Abstract:
A powder mixture for use in forming a sintered aluminium alloy, the powder mixture having a metallic content comprising, by weight: 1.5 - less than 3.8 % copper 0.3 - 1.5 % silicon 0.1 - 1.5 % magnesium and optionally one or more from the group consisting of 0.01 - 0.5 % silver 0.01 - 0.5 % zirconium 0.01 - 0.5 % vanadium 0.01 - 1.0 % titanium 0.01 - 1.0 % chromium with the balance being aluminium except for incidental impurities.
Field of the Invention

The present invention relates to sintered aluminium alloys, in particular to a powder mixture for use in forming a sintered aluminium alloy and also to a method for producing an article from a sintered aluminium alloy.

Background of the Invention

One form of producing articles from metal alloys is by powder metallurgy. This process typically comprises mixing elemental powders or master alloy powders in accordance with the desired alloy composition, together with a lubricant wax, prior to pressing the powder in a die to produce a "green" article. The article is ejected from the die and then sintered in a furnace at elevated temperatures such that the particles are bonded together to form an alloy. A significant advantage of this process is that it is cost effective in producing complex parts at, or very close to, final dimensions at production rates which range from less than one hundred to several thousand parts per hour.

Sintered aluminium alloys have been produced, in particular for automotive components where it is desirable to incorporate light weight alloys. An early sintered aluminium alloy produced by Alcoa and commonly referred to as Alcoa-Alloy 201 has a composition of 4.4 wt % copper, 0.5 wt % magnesium and 0.8 wt % silicon with the balance being aluminium. A different sintered aluminium alloy is Ampalloy 2712, which is produced as a powder mixture by Ampal Inc having a composition of 3.8 wt % copper, 1.0 wt % magnesium, 0.7 wt % silicon and 0.1 wt % tin, with the balance being aluminium.

To date, sintered aluminium alloys have found limited use for automotive components. More specifically, sintered aluminium alloys are primarily used to
manufacture cam shaft bearing caps. One reason for this is that the sintered aluminium alloys are considered to have insufficient strength. Notably, for Ampalloy 2712, the 0.1 % by weight tin was added to improve the strength of the alloy. This reflects the general understanding that to improve sintered aluminium alloys so they are more suitable for wider use in the automotive industry, such alloys require enriching with further additives to increase their strength.

One consequence of enriching the sintered aluminium alloys is that the green pressed articles undergo a greater amount of shrinkage when being sintered in the furnace. This makes it harder to control the shape and dimensions of the finished article and furthermore may require additional processing of the article which increases the cost of production.

**Summary of the Invention**

According to a first aspect of the present invention, there is provided a powder mixture for use in forming a sintered aluminium alloy, the powder mixture having a metallic content comprising, by weight:

- 1.5 – less than 3.8 % copper
- 0.3 – 1.5 % silicon
- 0.1 – 1.5 % magnesium

and optionally one or more from the group consisting of:

- 0.01 – 0.2 % tin
- 0.1 – 0.5 % silver
- 0.01 – 0.5 % zirconium
- 0.01 – 0.5 % vanadium
- 0.01 – 1.0 % titanium
- 0.01 – 1.0 % chromium

with the balance being aluminium except for incidental impurities.

According to a second aspect of the present invention, there is provided a sintered aluminium alloy formed from a powder mixture, the alloy comprising, by
weight:

1.5 - less than 3.8 % copper
0.3 - 1.5 % silicon
0.1 - 1.5 % magnesium

and optionally one or more from the group consisting of

0.01 - 0.2 % tin
0.1 - 0.5 % silver
0.01 - 0.5 % zirconium
0.01 - 0.5 % vanadium
0.01 - 1.0 % titanium
0.01 - 1.0 % chromium

with the balance being aluminium except for incidental impurities.

It is noted that throughout the specification, all references to percentages are weight percentages.

The copper content of the mixture may be 2.0 to 3.0 %, preferably 2.2 to 3.0 %, more preferably approximately 2.6 %.

