, the alloy also contains 0.2 – 0.4% Cu.

The alloy is cast into ingots by semi-continuous casting, e.g. direct chill (DC) casting. The ingots are homogenized and hot rolled to reroll gauge, then cold rolled and solution heat treated. The heat treated sheet may be quenched to a desired initial pre-aging temperature. The sheet product thus obtained is 5 subjected to the pre-aging procedure of this invention and this pre-aging can be either the final step of the solution heat treatment stage or it can be part of a separate reheating step.

For the pre-aging, the sheet material starts with an initial pre-aging temperature which is at least 80°C and may be as high as 175°C or more. A 10 preferred initial pre-aging temperature is in the range of 95 to 200°C, more preferably 95 to 185°C. Starting from this pre-aging temperature, the sheet material is rapidly cooled to ambient, e.g. 25°C, at a rate of more than 5°C/hour. This cooling rate is preferably in the range of 10 to 600°C/hour.

The combination of excellent bendability and paint bake response is 15 achieved by (a) the specific composition of the alloy and (b) the appropriate pre-aging procedure. This slows the natural aging, stabilizes yield strength at lower values and significantly improves the paint bake response in the sheet compared with conventionally produced counterparts.

#### Brief Description of the Drawings

20 In the drawings which illustrate this invention:

Figure 1 shows the effect of cooling rate on yield strength (YS) for different pre-aging temperatures;

Figure 2 shows the effect of cooling rate on longitudinal bendability for different pre-aging temperatures; and

25 Figure 3 shows the effect of cooling rate on transverse bendability for different pre-aging temperatures.

#### Best Modes For Carrying Out The Invention

It is a specific objective of this invention to provide a sheet material having a low T4P yield strength and a high T8 yield strength. The low T4P 30 yield strength promotes improved formability, particularly hemming performance without cracking. The high T8 yield strength indicates a good

paint bake response, i.e. after painting and baking the sheet has sufficient strength to resist dents and withstand other impacts. For this purpose the target physical properties for the sheet products of this invention are as follows:

	T4P, YS	90 – 120 MPa
5	T4P, UTS	>200 MPa
	T4P, El	>28% ASTM, >30% (Using JIS Specimen)
	BEND, $r_{min}/t$	<0.5
	T8 (0% strain), YS	>210 MPa
	T8 (2% strain), YS	>220 MPa

10 The alloy used in this invention is cast by direct chill (DC) casting. The ingots are homogenized for more than 5 hours at a temperature of more than 550°C. The ingot is hot rolled to a reroll exit gauge of about 2.5 – 6mm at an exit temperature of about 300 – 380°C. The cold roll is to about 1mm gauge and the solution heat treatment is typically at a temperature of about 530 – 570°C.

15 When the procedure includes an interannealing step, the reroll sheet is cold rolled to an intermediate gauge of about 2.0 – 3.0mm. This intermediate sheet is batch annealed at a temperature of about 345 – 410°C and then further cold rolled to about 1.0mm.

#### Example 1

20 Alloys containing 0.6% Mg, 0.8% Si, 0.25% Fe and 0.20% Mn and with or without 0.25% Cu were cast as 95mm X 228mm ingots to carry out the experiments. The ingots were scalped, homogenized at 560°C for 6 hours, hot rolled to 3.5mm gauge, cold rolled to 2.1mm in one pass, batch annealed at 360°C for one hour and cold rolled to 0.93mm gauge. This sheet material was 25 solution heat treated at 560°C for 5 minutes.

The solution heat treated sheet material was pre-aged by cooling from different pre-aging temperatures, including 105°C, 125°C, 150°C and 175°C. Different cooling rates were used ranging from 1.25°C/hour to 600°C/hour. YS (yield strength), UTS (tensile strength), El (total elongation), n (strain hardening 30 index) and Bendability (r/t) were measured. This r/t ratio was determined from triplicate specimens according to the ASTM E 290C standard wrap bend test method. The minimum r/t value was obtained by dividing with the sheet

thickness, the minimum radius of the mandrel that produced a crack free bend. The radius of the mandrels used for the measurements were 0.025 mm, 0.057 mm, 0.076 mm, 0.102 mm, 0.152 mm, 0.203 mm, 0.254 mm, 0.305 mm, 0.406 mm, 0.508 mm, 0.610 mm, 0.711 mm, 0.813 mm, 1.02 mm, 1.22 mm, 5 1.42 mm and so on.

