



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(54) Title: HIGH STRENGTH, DUCTILE, LOW DENSITY ALUMINUM ALLOYS AND PROCESS FOR MAKING SAME			
(57) Abstract			
<p>A process for making high strength, high ductility, low density aluminum-base alloys, consisting essentially of 2,5 to 5 wt % Li, 0,15 to 2 wt % Zr, 0 to 5 wt % of at least one element selected from the group consisting of Cu, Mg, Si, Se, Ti, U, Hf, Be, Cr, V, Mn, Fe, Co, Ni, balance Al. The alloy is given multiple aging treatments after being solutionized. The microstructure of the alloy is characterized by the precipitation of a composite phase in the aluminum matrix thereof.</p>			

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DESCRIPTIONHIGH STRENGTH, DUCTILE, LOW DENSITY ALUMINUM
ALLOYS AND PROCESS FOR MAKING SAME5 1. Field of the Invention

The invention relates to a process for making high strength, high ductility, low density aluminum-based alloys, and, in particular, to the alloys that are characterized by a homogeneous distribution of composite
10 precipitates in the aluminum matrix thereof. The microstructure is developed by heat treatment method consisting of initial solutionizing treatment followed by multiple aging treatments.

2. Background of the Invention

15 There is a growing need for structural alloys with improved specific strength to achieve substantial weight savings in aerospace applications. Aluminum-lithium alloys offer the potential of meeting the weight savings due to the pronounced effects of lithium on the mechanical and physical properties of aluminum alloys.
20 The addition of one weight percent lithium (~3.5 atom percent) decreases the density by ~3% and increases the elastic modulus by ~6% , hence giving a substantial increase in the specific modulus (E/ρ). Moreover, heat treatment of alloys results in the precipitation of
25 a coherent, metastable phase, δ' (Al_3Li) which offers considerable strengthening. Nevertheless, development and widespread application of the Al-Li alloy system have been impeded mainly due to its inherent
30 brittleness.

It has been shown that the poor toughness of alloys in the Al-Li system is due to brittle fracture along the grain or subgrain boundaries. The two dominant micro-
structural features responsible for their brittleness
35 appear to be the precipitation of intermetallic phases along the grain and/or subgrain boundaries and the marked planar slip in the alloys, which create stress concentrations at the grain boundaries. The inter-

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granular precipitates tend to embrittle the boundary, and simultaneously extract Li from the boundary region to form precipitate free zones which act as sites of strain localization. The planar slip is largely due to the shearable nature of δ' precipitates which result in decreased resistance to dislocation slip on planes containing the sheared δ' precipitates.

Several metallurgical approaches have been undertaken to circumvent these problems. It has been found that the PFZ (precipitate free zone) and precipitate-induced intergranular fracture can be reduced by controlling processing to avoid the intergranular precipitation of stable Al-Li, Al-Cu-Li, Al-Mg-Li phases. The problem of planar slip can be partly alleviated by promoting slip dispersion through the addition of dispersoid forming elements and the controlled co-precipitation of Al-Cu-Li, Al-Cu-Mg and/or Al-Li-Mg intermetallics. The dispersoid forming elements include Mn, Fe, Co, etc. The co-precipitation of Cu and/or Mg containing intermetallics appears to be relatively effective in dispersing the dislocation movement. However, the sluggish formation of these intermetallics requires the thermomechanical treatments involving stretching operations and multiple aging treatments (P.J. Gregson and M.M. Flower, *Acta Metallurgica*, vol. 33, pp. 527-537, 1985), or a high Cu content which adversely affects the density of alloys (B van der Brandt, P.J. von den Brink, H.F. de Jong, L. Katgerman, and H. Kleinjan, in "Aluminum-Lithium Alloy II", Metallurgical Society of AIME, pp. 433-446, 1984). Moreover, the properties of alloys thus processed were less than satisfactory.

Recently, a new approach has been suggested to modify the deformation behavior of Al-Li alloy system through the development of Zr modified δ' precipitate. This approach is based on the observation that the metastable Al_3Zr phase in the Al-Zr alloy system is highly resistant to dislocation shear and is of the same

