(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 0 818 553 B1

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention of the grant of the patent:
28.02.2001 Bulletin 2001/09

(51) Int Cl.[7]: **C22F 1/047**, C22C 21/06

(21) Application number: **97201948.3**

(22) Date of filing: **26.06.1997**

(54) **Method of manufacture of aluminium sheet of the AA5000 type**

Verfahren zur Herstellung von Blech aus Aluminium von Typ AA5000

Procédé de fabrication de feuille d'aluminium du type AA5000

(84) Designated Contracting States:
BE CH DE ES FR GB LI NL

(30) Priority: **28.06.1996 NL 1003453**

(43) Date of publication of application:
14.01.1998 Bulletin 1998/03

(73) Proprietor: **Corus Aluminium N.V.**
2570 Duffel (BE)

(72) Inventors:
- **Bottema, Jan**
  2082 BN Santpoort-Zuid (NL)
- **Kaasenbrood, Paul Hendrikus Theodorus**
  1973 VG IJmuiden (NL)
- **De Smet, Peter**
  9830 Sint-Martens-Latem (BE)

(74) Representative:
Hansen, Willem Joseph Maria et al
Corus Technology BV
Corus Intellectual Property Department
PO Box 10000
1970 CA IJmuiden (NL)

(56) References cited:
EP-A- 0 593 034
EP-A- 0 594 509
EP-A- 0 646 655
EP-A- 0 681 034
EP-A- 0 773 303
US-A- 4 540 449
US-A- 5 244 516

- **PATENT ABSTRACTS OF JAPAN vol. 018, no. 191 (C-1186), 4 April 1994 & JP 05 345963 A (FURUKAWA ALUM CO LTD;OTHERS: 02), 27 December 1993,**
- **W.HUFNAGEL: "ALUMINIUM-SCHLUESSEL" 1983 , ALUMINUM VERLAG , DUESSELDORF, DE XP002025184 * page 75 - page 81 ***

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

EP 0 818 553 B1

Description

[0001] This invention relates to a method for the manufacture of aluminium alloy sheet having a content of Mg of 1.5 to 8 wt%, particularly having a composition belonging to the AA5000 series. The invention also relates to the sheet produced by the method, which is particularly suitable for forming applications for example in the manufacture of body parts by pressing in the automotive industry.

[0002] In this specification sheet is not only taken to mean sheets obtained by cutting, but also strip-shaped sheet e.g. in coil form, which has not yet been cut.

[0003] In aluminium alloys containing Mg as a main alloying element it is known to include at least one of Mn, Cr or Zr approximately 0.10-0.5 wt% (percentage by weight; herein all percentages are by weight unless otherwise specified). Mn, Cr and Zr are so-called dispersoid-forming alloying elements and their addition to the aluminium alloy obtains an effective grain refinement during annealing following cold-rolling. Other alloying elements may also be added for this purpose such as Co, Ti, Ni or V, but in practice Mn, Cr and Zr are the most used. A disadvantage is that these alloying elements have low solubility in aluminium. A further disadvantage is that these alloying elements are relatively expensive, while the value and the applicability of scrap material containing in particular Zr and/or Cr is low.

[0004] Aluminium alloys containing Mg have also been disclosed in which the amounts of elements such as Mn, Cr and Zr are deliberately kept low. JP-A-5-345693 proposes an alloy having 6-10% Mg and a total of 0.01-0.2% of (Mn + Cr + Zr + V), which after rolling to final thickness is heat treated in a continuous annealing furnace at 450-550°C for less than 120s and then immediately cooled at 800°C/min or more to obtain a crystal grain size of 20-120μm. This heat treatment is as short as even 3s.

[0005] EP-A-681034 also describes an alloy having low content of (Fe + Mn + Cr + Ti + Zr) which is subjected at final thickness to heat treatment by heating at a rate of at least 100°C/min to 450-550°C and maintaining the maximum temperature for not more than 300s. The cooling rate is 100°C/min or more. The temperature range is selected to achieve a desired recrystallization. The fastest heating rate mentioned is 540°C/min, but no particular reason is given for the selection of heating rate.