The silicon content of the mixture may be 0.7 to 1.3 %, preferably 0.7 to 0.9 %, more preferably approximately 0.8 %.

The magnesium content of the mixture may be 0.2 to 1.3 %, preferably 0.2 to 1.0 %, more preferably 0.7 to 0.8 %, more preferably the magnesium content is approximately 0.6 %. The magnesium content may be reduced during sintering of the powder mixture, such that the magnesium content in the alloy is lower than that of the mixture. Without wishing to be bound by theory, this is thought to occur because of evaporation of the magnesium during the sintering process.

The mixture may also comprise a lubricant wax, in an amount of 1.0 to 1.5 % relative to the total metallic content. The lubricant wax is for assisting in the attainment of a more uniform green density distribution and ejection from a die of green articles formed by pressing the powder mixture in the die. The lubricant wax is not present in the sintered alloy as it is completely
removed during the sintering step used in the process of forming the sintered aluminium alloy article.

The sintered aluminium alloy formed from the mixture may have a tensile strength of at least 170 MPa, preferably at least 185 MPa, more preferably at least 190 MPa.

The sintered aluminium alloy formed from the mixture may have a ductility of at least 1.5 %, preferably at least 1.8 %, more preferably at least 2.0 %.

The sintered aluminium alloy formed from the mixture may have a dimensional change ("dc") from die size after sintering of -1.5 % to +1.5 %, preferably -0.5 % to +0.5 %, more preferably -0.1 % to +0.1 %. Throughout the specification, the dimensional change ("dc") is defined by the following equation:

\[ dc = \frac{(L - L_0)}{L_0} \]

where \( L \) = the length of a rectangular specimen shaped in a rectangular die, after sintering; and \( L_0 \) = the length of the rectangular die used to form the specimen.

The powder mixture may optionally comprise an amount of any one or more of tin, silver, zirconium, titanium, chromium or vanadium.

Tin may be added to the powder mixture to improve the sintering response of the mixture, i.e. the density of the aluminium alloy after sintering.

Silver may be added to the powder mixture to improve the heat treatment response of the alloy formed from the mixture, i.e. the strength of the aluminium alloy after it has been subjected to post sintering heat treatment.

Zirconium, titanium, chromium and/or vanadium may be added to the powder mixture to control the grain size of the alloy formed from the mixture.
Iron and/or manganese may also be present in the powder mixture as incidental impurities. Typically, iron and/or manganese are impurities in the elemental aluminium powder.

Preferably, the iron content is less than 0.5%.
Preferably, the manganese content is less than 0.5%.

According to a third aspect of the present invention, there is provided a method for producing an article from a sintered aluminium alloy, the method comprising the steps of:

- providing a powder mixture according to the first aspect of the present invention;
- pressing the powder mixture in a die to form a green article;
- ejecting the green article from the die; and
- sintering the green article to produce the sintered aluminium alloy article.

The method may also comprise the step of heat treating the article after it has been sintered.

The step of heat treating may comprise solution treating, quenching and aging the article. This treatment of the article is used to harden the alloy.

The article may be quenched immediately after the step of sintering, i.e. from the sintering temperature. In another arrangement, the heat treatment step may also comprise reheating the article after it has been cooled from the sintering temperature.

If reheated after being cooled from the sintering temperature, the article may be reheated to a temperature of 500°C for a period of 1 hour.

The aging time may be 6 - 24 hours.
The aging temperature may be 140 - 200°C.

The method may also comprise the step of sizing the article. The step of sizing generally comprises pressing the article in a closed die to further improve the dimensional accuracy of the article. If the article
is heat treated, the step of sizing preferably occurs after quenching and preferably before ageing.

The step of providing the powder mixture may comprise mixing elemental and/or master alloy powders to form the powder mixture.

The master alloy powders may be for example Al-Mg, Mg-Si and Al-Si powders.

The step of providing the powder mixture may comprise blending the powder mixture. Blending improves the homogeneity of the powder mixture thus reducing the likelihood of there being any variations in composition between small samples of the powder mixture.