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crystal structure ($L1_2$) as δ' . In this regard, attempts have been made to produce a ternary ordered composite $Al_3(Li, Zr)$ phase in the aluminum matrix with an alloy of Al-2.34 Li-1.07Zr (F.W. Gayle and J.B. Vander Sande, Scripta Metallurgica, Vol. 18, pp. 473-478, 1984).
5 However, the process for developing a homogeneous distribution of such phase has required the strict control of processing parameters during the thermomechanical processing, as well as prolonged solutionizing and/or aging treatments. From the practical point of view,
10 this process is quite undesirable and may also result in undesirable microstructural features such as recrystallization and wide precipitate free zones. Moreover, the process cannot be effectively applied to low Zr (e.g.,
15 0.2 wt% Zr) containing alloys which produce a small volume fraction of heterogeneously distributed coarse composite precipitates (P.L. Makin and B. Ralph, Journal of Materials Science, vol. 19, pp. 3835-3843, 1984; P.J. Gregson and H.M. Flower, Journal of Materials Science Letters, vol. 3, pp. 829-834, 1984; P.L. Makin, D.J. Lloyd, and W.M. Stobbs, Philosophical Magazine A, vol. 51, pp. L41-L47, 1985).

Despite considerable efforts to develop low density aluminum alloys, conventional techniques, such as those
25 discussed above, have been unable to provide low density aluminium alloys having the sought for combination of high strength, high ductility and low density. As a result, conventional aluminum-lithium alloy systems have not been entirely satisfactory for applications such as
30 aircraft structural components, wherein high strength, high ductility and low density are required.

SUMMARY OF THE INVENTION

The present invention provides a process for making aluminium-lithium alloys containing a high density of
35 substantially uniformly distributed shear resistant dispersoids which markedly improve the strength and ductility thereof. The low density aluminum-base alloys, of the invention consist essentially of the

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formula $Al_{bal}Zr_aLi_bX_c$, wherein X is at least one element selected from the group consisting of Cu, Mg, Si, Sc, Ti, U, Hf, Cr, V, Mn, Fe, Co and Ni, "a" ranges from about 0.15-2 wt%, "b" ranges from about 2.5-5 wt%, "c" ranges from about 0-5 wt% and the balance is aluminum. The microstructure of these alloys is characterized by the precipitation of composite $Al_3(Li, Zr)$ phase in the aluminum matrix thereof. This microstructure is developed in accordance with the process of the present invention by subjecting an alloy having the formula delineated above to solutionizing treatment followed by multiple aging treatments. An improved process for making high strength, high ductility, low density aluminum-based alloy is thereby provided wherein the aluminum-based alloy produced has an improved combination of strength and ductility (at the same density).

The high strength, high ductility, low density aluminum-based alloy produced in accordance with the present invention has a controlled composite $Al_3(Li, Zr)$ precipitate which, advantageously, offers a wide range of strength and ductility combinations.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention will be more fully understood and further advantages will become apparent when reference is made to the following detailed description and the accompanying drawings, in which:

Fig. 1 is a dark field transmission electron micrograph of an alloy having the composition Al-3.1Li-2Cu-1Mg-0.5Zr, the alloy having been subjected to double aging treatments (170°C for 4 hrs. followed by 190°C for 16 hrs.) to develop a composite precipitate in the aluminum matrix thereof;

Fig. 2 is a weak beam dark field micrograph of an alloy having the composition Al-3.7Li-0.5Zr, illustrating the resistance of the composite precipitate to dislocation shear during deformation;

Fig. 3(a) shows the planar slip observed in an

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alloy having the composition Al-3.7Li-0.5Zr, the alloy having been subjected to a conventional aging treatment (180°C for 16 hours);

Fig. 3(b) shows the beneficial effect of subjecting the alloy of Fig.3(a) to treatment in accordance with the claimed process (160°C for 4 hrs. followed by 180°C for 16 hrs.), thereby promoting the homogeneous deformation thereof;

Fig. 4 shows the sheared δ' precipitates observed in an alloy having the composition Al-3.1Li-2Cu-1Mg-0.5Zr, the alloy having been subjected to a conventional aging treatment (190°C for 16 hours); and

Fig. 5 shows the development of composite precipitates in an alloy having the composition Al-3.2Li-3Cu-1.5Mg-0.2Zr, the alloy having been subjected to treatment in accordance with the claimed process (170°C for 4 hrs. followed by 190°C for 16 hrs).

DESCRIPTION OF THE PREFERRED EMBODIMENTS

In general, the present invention relates to the process of making high strength, high ductility, and low density Al-Li-Zr-X alloys. The process involves the use of multiple aging steps during heat treatment of the alloy. The alloy is characterized by a unique microstructure consisting essentially of "composite" $Al_3(Li,Zr)$ precipitate in an aluminum matrix (Fig. 1) due to the heat treatment as hereinafter described. The alloy may also contain other Li, Cu and/or Mg containing precipitates provided such precipitates do not significantly deteriorate the mechanical and physical properties of the alloy.