[0006] EP-A-646655 also describes alloy sheets having low (Cr + Mn + Zr + V) which at final thickness are subjected to the heat treatment of heating at at least 3°C/s to 500-580°C, maintaining the maximum temperature for 0-60s and cooling at at least 2°C/s. It is said that if the temperature exceeds 580°C, the heating rate is less than 3°C/s or the temperature is maintained for more than 60s, abnormal grain growth occurs.

[0007] The object of the present invention is to provide a method of manufacture of an aluminium alloy sheet having good formability in which the contents of Mn, Cr and Zr in particular are low.

[0008] According to the invention there is provided a method for the manufacture of aluminium alloy sheet having a content of Mg of 1.5 to 8.0 wt% and a total content of (Mn + Cr + Zr) of not more than 0.1 wt%, comprising a solution heat treatment step at final thickness of the sheet in a continuous annealing furnace, characterised in that in said solution heat treatment the heating rate of the sheet in the heating region of said continuous annealing furnace is at least 50°C/s. It has unexpectedly been found that the use of a very high heating speed in the continuous annealing furnace, which can be achieved for example by homogenous heating by means of inductive heating, gives improved mechanical properties to the sheet.

[0009] First the composition of the alloy of the sheet produced by the invention will be discussed.

[0010] In the aluminium sheet the sum of the (Mn + Cr + Zr)-content is less than or equal to 0.1%. Typically therefore, deliberately no Mn, Cr or Zr is added to the alloy, other than what is by chance or inevitably present in the recycling scrap used. This achieves the effect that adding these relatively expensive alloying elements may be omitted, while at the same time the value and the applicability of the recycling scrap of the aluminium sheet produced in accordance with the invention increase.

[0011] Preferably the average grain size after the solution heat treatment is in the range 20-80μm. In this context grain size is taken to mean the average grain diameter according to ASTM-E-112. This achieves the effect that the formability of the aluminium sheet is optimal. With a grain size smaller than approximately 20 μm, the formability of the aluminium sheet may sharply decrease, while it may also not be possible to suppress the formation of so-called Lüders lines. If the grain size in the aluminium sheet is larger than approximately 80μm, then a so-called orange peel can form during pressing. This is an undesired effect.

[0012] The Fe-content is preferably less than 0.5% and more preferably less than approximately 0.25%. With an Fe-content larger than approximately 0.5%, relatively large intermetallic compounds may form which can greatly reduce the formability of the aluminium sheet. An Fe-content less than 0.25% achieves the effect that the aluminium sheet combines a good formability with an average grain size in the desired range.

[0013] The Si-content is preferably less than approximately 0.4% more and preferably less than 0.2%. This achieves the effect that the aluminium sheet combines a good formability with an average grain size in the desired range.

[0014] The Cu-content may be less than 1.0% and preferably in the range 0.3-0.7%. This achieves the effect that the aluminium sheet is thermally hardenable.

[0015] Alternatively, Cu is in a range up to a maximum 0.20%. In this case the element Cu is to be regarded as an impurity and is not deliberately added to the alloy, other than what is present by chance or inevitably in the recycling scrap used.

[0016] Ti may be present as grain refining element for the cast micro-structure. The Ti-content is preferably less than 0.2% and more preferably less than 0.1% in order to prevent the formation of coarse $TiAl_3$ particles during solidification. The Ti may be added in combination with the elements B or C and preferably the B content is in a range less than 0.01% to prevent the presence of TiB_2 particles in the structure.

[0017] Zn may be present in a range up to a maximum 0.25%. Generally Zn is to be regarded as an impurity and is not deliberately added to the alloy other than what is present by chance or inevitably in the recycling scrap used. Zn can be tolerated in a greater range than other impurities but the Zn content is desirably limited to the stated range.

[0018] Other dispersoid-forming elements, such as Co, Ni and V are also preferably only at impurity level, if present.