The method may also comprise the step of mixing a lubricant wax with the powder mixture. In another arrangement, the powder mixture provided may already have had a lubricant wax added thereto.

The method may also comprise the step of dewaxing the article.

The step of dewaxing may comprise heating the green article to a dewaxing temperature, which is preferably 250°C to 450°C, more preferably approximately 350°C.

The step of sintering may occur in the presence of a sintering gas. Preferably, the sintering gas is nitrogen gas which has a dew point of ≤-60°C.

The step of sintering may comprise heating the article to a temperature of 560°C to 610°C, more preferably approximately 590°C.

The step of sintering preferably occurs for 10 to 30 minutes, more preferably 18 to 22 minutes.

The rate of dimensional change of the alloy during sintering is preferably 0.02 to 0.05 % per minute, more preferably 0.03 to 0.04 % per minute.

The powder mixture is pressed in the die at a compaction pressure of preferably 200 to 400 MPa, more preferably 285 to 315 MPa and more preferably approximately 300 MPa.
Examples

Sintered alloys were prepared firstly by mixing elemental powders according to specified compositions with one weight percent Aerowax C. Each powder mixture was agitated for four minutes with a sieving machine to break up clusters and then mixed for 25 min in a tubular powder mixer. The compaction of the specimens was performed using a hand operated Carver hydraulic press and a floating rectangular die. Specimens were 55.3 x 10.5 x 5.0 mm³ in size. Subsequent sintering and dewaxing of the compacted specimens occurred in a horizontal tube furnace in a high purity nitrogen atmosphere having a dew point < -65°C and a gas flow rate of 9.5 l/min. An electrical resistance heated tube furnace was used having a diameter of 162 mm. Eight (8) rectangular bars were sintered each time. The heating rate from dewaxing to sintering was ~ 20°C/min and the dewaxing was performed at approximately 350°C for 30 min. After sintering, the specimens were removed from the heated zone of the furnace and cooled (quenched) to room temperature in ambient air. The quench delay was <1 min.

Example 1

Alloys according to the compositions set out in Table 1 were prepared according to the above mentioned method.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Copper</th>
<th>Magnesium</th>
<th>Silicon</th>
<th>Aluminium</th>
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<tr>
<td>A</td>
<td>2.6</td>
<td>1.0</td>
<td>1.1</td>
<td>Balance</td>
</tr>
<tr>
<td>B</td>
<td>2.8</td>
<td>1.0</td>
<td>1.1</td>
<td>Balance</td>
</tr>
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Alloys A and B were prepared at compaction pressures of 250, 285, 300 and 315 MPa and at sintering times varying between 15 and 25 minutes. Figures 1 (a) and (b) are graphs showing the effects of changing the compaction pressure, sintering time and copper content of
the alloy on the percentage dimensional change of the alloys formed. Figure 1 (a) shows that the reduction in the compaction pressure from 300 MPa to 285 MPa increased the dimensional change of the alloy specimens by 0.1%. Reducing the compaction pressure to 250 MPa had an even greater effect on the dimensional change. Figure 1 (b) however, shows that an increase in the compaction pressure from 300 MPa to 315 MPa had much less of an effect on the dimensional change of the alloy. Notably, Figure 2 also shows that alloys with dimensional change approaching zero may be formed with a sintering time of approximately 20 minutes. This is a significant reduction over conventional sintering times for sintered aluminium alloys, which typically require sintering for approximately 30 minutes. Reducing the sintering time increases the potential throughput of sintered alloy articles hence increasing the cost effectiveness of producing articles from such alloys.

Tensile bars for mechanical property testing were machined from samples of Alloy A. These bars had a gauge length of 10 mm and a rectangular cross-section of approximately 3 mm x 40 mm. From the subsequent mechanical testing, Alloy A was found to have an ultimate tensile strength of 194 ± 4 MPa and a ductility of 2.2 % ± 0.2 %.