The measurements were made based on T4P temper with natural aging of two and four weeks. The term "P" means that the sheet material has been pre-aged. T8 represents the YS after a simulated paint bake of 2% strain and 30 minutes at 177°C.

10 Tables 1 and 2 show the mechanical properties for a sheet formed from an alloy containing 0.6% Mg, 0.8% Si, 0.25% Fe, 0.20% Mn and the balance Al and incidental impurities. From Table 1 (two weeks of natural aging) it can be seen that good combinations of low T4P yield strengths and high T8 yield strengths were obtained for a number of combinations of pre-aging temperatures 15 between 105°C and 175°C and cooling rates between 20 and 600°C/hour. Particularly good results were obtained by cooling from 125°C at 20°C/hour, 150°C at 60°C/hour and 175°C at 600°C/hour. Also shown in Tables 1 and 2 are results without a pre-age. The T8 properties are significantly reduced compared to the pre-age practice.

20 Table 2 is similar to Table 1 except that the samples were naturally aged for four weeks. The results are not significantly different from those of Table 1. The stability of properties over time is a particularly desirable feature.

Figures 1, 2 and 3 show the effects of cooling rates from different start of 25 cooling temperatures on the yield strength and bendability. Figure 1 shows that the use of slower cooling rates from high temperatures increases the yield strength in the T4P and T8 tempers due to artificial aging and affects bendability adversely. The best combination of properties is obtained with faster cooling rates from high start of cooling temperatures as seen in Table 1.

**Table 1.** Mechanical Properties of Al-0.6% Mg-0.8% Si-0.25% Fe-0.20% Mn Alloy Pre-aged in Different Conditions and Naturally Aged for 2 Weeks

Pre-aging Temp (°C)	Cooling rate (°C/h)	Temper	YS (MPa)	UTS (MPa)	% El	n	Bendability	
							L	T
No Pre-age	-	T4	115.2	234.8	28.1	0.28	0.21	0.21
105	1.25	T8	173.7	248.9	22.0	0.20	-	-
		T4P	131.6	243.8	23.9	0.26	0.33	0.28
	20	T8	242.0	299.6	19.1	0.15	-	-
		T4P	105.8	222.7	23.0	0.29	0.08	0.05
	60	T8	214.7	280.3	20.1	0.17	-	-
		T4P	108.6	226.0	21.3	0.29	0.08	0.08
125	1.25	T8	216.6	281.8	19.7	0.16	-	-
		T4P	171.1	268.4	21.8	0.21	0.64	0.43
	20	T8	264.2	310.7	17.6	0.12	-	-
		T4P	105.2	220.7	23.1	0.29	0.03	0.03
	60	T8	234.0	292.7	18.4	0.15	-	-
		T4P	107.1	225.0	23.5	0.29	0.08	0.03
150	1.25	T8	223.1	287.4	18.7	0.16	-	-
		T4P	246.9	304.9	15.8	0.12	1.54	1.55
	60	T8	296.8	322.0	13.2	0.08	-	-
		T4P	106.7	220.0	23.1	0.28	0.06	0.03
	240	T8	249.5	300.6	16.6	0.13	-	-
		T4P	112.5	223.3	22.4	0.28	0.08	0.08
175	1.25	T8	232.3	293.1	18.2	0.15	-	-
		T4P	298.0	325.5	10.6	0.06	2.67	2.29
	120	T8	312.3	324.0	8.5	0.05	-	-
		T4P	113.1	222.5	22.5	0.27	0.18	0.03
	600	T8	249.3	297.0	16.4	0.13	-	-
		T4P	106.2	220.1	24.9	0.28	0.03	0.03
		T8	245.9	295.9	17.1	0.13	-	-

**Table 2.** Mechanical Properties of Al-0.6% Mg-0.8% Si-0.25% Fe-0.2% Mn Alloy Pre-aged in Different Conditions and Naturally Aged for 4 Weeks