The factors governing the properties of the Al-Li-Zr-X alloys are primarily its Li content and microstructure and secondarily the residual alloying elements. The microstructure is determined largely by the composition and the final thermomechanical treatments such as extrusion, forging and/or heat treatment parameters. Normally, an alloy in the as-processed condition (cast, extruded or forged) has large

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intermetallic particles. Further processing is required to develop certain microstructural features for certain characteristic properties.

The alloy is given an initial solutionizing treatment, that is, heating at a temperature (T_1) for a period of time sufficient to substantially dissolve most of the intermetallic particles present during the forging or extrusion process, followed by cooling to ambient temperature at a sufficiently high rate to retain alloying elements in said solution. Generally, the time at temperature T_1 , will be dependent on the composition of the alloy and the method of fabrication (e.g., ingot cast, powder metallurgy processed) and will typically range from about 0.1 to 10 hours. The alloy is then reheated to an aging temperature, T_2 , for a period of time sufficient to activate the nucleation of composite $Al_3(Li, Zr)$ precipitates, and cooled to ambient temperature, followed by a second aging treatment at temperature, T_3 , for a period of time sufficient for the growth of the composite $Al_3(Li, Zr)$ precipitate and a dissolution of δ' precipitate whose nucleation is not aided by Zr. The alloy at this point is characterized by a unique microstructure which consists essentially of composite $Al_3(Li, Zr)$ precipitate. This composite $Al_3(Li, Zr)$ precipitate is resistant to dislocation shear and quite effective in dispersing dislocation motion (Fig. 2). The result is that the alloy containing an optimum amount of composite $Al_3(Li, Zr)$ precipitate deforms by a homogeneous mode of deformation resulting in improved mechanical properties. Fig. 3(b) clearly shows the homogeneous mode of deformation in an alloy subjected to the process claimed in this invention, while Fig. 3(a) shows the severe planar slip observed in a conventionally processed alloy due to the shearing of δ' precipitates by dislocations (see Fig. 4). The combination of ductility with high strength is best achieved in accordance with the invention when the density of the

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shear resistant dispersoids ranges from about 10 to 60 percent by volume, and preferably from about 20-40 percent by volume.

The exact temperature, T_1 , to which the alloy is heated in the solutionizing step is not critical as long as there is a dissolution of intermetallic particles at this temperature. The exact temperature, T_2 , in the first aging step where the nucleation of composite Al_3 (Li, Zr) precipitate is promoted, depends upon the alloying elements present and upon the final aging step. The optimum temperature range for T_2 , is from about 100°C to 180°C. The exact temperature, T_3 , whose range is from 120°C to 200°C, depends on the alloying elements present and mechanical properties desired. Generally, the times at temperatures T_2 and T_3 are different depending upon the composition of the alloy and the thermomechanical processing history, and will typically range from about 0.1 to 100 hours.

EXAMPLE 1

The ability of composite Al_3 (Li, Zr) precipitates to modify the deformation behavior of Al-Li-Zr alloys is illustrated as follows:

Fig. 2 is a weak beam dark field transmission electron micrograph showing microstructure of a deformed alloy (Al-3.7Li-0.5Zr) which has been solutionized at 540°C for 4 hrs. and subsequently aged at 160°C for 4 hrs. followed by final aging at 180°C for 16 hrs. Such heat treatment promotes the precipitation of composite Al_3 (Li, Zr) which is highly resistant to dislocation shear and is quite effective in dispersing the dislocation movement.

Fig. 3(a) shows a bright field electron micrograph showing microstructure of a deformed alloy (Al-3.7Li-0.5Zr) which has not been given the claimed process. The alloy had been aged, for 16 hrs. at 180°C after solutionizing at 540° for 4 hrs. This alloy showed the pronounced planar slip which is the common deformation characteristic of brittle alloy.

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In contrast, Fig. 3(b) illustrates the beneficial effect of the claimed process on the deformation behavior of an alloy having the composition Al-3.7Li-0.5Zr. After solutionizing at 540°C for 4 hrs., the alloy had been subjected to the double aging treatment of 160°C for 4 hrs. and 180°C for 16 hrs. The deformation mode of this alloy is quite homogeneous indicating high ductility.

Example 2

An alloy having a composition of Al-3.1Li-2Cu-1Mg-0.5Zr was developed for medium strength applications as shown in Table I. The alloy was solutionized at 540°C for 2.5 hrs., quenched into water at about 20°C and given conventional single aging and the claimed double aging treatments.