Samples of Alloys A and B were also produced at varying sintering temperatures from 586°C to 594°C. The compaction pressure of the green samples was 300 MPa. Figure 2 shows the effects of changing the sintering temperature on the dimensional change of Alloys A and B. As shown, for sintering temperatures between 588°C and 592°C, the dimensional change in the alloys was approximately constant.

Example 2

Sintered alloys having compositions according to Table 2 were prepared in accordance with the aforementioned method.
Alloys C to F were prepared at compaction pressures of 250 MPa and 300 MPa and were sintered for between 15 and 25 minutes. Figures 3 (a) and (b) are graphs showing the effect on the percentage dimensional change of Alloys C to F by varying the compaction pressure, the sintering time and the silicon content. The alloys showed a relatively low tolerance to changes in the silicon content. A 0.1 weight % reduction of silicon for example caused a reduction of 0.2 % in the dimensional change during sintering.

Example 3

Sintered alloys having compositions according to Table 3 were prepared in accordance with the aforementioned method.

Table 3

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Copper</th>
<th>Magnesium</th>
<th>Silicon</th>
<th>Aluminium</th>
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</thead>
<tbody>
<tr>
<td>G</td>
<td>2.6</td>
<td>0.6</td>
<td>0.7</td>
<td>Balance</td>
</tr>
<tr>
<td>H</td>
<td>2.6</td>
<td>0.6</td>
<td>0.8</td>
<td>Balance</td>
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<tr>
<td>I</td>
<td>2.6</td>
<td>0.6</td>
<td>0.9</td>
<td>Balance</td>
</tr>
<tr>
<td>J</td>
<td>2.6</td>
<td>0.4</td>
<td>0.8</td>
<td>Balance</td>
</tr>
<tr>
<td>K</td>
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<td>0.8</td>
<td>0.8</td>
<td>Balance</td>
</tr>
<tr>
<td>L</td>
<td>2.2</td>
<td>0.6</td>
<td>0.8</td>
<td>Balance</td>
</tr>
<tr>
<td>M</td>
<td>2.8</td>
<td>0.6</td>
<td>0.8</td>
<td>Balance</td>
</tr>
</tbody>
</table>
Mechanical properties (Ultimate Tensile Strength, 0.2% Proof Strength and Ductility) were measured for a number of samples of each of alloys G-M. The results of this mechanical testing are set out in Figures 4 (a) – (c).

Example 4

Sintered alloy H was prepared generally in accordance with the aforementioned method but for varying compaction pressures ranging from 250-315 MPa and for varying sintering temperatures ranging from 586-594°C and for varying sintering times ranging from 15-25mins. The purpose of this testing was to study the tolerance of the sintering process variable.

Figure 5 is a graph showing the variation in dimensional change of alloy H for the different compaction pressures over the range of sintering times. It can be seen in Figure 5 that decreasing the compaction pressure from 300 MPa to 285 MPa had a greater effect than increasing the compaction pressure to 315 MPa.

Figure 6 is a graph showing the variation in dimensional change of alloy H for the different sintering temperatures for a sintering time of 20 minutes. The dimensional change was nearly constant across sintering temperatures of 590±4°C at the higher compaction pressures. However, for the lower compaction pressures, there was less tolerance for an increase in the sintering temperature which caused a greater shift in the dimensional change.

Example 5

Sintered alloys having the following compositions were prepared generally in accordance with the aforementioned method but for varying sintering times:

1. Al-xCu-0.6Mg-0.8Si, where x = 1.8, 2.2, 2.6, 3.0, 3.4, 4.2
2. Al-2.6Cu-yMg-0.8Si, where y = 0.2, 0.4, 0.6, 0.8, 1.0

3. Al-2.6Cu-0.6Mg-zSi, where z = 0.5, 0.7, 0.8, 0.9, 1.1

Figures 7 (a) - (c) show the variation in the dimensional change due to variations in the alloy composition. Notably the alloy has a relatively high tolerance to changes in magnesium and copper content but a low tolerance to variations in the silicon content. For the given sintering and compaction conditions, alloys having a copper content of 2.2 - 3.0%, a magnesium content of 0.2 - 1.0% and a silicon content of 0.7-0.9% had a dimensional change of 0.00 - 0.1%. Minor variations in the dimensional change due to changes in the copper content can be corrected by changing the compaction pressure and/or sintering time.