Pre-aging Temp. (°C)	Cooling rate (°C/h)	Temper	YS (MPa)	UTS (MPa)	%El	n	Bendability	
							L	T
No Pre-age	-	T4 T8	116.7 172.2	235.5 248.1	25.8 20.1	0.28 0.20	0.21	0.16
105	1.25	T4P T8	133.2 244.5	246.2 302.4	23.6 18.8	0.26 0.15	0.27	0.22
		T4P T8	114.0 215.2	231.7 281.4	23.9 20.1	0.28 0.17	0.11	0.05
	20	T4P T8	114.7 213.0	233.4 281.4	24.9 19.5	0.28 0.17	0.11	0.08
		T4P T8	172.2 268.0	268.9 313.9	22.8 17.2	0.21 0.12	0.54	0.44
	60	T4P T8	114.5 235.6	230.8 296.3	24.3 18.3	0.28 0.15	0.05	0.02
		T4P T8	115.3 223.1	233.2 287.7	24.9 18.9	0.28 0.16	0.08	0.03
125	1.25	T4P T8	251.1 298.3	312.2 323.6	17.3 13.4	0.12 0.08	1.52	1.52
		T4P T8	114.5 248.1	227.6 300.9	23.6 16.6	0.28 0.13	-	-
	20	T4P T8	118.5 230.5	234.9 290.4	24.3 18.3	0.28 0.16	0.21	0.08
		T4P T8	301.1 310.4	326.9 322.3	9.3 8.3	0.05 0.05	2.62	2.30
	60	T4P T8	120.3 253.9	225.8 301.5	22.7 16.6	0.26 0.12	0.11	0.03
		T4P T8	119.0 247.4	227.8 298.1	23.9 17.3	0.27 0.13	0.21	0.03
150	1.25	T4P T8P	310.4	322.3	8.3	0.05	-	-
		T4P T8	114.5 248.1	227.6 300.9	23.6 16.6	0.28 0.13	-	-
	20	T4P T8	118.5 230.5	234.9 290.4	24.3 18.3	0.28 0.16	-	-
		T4P T8	301.1 310.4	326.9 322.3	9.3 8.3	0.05 0.05	-	-
	60	T4P T8	120.3 253.9	225.8 301.5	22.7 16.6	0.26 0.12	-	-
		T4P T8	119.0 247.4	227.8 298.1	23.9 17.3	0.27 0.13	-	-
175	1.25	T4P T8P	310.4	322.3	8.3	0.05	-	-
		T4P T8	120.3 253.9	225.8 301.5	22.7 16.6	0.26 0.12	-	-
	20	T4P T8	118.5 230.5	234.9 290.4	24.3 18.3	0.28 0.16	-	-
		T4P T8	301.1 310.4	326.9 322.3	9.3 8.3	0.05 0.05	-	-
	60	T4P T8	120.3 253.9	225.8 301.5	22.7 16.6	0.26 0.12	-	-
		T4P T8	119.0 247.4	227.8 298.1	23.9 17.3	0.27 0.13	-	-

5 Tables 3 and 4 summarize the average tensile properties of the 0.25% Cu containing alloy after two and four weeks of natural aging. The trends obtained from this alloy are very similar to the Cu free alloy. Generally, the artificial aging response of the alloy is better and this translates into a higher yield strength, especially in situations where cooling is carried out from high  
10 temperatures. In general, the paint bake response and bendability following cooling from 125°C at 20°C/hour are excellent after two weeks of natural aging, although there is a slight deterioration after four weeks of natural aging.

**Table 3.** Mechanical Properties of Al-0.6% Mg-0.8% Si-0.3% Cu-0.25% Fe-0.20% Mn Alloy Pre-aged in Different Conditions and Naturally Aged for 2 Weeks