[0019] In the method for the manufacture of the aluminium sheet in accordance with the invention the sheet may be brought to its final thickness (finished thickness at end of the rolling processes) by conventional rolling techniques. The aluminium sheet is then solution heat-treated so that recrystallization takes place, in a continuous annealing furnace. The heating rate of the aluminium sheet in the heating section of the continuous annealing furnace is at least 50°C/s, and is preferably over 80°C/s. The maximum temperature is preferably within the temperature range 450-570°C and the time at this temperature is preferably 5-300s, and more preferably 5-30s. Immediately following solution heat-treatment the aluminium sheet is preferably cooled to below 150°C at a cooling rate of at least 100°C/min.

[0020] The method in accordance with the invention has many advantages. Firstly the mechanical properties of the aluminium sheet produced in accordance with the invention can be at least comparable to those of aluminium sheet of the AA5000 type alloy in the stated range of the Mg-content in which at least one of the alloying elements Zr, Cr or Mn is also present in a range greater than 0.1%. Secondly the formability of the aluminium sheet in accordance with the invention can be greater than an aluminium sheet of a similar type of alloy with about the same Mg-content, but with the presence of such alloying elements as Zr, Cr or Mn. This is achieved in part because the grain size is in the stated range attained with the heat-treatment in accordance with the invention. Furthermore, because of the very short annealing time virtually no Mg-oxide scale forms on the surface of the aluminium sheet, so that no discolouration and less problems with pressing occur. Also the rolling oil present following cold-rolling gives far less problems in continuous annealing because no staining occurs as a result of partial burning. Moreover the throughput time of the product is very short.

[0021] Figs. 1 and 2 show graphically the test results of the following Examples.

Example 1

[0022] Two types of aluminium sheet of different alloy compositions A and B belonging to the AA5000-series were cold-rolled to a finished thickness of 1.06mm and then solution heat-treated in two different processes I and II. Table 1 lists the chemical compositions A and B of the aluminium sheets, in which A is a comparative composition and B is an alloy composition in accordance with the invention.

Table 1

The alloy composition of the tested aluminium sheets (wt%)							
Sheet	Mg	Mn	Cu	Fe	Si	Zr	Cr
A	4.05	0.37	0.01	0.28	0.10	0.01	0.01
B	4.80	0.05	0.01	0.18	0.11	0.01	0.01

The two different processes of solution heat treatment are:

I. heating the aluminium sheet at a heating rate of approximately 25°C/hour to a temperature of 360°C, then holding for 1 hour at 360°C, and then allowing to cool in air to below 150°C at a cooling rate of approximately 10°C/hour. This is a known method and is often designated by the term 'batch-anneal'.

II. annealing the aluminum sheet in a continuous annealing furnace in which the aluminium sheet is heated by inductive heating at about 100°C/s, the aluminium sheet is held for 10 seconds at a temperature of 530°C, and then quenched to below a temperature of 150°C.

[0023] Following these solution heat-treatments the uniform elongation Ag, the total elongation A80, and the work hardening exponent "n" of the four different aluminium sheet products were determined together with the grain size. The results are given in Fig. 1 and Table 2. In Fig. 1, A-I gives the sheet of composition A treated by treatment I, etc.

[0024] It can be seen from these results that with the same alloy types the grain size of statically annealed material (treatment I) is smaller than of continuously annealed material (treatment II). At the same time it can be seen that with the same alloy types continuously annealed material has a higher Ag, A80 and "n" exponent than batch annealed material.

[0025] When comparing Alloy A-I with Alloy B-I, and Alloy A-II with Alloy B-II it can be seen that best results of elongation and "n" exponent are obtained with the aluminium sheet of composition and the solution heat treatment in accordance with the invention, while the average grain size is in the desired range.