In the claims which follow and in the preceding description of the invention, except where the context requires otherwise due to express language or necessary implication, the word "comprise" or variations such as "comprises" or "comprising" is used in an inclusive sense, i.e. to specify the presence of the stated features but not to preclude the presence or addition of further features in various embodiments of the invention.
CLAIMS

1. A powder mixture for use in forming a sintered aluminium alloy, the powder mixture having a metallic content comprising, by weight:
   
   1.5 - less than 3.8% copper
   0.3 - 1.5% silicon
   0.1 - 1.5% magnesium

   and optionally one or more from the group consisting of

   0.01 - 0.2% tin
   0.1 - 0.5% silver
   0.01 - 0.5% zirconium
   0.01 - 0.5% vanadium
   0.01 - 1.0% titanium
   0.01 - 1.0% chromium

   with the balance being aluminium except for incidental impurities.

2. A powder mixture as claimed in claim 1, wherein the copper content is 2.2 to 3.0%.

3. A powder mixture as claimed in claim 1, wherein the copper content is approximately 2.6%.

4. A powder mixture as claimed in any one of the preceding claims, wherein the silicon content is 0.7-0.9%.

5. A powder mixture as claimed in any one of claims 1-3, wherein the silicon content is approximately 0.8%.

6. A powder mixture as claimed in any one of the preceding claims, wherein the magnesium content is 0.2-1.0%.

7. A powder mixture as claimed in any one of claims 1-5, wherein the magnesium content is approximately 0.6%.
8. A powder mixture as claimed in any one of the preceding claims, wherein the mixture also comprises a lubricant wax, in an amount of 1.0-1.5% by weight relative to the total metallic content.

10. A sintered aluminium alloy article formed from a powder mixture, the alloy comprising, by weight:
   1.5 - less than 3.8% copper
   0.3 - 1.5% silicon
   0.1 - 1.5% magnesium

10 and optionally one or more from the group consisting of
   0.01 - 0.2% tin
   0.1 - 0.5% silver
   0.01 - 0.5% zirconium
   0.01 - 0.5% vanadium
   0.01 - 1.0% titanium
   0.01 - 1.0% chromium

with the balance being aluminium except for incidental impurities.

11. A sintered aluminium alloy article formed from a powder mixture as claimed in any one of claims 1 - 9.

12. A sintered aluminium alloy article as claimed in claim 10 or 11, wherein the alloy has a tensile strength of at least 170 MPa.

13. A sintered aluminium alloy article as claimed in any one of claims 10-12, wherein the alloy has a ductility of at least 1.5%.

14. A sintered aluminium as claimed in any one of claims 10-13, wherein the alloy has a dimensional change from die size after sintering of -1.5% to +1.5%.

15. A method for producing an article from a sintered aluminium alloy, the method comprising the steps of:
providing a powder mixture as claimed in any one of claims 1-9;
pressing the powder mixture in a die to form a green article;
ejecting the green article from the die; and
sintering the green article to produce the sintered aluminium alloy article.

16. A method as claimed in claim 15, wherein the method also comprises the step of heat treating the article after it has been sintered.

17. A method as claimed in claim 16, wherein the step of heat treating comprises solution treating, quenching and aging the article.

18. A method as claimed in any one of claims 15-17, wherein the method also comprises the step of sizing the article by pressing the article in a closed die.

19. A method as claimed in any one of claims 15-18, wherein the step of providing the powder mixture may comprise mixing elemental and/or master alloy powders to form the powder mixture.

20. A method as claimed in any one of claims 15-19, wherein the method also comprises the step of mixing lubricant wax with the powder mixture.