Pre-aging Temp. (°C)	Cooling rate (°C/h)	Temper	YS (MPa)	UTS (MPa)	% El	n	Bendability	
							L	T
No Pre-age	-	T4	122.5	252.8	25.8	0.29	0.27	0.16
105	1.25	T4P	146.5	270.8	25.7	0.26	0.56	0.41
		T8	263.1	325.7	19.9	0.15	-	-
	20	T4P	110.9	239.3	22.9	0.30	0.23	0.03
		T8	235.1	305.8	19.8	0.16	-	-
	60	T4P	116.3	245.5	25.9	0.29	0.28	0.09
		T8	235.5	305.2	19.5	0.16	-	-
125	1.25	T4P	216.8	316.6	21.1	0.19	1.12	1.12
		T8	290.8	339.3	17.6	0.12	-	-
	20	T4P	112.7	242.1	27.0	0.30	0.06	0.06
		T8	253.7	316.6	19.1	0.15	-	-
	60	T4P	116.4	246.6	25.2	0.29	0.28	0.08
		T8	244.1	309.8	18.1	0.15	-	-
150	1.25	T4P	269.7	340.8	16.0	0.12	2.36	1.78
		T8	314.2	347.4	14.2	0.09	-	-
	60	T4P	131.6	253.7	24.6	0.27	0.34	0.17
		T8	275.5	331.3	17.3	0.13	-	-
	240	T4P	121.3	248.1	25.8	0.28	0.28	0.08
		T8	247.4	312.2	19.1	0.15	-	-
175	1.25	T4P	306.0	351.3	12.3	0.08	2.67	2.34
		T8	334.0	350.1	10.3	0.06	-	-
	120	T4P	163.2	270.8	21.0	0.23	0.46	0.25
		T8	292.0	337.4	17.4	0.11	-	-
	600	T4P	142.4	253.9	22.6	0.25	0.28	0.18
		T8	280.4	331.7	16.5	0.12	-	-

**Table 4.** Mechanical Properties of Al-0.6% Mg-0.8% Si-0.3% Cu-0.25% Fe-0.20% Mn Alloy Pre-aged in Different Conditions and Naturally Aged for 4 Weeks

Pre-aging Temp. (°C)	Cooling rate (°C/h)	Temper	YS (MPa)	UTS (MPa)	% El	n	Bendability	
							L	T
No Pre-age	-	T4	127.0	260.0	26.6	0.28	0.33	0.22
105	1.25	T4P	149.4	273.6	25.9	0.26	0.56	0.28
		T8	264.7	327.9	19.7	0.15	-	-
	20	T4P	119.4	249.5	26.5	0.29	0.28	0.08
		T8	233.1	305.1	20.3	0.17	-	-
	60	T4P	121.7	250.6	25.5	0.29	0.23	0.08
		T8	222.0	291.2	20.1	0.17	-	-
125	1.25	T4P	216.9	317.2	21.6	0.19	1.12	1.12
		T8	294.1	342.6	17.9	0.12	-	-
	20	T4P	127.6	253.9	25.6	0.28	0.28	0.03
		T8	255.3	319.8	20.0	0.15	-	-
	60	T4P	124.0	253.4	25.3	0.28	0.28	0.03
		T8	240.5	309.2	20.1	0.16	-	-
150	1.25	T4P	270.3	342.5	16.5	0.12	2.29	1.74
		T8	317.3	350.0	14.7	0.09	-	-
	60	T4P	132.0	255.1	23.2	0.27	0.28	0.28
		T8	271.9	326.8	17.8	0.13	-	-
	240	T4P	127.7	255.6	26.4	0.28	0.33	0.17
		T8	251.3	314.2	18.3	0.15	-	-
175	1.25	T4P	308.3	352.8	12.2	0.08	2.68	2.35
		T8	335.9	351.6	10.5	0.06	-	-
	120	T4P	169.0	270.8	20.3	0.22	0.39	0.28
		T8	295.0	338.2	17.0	0.11	-	-
	600	T4P	151.0	255.3	21.5	0.23	0.27	0.16
		T8	292.5	337.0	15.4	0.11	-	-

5 The pre-aged sheet material obtained according to this invention can be coiled for future use. It is also possible to have the alloy sheet move directly from solution heat treatment to a cleaning bath where the rapid cooling pre-aging takes place.

10 It is further possible to conduct the pre-aging by starting with the pre-aging temperature and first naturally cooling the sheet in still air at a cooling rate of 1 – 1.5°C/hour and thereafter continuing with a rapid cooling in accordance with the pre-aging process described hereinbefore.