Table 2

The grain size of the different aluminium sheets following solution heat treatment		
Aluminium sheet	ASTM grain size number	average grain size (μm)
Alloy A-I	10.3	10
Alloy A-II	9.2	15
Alloy B-I	8.0	25
Alloy B-II	7.2	30

Example 2

[0026] Three aluminium sheets with a chemical composition in accordance with plate B of Table 1, were cold rolled to a finished sheet thickness of 1.06mm and then solution heat treated in three different processes a, b and c. The three different processes were:

a. heating the aluminium sheet at a heating rate of approximately 25°C/hour to a temperature of 360°C, then holding for 1 hour at 360°C, and then allowing to cool in air to below 150°C at a cooling rate of approximately 10°C/hour (batch annealing).
b. annealing the aluminium sheet in a continuous annealing furnace in which the aluminium sheet is heated by inductive heating at a heating rate of approximately 5°C/sec, the aluminium sheet is held for 10 sec. at a temperature of 530°C, and then quenched to below at a temperature of 150°C at a cooling rate of approximately 150°C/sec.
c. annealing the aluminium sheet in a continuous annealing furnace in which the aluminium sheet is heated by inductive heating at a heating rate of approximately 100°C/sec, the aluminium sheet is held for 10 sec. at a temperature of 530°C, and then quenched to below at a temperature of 150°C at a cooling rate of approximately 150°C/sec.

[0027] Following solution heat treatment the Ag, A80 and the work hardening exponent "n" were determined as in Example 1. The results are shown in Fig. 2, in which B-a gives the results for sheet of composition B treated by process a, etc.

[0028] From these results it can be seen that continuously annealed sheets (treatment b and c) have a higher Ag, A80 and "n"-exponent than batch annealed sheet (treatment a).

[0029] It can also be seen that continuous annealed sheet with a heating rate of approximately 100°C/sec has a higher Ag, A80 and "n"-exponent than sheet with a heating rate of approximately 5°C/sec.

Claims

1. A method for the manufacture of aluminium alloy sheet having a content of Mg of 1.5 to 8.0 wt% and a total content of (Mn + Cr + Zr) of not more than 0.1 wt%, comprising a solution heat treatment step at final thickness of the sheet in a continuous annealing furnace, characterised in that in said solution heat treatment the heating rate of the sheet in the heating region of said continuous annealing furnace is at least 50°C/s.

2. A method according to claim 1, wherein said heating rate is at least 80°C/s.

3. A method according to claim 1 or 2, wherein in said solution heat treatment, the sheet is held at a maximum temperature in the range 450 to 570°C for a time period in the range 5 to 300s.

4. A method according to claim 3 wherein said time period is in the range 5 to 30s.

5. A method according to any one of the preceding claims, wherein in said solution heat treatment the cooling rate of said sheet is at least 100°C/min to below 150°C.

6. A method according to any one of the preceding claims, wherein in said heating region of said continuous annealing furnace, the sheet is heated by induction heating.

7. A method according to any one of the preceding claims, wherein said aluminium alloy belongs to the AA5000 series.

8. A method according to any one of the preceding claims, wherein the average grain size of the aluminium alloy after the solution heat treatment is in the range 20 to 80μm.

9. A method according to any one of the preceding claims, wherein said aluminium alloy has a content of Fe of not more than 0.5 wt%.

10. A method according to any one of the preceding claims, wherein said aluminium alloy has a content of Si of not more than 0.4 wt%.

11. A method according to any one of the preceding claims, wherein said aluminium alloy has a content of Cu or not more than 1.0 wt%.

12. A method according to claim 11, wherein said aluminium alloy has a Cu content of not more than 0.2 wt%.

Patentansprüche

1. Ein Verfahren zur Herstellung von Aluminiumlegierungsblech mit einem Mg-Gehalt von 1,5 bis 8,0 Gew.-% und einem Gesamtgehalt von (Mn + Cr + Zr) von nicht mehr als 0,1% Gew.-%, das einen Lösungswärmebehandlungsschritt bei der Enddicke des Bleches in einem kontinuierlichen Vergütungsofen umfaßt, dadurch gekennzeichnet, daß die Erwärmungsrate des Bleches in dem Erwärmungsbereich des kontinuierlichen Vergütungsofens bei der Lösungswärmebehandlung wenigstens 50°C/s beträgt.