21. A method as claimed in claim 20, wherein the method also comprises the step of dewaxing the article by heating the green article to a dewaxing temperature.

22. A method as claimed in claim 21, wherein the dewaxing temperature is 250-450°C.

23. A method as claimed in any one of claims 15-22,
wherein the step of sintering occurs in the presence of a sintering gas.

24. A method as claimed in any one of claims 15-23, wherein the step of sintering comprises heating the article to 560-610°C.

25. A method as claimed in any one of claims 15-24, wherein the step of sintering occurs for 10-30 minutes.

26. A method as claimed in any one of claims 15-25, wherein the rate of dimensional change of the alloy during sintering is 0.02-0.05 % per minute.

27. A method as claimed in any one of claims 15-26, wherein the powder mixture is pressed in the die at a compaction pressure of 285-315 MPa.
Figure 1(a)

Figure 1(b)
Figure 2
Figure 3(a)
Figure 4(a)

Figure 4(b)
Figure 4(c)
Figure 5

Figure 6
Figure 7(a)

Figure 7(b)
Figure 7(c)
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl.

B22F 1/00 (2006.01) C22C 21/02 (2006.01) C22C 27/76 (2006.01)
C22C 1/04 (2006.01) C22C 21/08 (2006.01)

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)

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 practicable, search terms used)

EPDOCC and WPI (ALLOYS database): CUL>= 1.5 AND CUH<= 3.8, SIL<= 0.3 AND SIH<= 1.5, MGL>= 0.1 AND MGH<= 1.5
STN database: AL and (3.8<= cu>= 1.5 and 1.5<= si>= 0.3 and 1.5<= mg>= 0.1) and Google patents search with keywords such as sinter, aluminium, alloy, powder. Science direct search with keywords sintered aluminium alloy

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
<thead>
<tr>
<th>Category*</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
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<tbody>
<tr>
<td>X</td>
<td>US 4177069 A (KOBAYASHI et al.) 4 December 1979 see abstract, column 2 lines 52-column 3 lines 8, Table 2, claims 1-3, examples 2, 3, 8 and 9</td>
<td>1-7, 10-20, 23-27</td>
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<tr>
<td>Y</td>
<td>Age Hardening of Al-Cu-Mg-Si-(Sn) alloy (KENT et al.) Materials Science and Engineering A, 405, Pages 65-73, 19 May 2005</td>
<td>8, 21-22</td>
</tr>
<tr>
<td>Y</td>
<td>The effect of tin and nitrogen on liquid phase sintering of Al-Cu-Mg-Si alloys (SCHAFFER et al.) Acta Materialia, 56, Pages 2615-2624, 10 March 2008</td>
<td>8, 21-22</td>
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</table>

X Further documents are listed in the continuation of Box C. X See patent family annex

* Special categories of cited documents:
  "A" - document defining the general state of the art which is not considered to be of particular relevance
  "E" - earlier application or patent but published on or after the international filing date
  "L" - document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
  "O" - document referring to an oral disclosure, use, exhibition or other means
  "P" - document published prior to the international filing date but later than the priority date claimed

T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

X document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

Y document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

& document member of the same patent family

Date of the actual completion of the international search 30 June 2009
Date of mailing of the international search report 18 JUL 2009

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Form PCT/ISA/210 (second sheet) (July 2008)
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<td>On enhancing the mechanical properties of aluminium P/M alloys (BISHOP D.P. et al.) Materials Science and Engineering A290, Pages 16-24, 13 April 2000</td>
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<td>A</td>
<td>Precipitation processes in an Al-2.5Cu-1.5 Mg (wt %) alloy microalloyed with Ag and Si (RAVIPRASAD et al.) Acta Materialia, 51, pages 5037-5050, 19 June 2003</td>
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This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

<table>
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<tr>
<td>US 4177069</td>
<td>DE 2815159 GB 1600439 JP 53125205</td>
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<td>JP 53125912</td>
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Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.

END OF ANNEX