Claims:

1. A process for producing an aluminum alloy sheet having excellent bendability for use in forming panels for automobiles, the process comprising the steps of:

5 semi-continuously casting an aluminum alloy ingot comprising 0.5 - 0.75% by weight Mg, 0.7 - 0.85% by weight Si, 0.15 - 0.35% by weight Mn and 0.1 - 0.3% by weight Fe and the remainder Al and incidental impurities, subjecting the cast alloy ingot to hot rolling and cold rolling, followed by solution heat treatment of the formed sheet, and

10 pre-aging the sheet material by rapidly cooling from an initial pre-aging temperature of at least 80°C to room temperature at a cooling rate of more than 5°C/hour.

2. A process according to claim 1 wherein the alloy also contains from 0.2 - 0.4% by weight Cu.

15 3. A process according to claim 1 or 2 wherein the initial pre-aging temperature is in the range of about 95 to 185°C.

4. A process according to claim 1, 2 or 3 wherein the cooling rate is in the range of 10 to 600°C/hour.

5. A process according to claim 4 wherein the initial pre-aging 20 temperature is in the range of about 95 to 200°C and the cooling rate is in the range of 10 to 600°C/hour.

6. Aluminum alloy sheet material having improved bendability, produced by a process comprising the steps of:

25 semi-continuously casting an aluminum alloy comprising 0.50 to 0.75 by weight Mg, 0.7 to 0.85% by weight Si, 0.1 to 0.3% by weight Fe, 0.15 to 0.35% by weight Mn, and the balance Al and incidental impurities,

subjecting the cast alloy to hot rolling and cold rolling, followed by solution heat treatment of the formed sheet, and

pre-aging the sheet material by rapidly cooling from an initial pre-aging temperature of at least 80°C to room temperature at a cooling rate of more than 5°C/hour.

7. An aluminum alloy sheet material according to claim 6 wherein  
5 the alloy also contains 0.2 to 0.4% Cu.

8. An aluminum alloy sheet material according to claim 6 or 7 obtained by a process wherein the initial pre-aging temperature is in the range of about 95 to 200°C.

9. An aluminum alloy sheet material according to claim 6, 7 or 8  
10 obtained by a process wherein the sheet is cooled at a rate in the range of 10 to 600°C/hour.

10. An aluminum alloy sheet material according to any one of claims 6 - 9 wherein the sheet material has a bendability (r/t) value of less than 0.05.