2. Ein Verfahren nach Anspruch 1, worin die Erwärmungsrate wenigstens 80°C/s beträgt.

3. Ein Verfahren nach Anspruch 1 oder 2, worin das Blech bei der Lösungswärmebehandlung bei einer Maximaltemperatur im Bereich von 450 bis 570°C für einen Zeitraum im Bereich von 5 bis 300s gehalten wird.

4. Ein Verfahren nach Anspruch 3, worin die Zeitdauer im Bereich von 5 bis 30s liegt.

5. Ein Verfahren nach einem der vorhergehenden Ansprüche, worin die Abkühlrate des Bleches bei der Lösungswärmebehandlung wenigstens 100°C/min bis unterhalb von 150°C beträgt.

6. Ein Verfahren nach einem der vorhergehenden Ansprüche, worin das Blech in dem Erwärmungsbereich des kontinuierlichen Vergütungsofens durch Induktionserwärmung erwärmt wird.

7. Ein Verfahren nach einem der vorhergehenden Ansprüche, worin die Aluminiumlegierung zu der AA 5000 Klasse gehört.

8. Ein Verfahren nach einem der vorhergehenden Ansprüche, worin die durchschnittliche Korngröße der Aluminiumlegierung nach der Lösungswärmebehandlung im Bereich von 20 bis 80 μm liegt.

9. Ein Verfahren nach einem der vorhergehenden Ansprüche, worin die Aluminiumlegierung einen Fe-Gehalt von nicht mehr als 0,5 Gew.-% aufweist.

10. Ein Verfahren nach einem der vorhergehenden Ansprüche, worin die Aluminiumlegierung einen Si-Gehalt von nicht mehr als 0,4 Gew.-% aufweist.

11. Ein Verfahren nach einem der vorhergehenden Ansprüche, worin die Aluminiumlegierung einen Cu-Gehalt von nicht mehr als 1,0 Gew.-% aufweist.

12. Ein Verfahren nach Anspruch 11, worin die Aluminiumlegierung einen Cu-Gehalt von nicht mehr als 0,2 Gew.-% aufweist.

Revendications

1. Procédé de fabrication d'une feuille en alliage d'aluminium ayant une teneur en Mg de 1,5 à 8,0 % en poids et une teneur totale en (Mn + Cr + Zr) non supérieure à 0,1 % en poids, qui comprend une étape de traitement thermique de solution, à l'épaisseur finale de la feuille, dans un four de recuit continu, procédé caractérisé en ce que, dans ledit traitement thermique de solution, la vitesse de chauffage de la feuille dans la zone chauffante dudit four de recuit continu est d'au moins 50 °C par seconde.

2. Procédé selon la revendication 1, dans lequel ladite vitesse de chauffage est d'au moins 80 °C par seconde.

3. Procédé selon la revendication 1 ou 2, dans lequel, lors dudit traitement thermique de solution, la feuille est maintenue à une température maximale comprise dans l'intervalle allant de 450 à 570 °C pendant une durée de 5 à 300 secondes.

4. Procédé selon la revendication 3, dans lequel ladite durée est de 5 à 30 secondes.

5. Procédé selon l'une quelconque des revendications précédentes, dans lequel, lors dudit traitement thermique de solution, la vitesse de refroidissement de ladite feuille est d'au moins 100 °C par minute, jusqu'à une température inférieure à 150 °C.

6. Procédé selon l'une quelconque des revendications précédentes, dans lequel la feuille est chauffée par chauffage par induction, dans ladite zone chauffante dudit four de recuit continu.

7. Procédé selon l'une quelconque des revendications précédentes, dans lequel ledit alliage d'aluminium appartient à la catégorie AA5000.

8. Procédé selon l'une quelconque des revendications précédentes, dans lequel, après le traitement thermique de solution, la taille de grain moyenne de l'alliage d'aluminium est comprise dans l'intervalle allant de 20 à 80 µm.

9. Procédé selon l'une quelconque des revendications précédentes, dans lequel ledit alliage d'aluminium a une teneur en Fe non supérieure à 0,5 % en poids.