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TABLE I

	0.2% Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation to Failure (%)
5 Aged at 190°C for 16 hrs.	524	592	3.6
Aged at 170°C for 4 hrs. and 190°C for 16 hrs.	530	606	6.1
10 Conventional aging treatment (190°C for 16 hrs.) showed poor ductility (3.6%) due to the shearing of δ' precipitate (Fig. 4), while composite precipitate developed by double aging (Fig. 1) improve both strength and ductility (6.1% elongation).			

Example 3

A high strength Al-Li alloy was made to satisfy the requirements for high strength applications for aerospace structure. An alloy having a composition of Al-3.2Li-2Cu-2Mg-0.5Zr was solutionized at 542°C for 4 hrs. As shown in Table II, conventional aging treatment (190°C for 16 hrs.) showed lower strength (yield strength of 521 MPa) and ductility (3.6%). However, double aging of the alloy (160°C for 4 hrs. followed by 180°C for 16 hrs.) gave significantly higher strength (yield strength of 554 MPa) and ductility (5.5%), which meets property requirements for high strength alloys needed for aerospace structural applications.

TABLE II

	0.2% Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation to Failure (%)
30 Aged at 190°C for 16 hrs.	521	595	3.6
35 Aged at 160°C for 4 hrs. and 180°C for 16 hrs.	554	631	5.5

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Example 4

This example illustrates the beneficial effect of the claimed process on the mechanical properties of a simple ternary alloy Al-3.7Li-0.5Zr. The alloy was solutionized at 540°C for 4 hrs., and subsequently aged as shown in Table III. The resulting tensile properties show that the claimed process results in improved strength and ductility compared to the conventional process.

TABLE III

Aging Treatment	0.2% Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation to Fracture (%)
140°C, 16 hr.	424	442	4.2
120°C, 4 hr. + 140°C, 16 hr.	434	460	6.0
160°C, 16 hr.	419	431	3.2
140°C, 4 hr. + 160°C, 16 hr.	425	448	4.8
140°C, 16 hr. + 160°C, 16 hr.	426	451	4.6

Example 5

A wide range of mechanical properties can be achieved by using multiple aging conditions. For example, a triple aging treatment (120°C, 4 hrs. + 140°C, 16 hrs. + 160°C, 4 hrs.) produced yield strength of 446 MPa and ultimate tensile strength of 464 MPa with 4.6% elongation. As a result, a variety of heat treatments of the alloys according to the claims can be employed to produce alloys having a variety of mechanical properties.

Example 6

This example illustrates the potential of the claimed process for the development of composite precipitate in low Zr containing Al-Li alloys. Fig. 5 shows the dark field electron micrograph of a typical

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alloy Al-3.2Li-3Cu-1.5Mg-0.2Zr which had been
solutionized at 540°C for 4 hrs., reheated to 170°C for
4 hrs. followed by final aging at 190°C for 16 hrs. The
large volume fraction of composite Al₃ (Li, Zr) precipi-
5 tate observed in such an alloy indicates that the
claimed process is also quite effective in Al-Li alloys
having low Zr content of 0.2%. Having thus described
the invention in rather full detail, it will be
understood that such detail need not be strictly adhered
10 to but that further changes and modifications may
suggest themselves to one skilled in the art, all
falling within the scope of the present invention as
defined by the subjoined claims.

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What is claimed is:

1. A process for increasing the strength and ductility of low density aluminum-base alloys comprising the steps of subjecting an Al-Li alloy, to multiple aging treatments to form therein a microstructure wherein a high density of shear resistant dispersoids are substantially uniformly distributed, said alloy consisting essentially of the formula $Al_{ba}Li_bX_c$, wherein X is at least one element selected from the group consisting of Cu, Mg, Si, Sc, Ti, U, Hf, Be, Cr, V, Mn, Fe, Co and Ni, "a" ranges from about 0.15-2 wt%, "b" ranges from about 2.5-5 wt%, "c" ranges from 0 to about 5 wt% and the balance is aluminum.

2. A process according to claim 1 wherein said alloy is characterized by the precipitation of composite Al_3 (Li, Zr) phase in an aluminum matrix.