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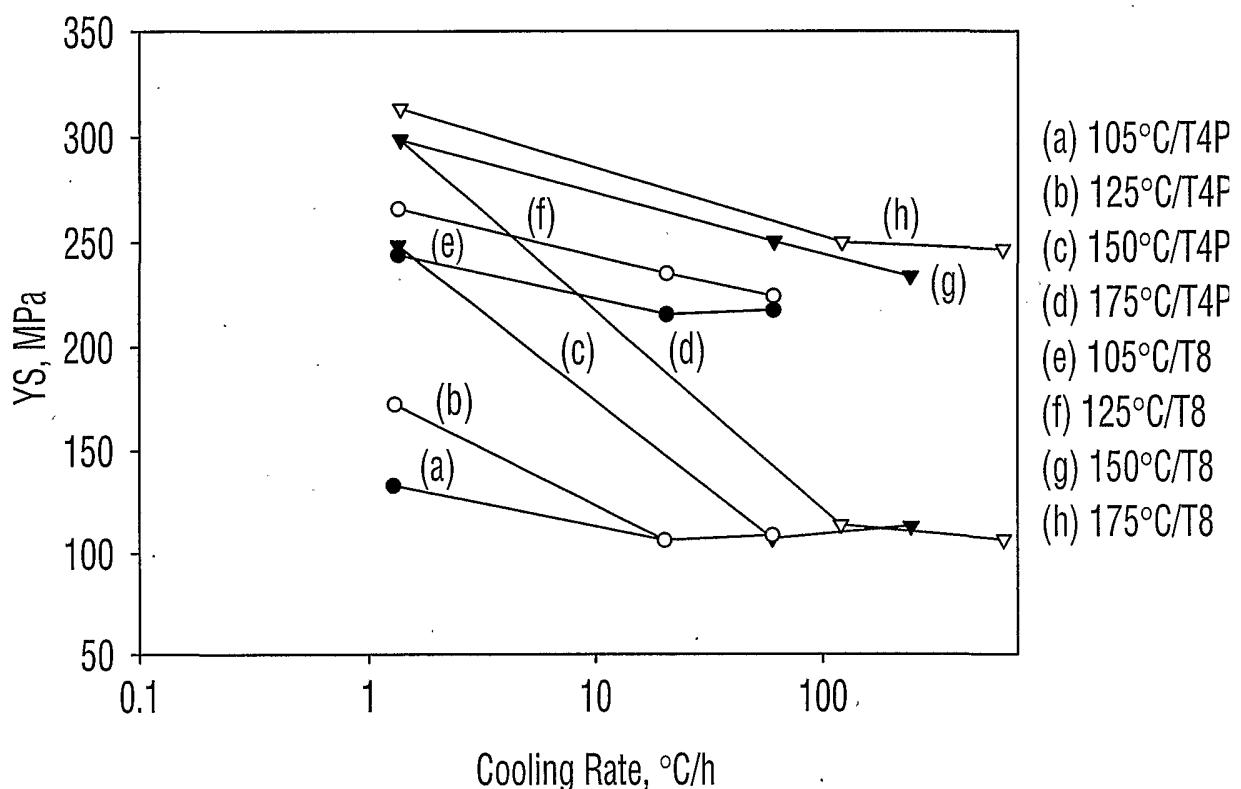
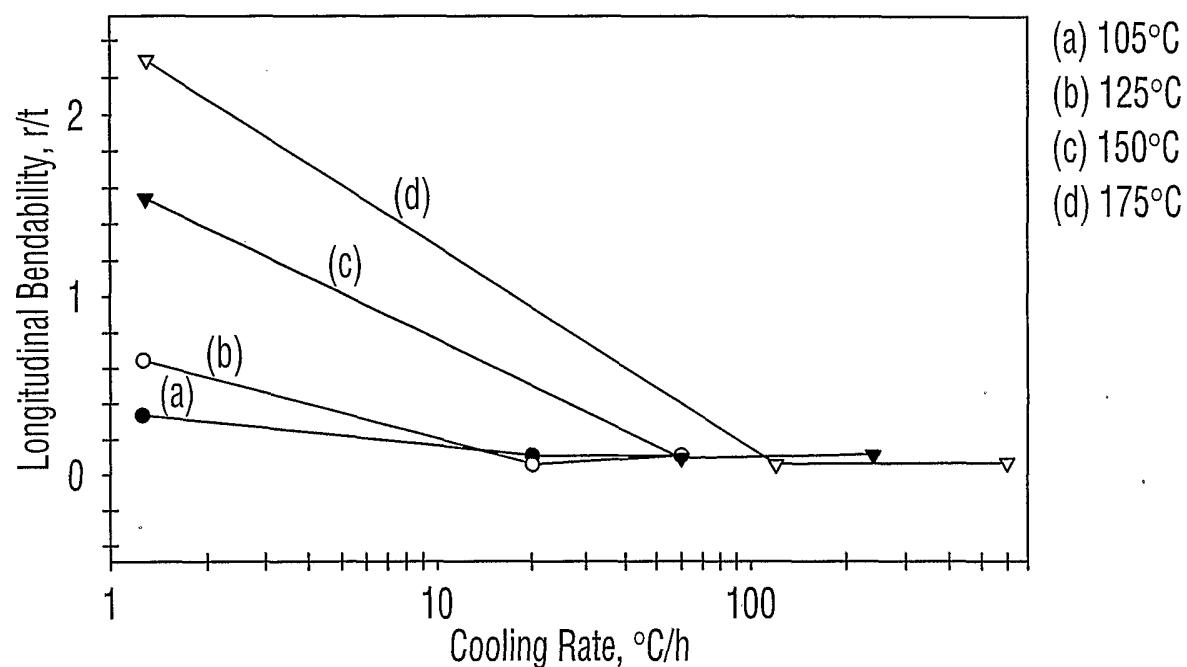
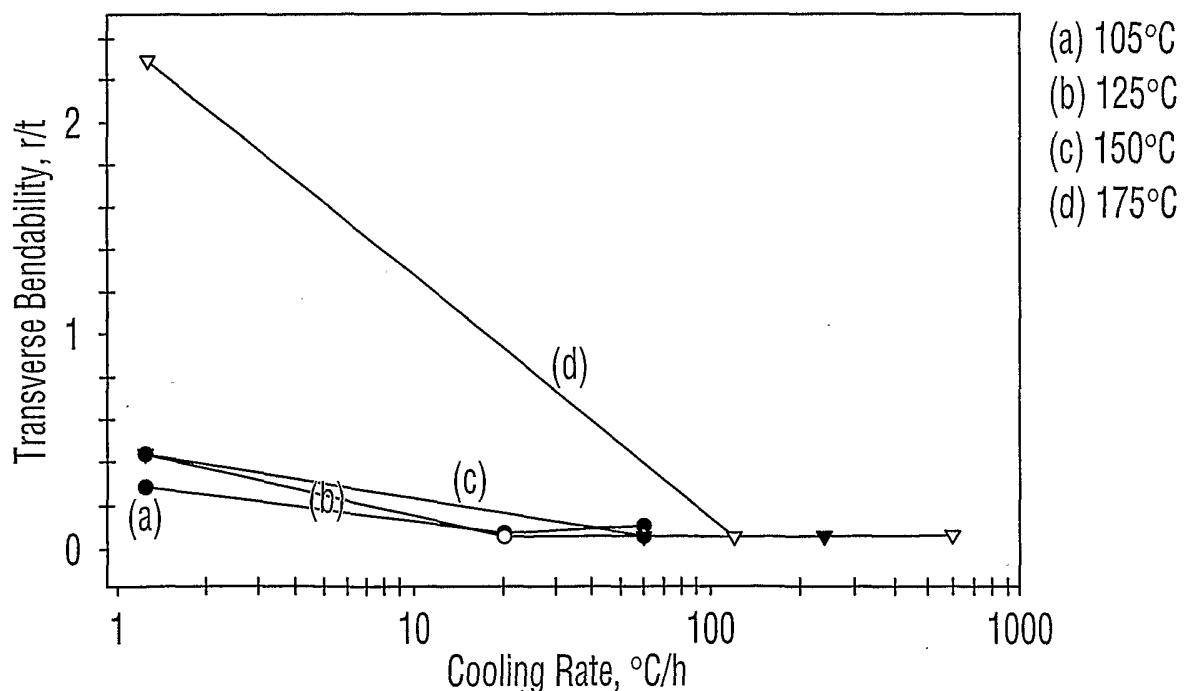