10. Procédé selon l'une quelconque des revendications précédentes, dans lequel ledit alliage d'aluminium a une teneur en Si non supérieure à 0,4 % en poids.

11. Procédé selon l'une quelconque des revendications précédentes, dans lequel ledit alliage d'aluminium a une teneur en Cu non supérieure à 1,0 % en poids.

12. Procédé selon la revendication 11, dans lequel ledit alliage d'aluminium a une teneur en Cu non supérieure à 0,2 % en poids.

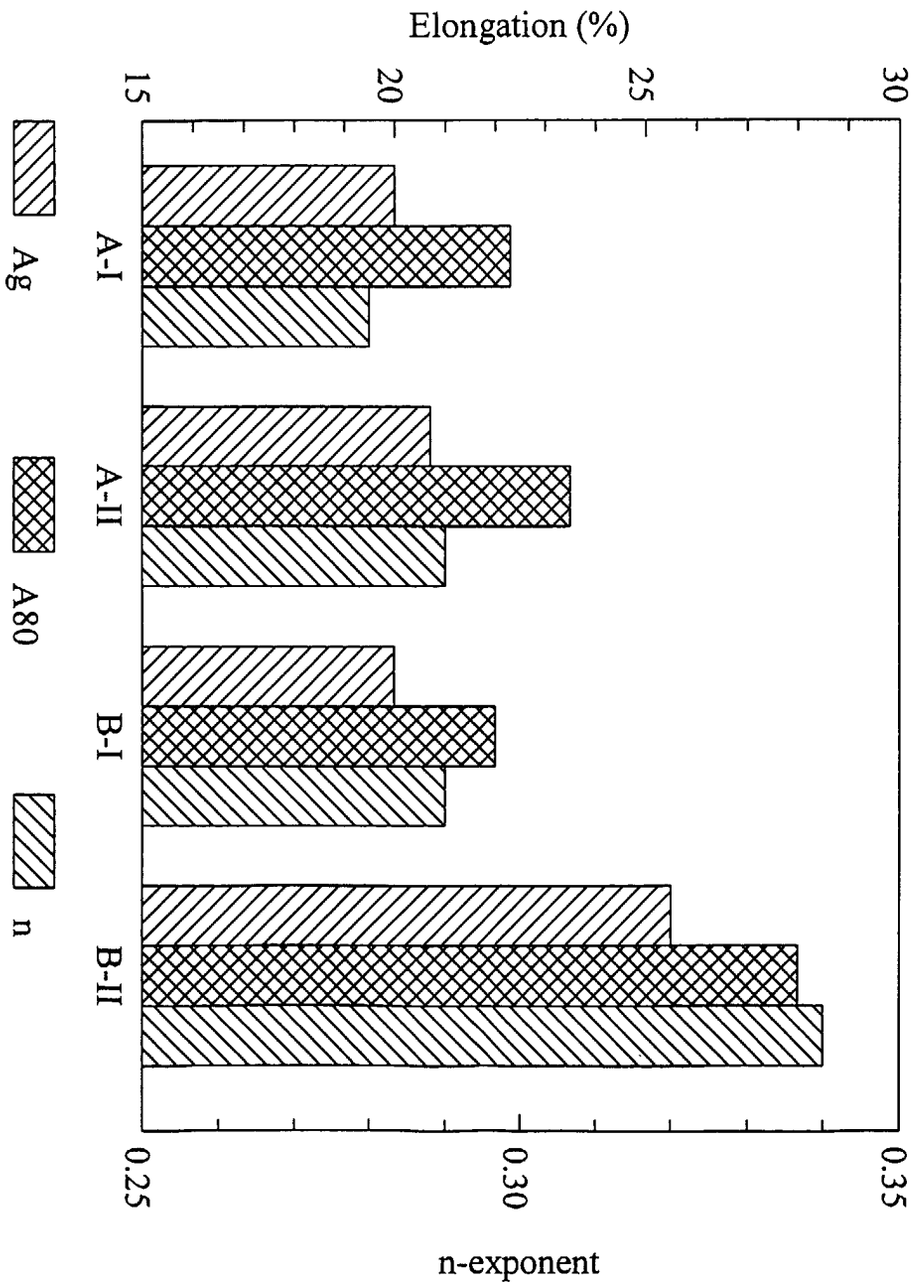
Elongation (%)
15
20
25
30
Ag
A80
n
A-I
A-II
B-I
B-II
0.25
0.30
0.35
n-exponent