FIG. 1

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**FIG. 2****FIG. 3**

## INTERNATIONAL SEARCH REPORT

International Application No  
PCT/CA 02/00653A. CLASSIFICATION OF SUBJECT MATTER  
IPC 7 C22C21/02 C22C21/08 C22F1/05

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)  
IPC 7 C22C C22F

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

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

CHEM ABS Data, EPO-Internal

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	WO 96 07768 A (WHEELER MICHAEL J ;ALCAN INT LTD (CA); MAROIS PIERRE H (CA); GUPTA) 14 March 1996 (1996-03-14) page 9, line 35 -page 10, line 23; example AA6009; table 1 page 6, line 9 - line 4 page 10, line 24 -page 11, line 14 claims 1,5-7; figure 1 ---	6-10
A	page 6, line 9 - line 4 page 10, line 24 -page 11, line 14 claims 1,5-7; figure 1 ---	1-5

 Further documents are listed in the continuation of box C. Patent family members are listed in annex.

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Date of the actual completion of the international search  23 July 2002	Date of mailing of the international search report  31/07/2002
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer  Patton, G

## INTERNATIONAL SEARCH REPORT

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A	WO 98 37251 A (EVANS DANIEL RONALD ;LLOYD DAVID JAMES (CA); MAROIS PIERRE HENRY ()) 27 August 1998 (1998-08-27) page 5, line 21 -page 8, line 15 page 10, line 1 -page 11, line 20 claims 1,3,7-9,11-13 ----	1-10
A	US 4 808 247 A (KOMATSUBARA TOSHIO ET AL) 28 February 1989 (1989-02-28) example 12; table 1	6-10
A	column 15, line 1 - line 39; claims 1,6-8,13,17,20; figure 1; example 5; tables 9,10 ----	1-5
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