Fig. 1

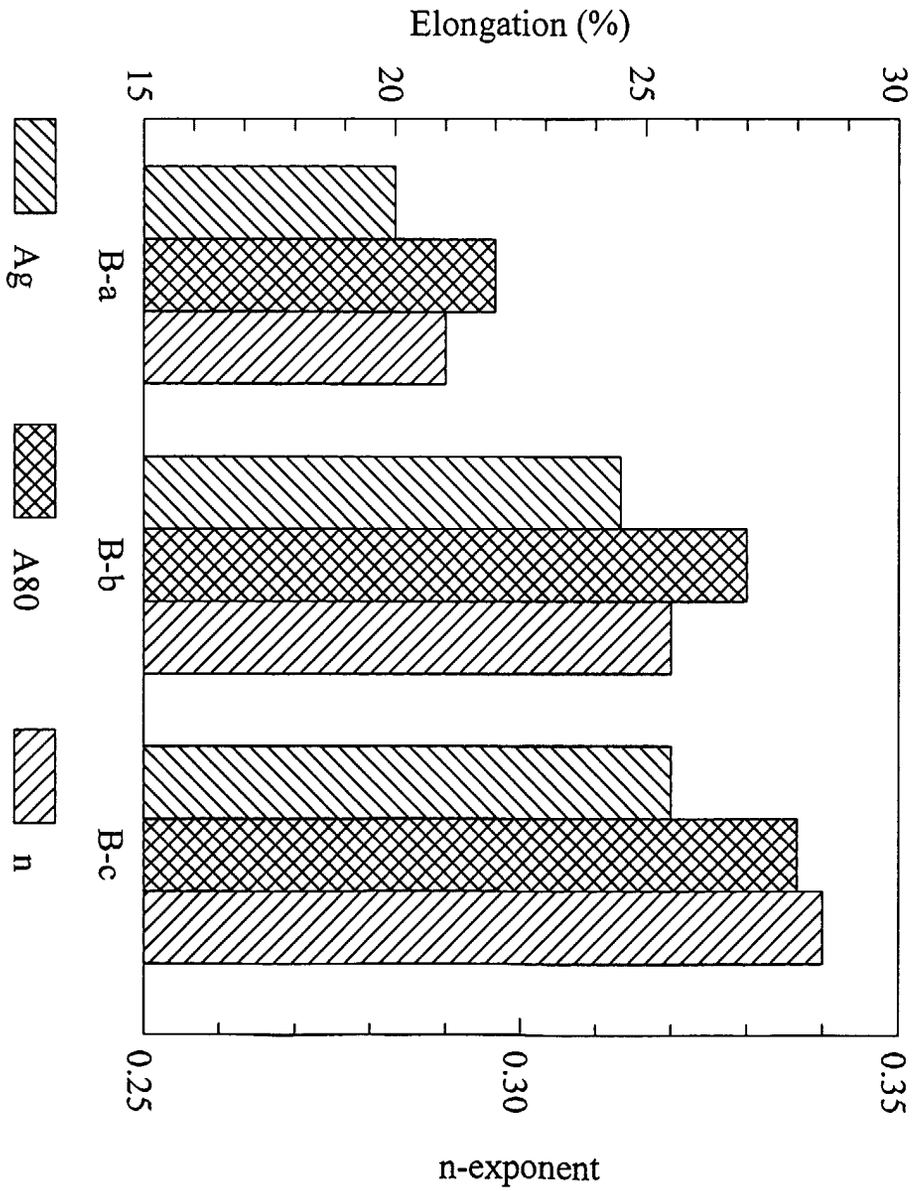

Fig. 2