3. A process as recited by claim 1, wherein the number of aging treatments ranges from 2 to 10.

4. A process as recited by claim 1, wherein the number of aging treatments ranges from 2 to 5.

5. A process for making high strength, high ductility, low density aluminum-lithium alloy, comprising the steps of:

heating an aluminum alloy, consisting essentially of the formula $Al_{ba}Li_bX_c$, wherein X is at least one element selected from the group consisting of Cu, Mg, V, Si, Sc, Ti, U, Hf, Be, Cr, Mn, Fe, Co and Ni, "a" ranges from about 0.15-2 wt%, "b" ranges from about 2.5-5 wt%, "c" ranges from 0 to about 5 wt% and balance of aluminum, to a temperature, T_1 , for a period of time sufficient to substantially dissolve most of the intermetallic particles therein;

cooling said alloy to ambient temperature at rates sufficient to retain its elements in supersaturated solid solution;

heating said alloy to a temperature, T_2 , for a period of time sufficient to activate nucleation of composite Al_3 (Li, Zr) precipitates;

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cooling said alloy to ambient temperature;
heating said alloy to a temperature, T_3 , for a
period of time sufficient to effect additional growth of
composite Al_3 (Li, Zr) precipitates, and dissolution
5 of δ' precipitates whose nucleation is not aided by Zr;
and

cooling said alloy to ambient temperature to
produce therein a controlled precipitation of composite
 Al_3 (Li, Zr) phase in said aluminum matrix.

10 6. A process according to claim 1, further
comprising the step of stretching said solutionized
alloy.

7. A process according to claim 5, further
comprising the step of stretching said alloy.

15 8. A process according to claim 5 wherein T_1
ranges from about 500°C to 555°C, T_2 ranges from about
100°C to 180°C and T_3 ranges from about 120°C to 200°C.

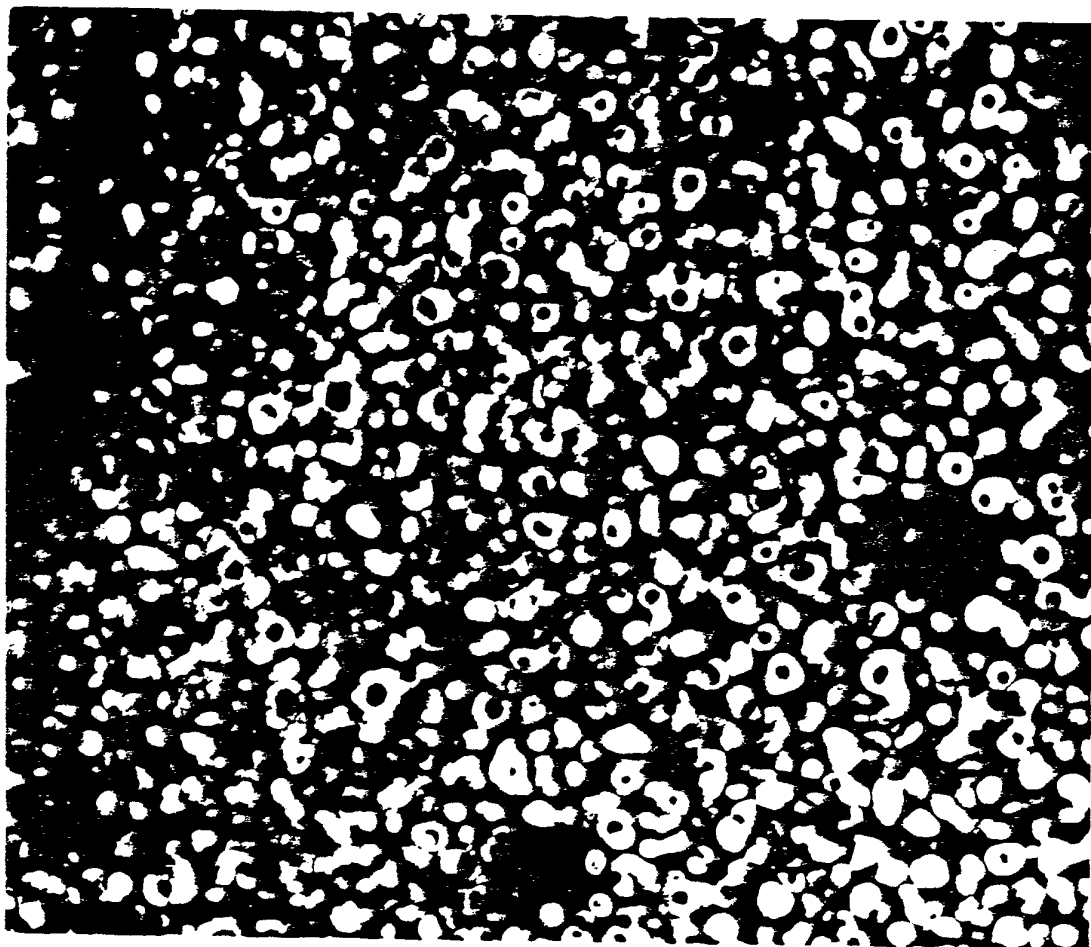
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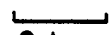
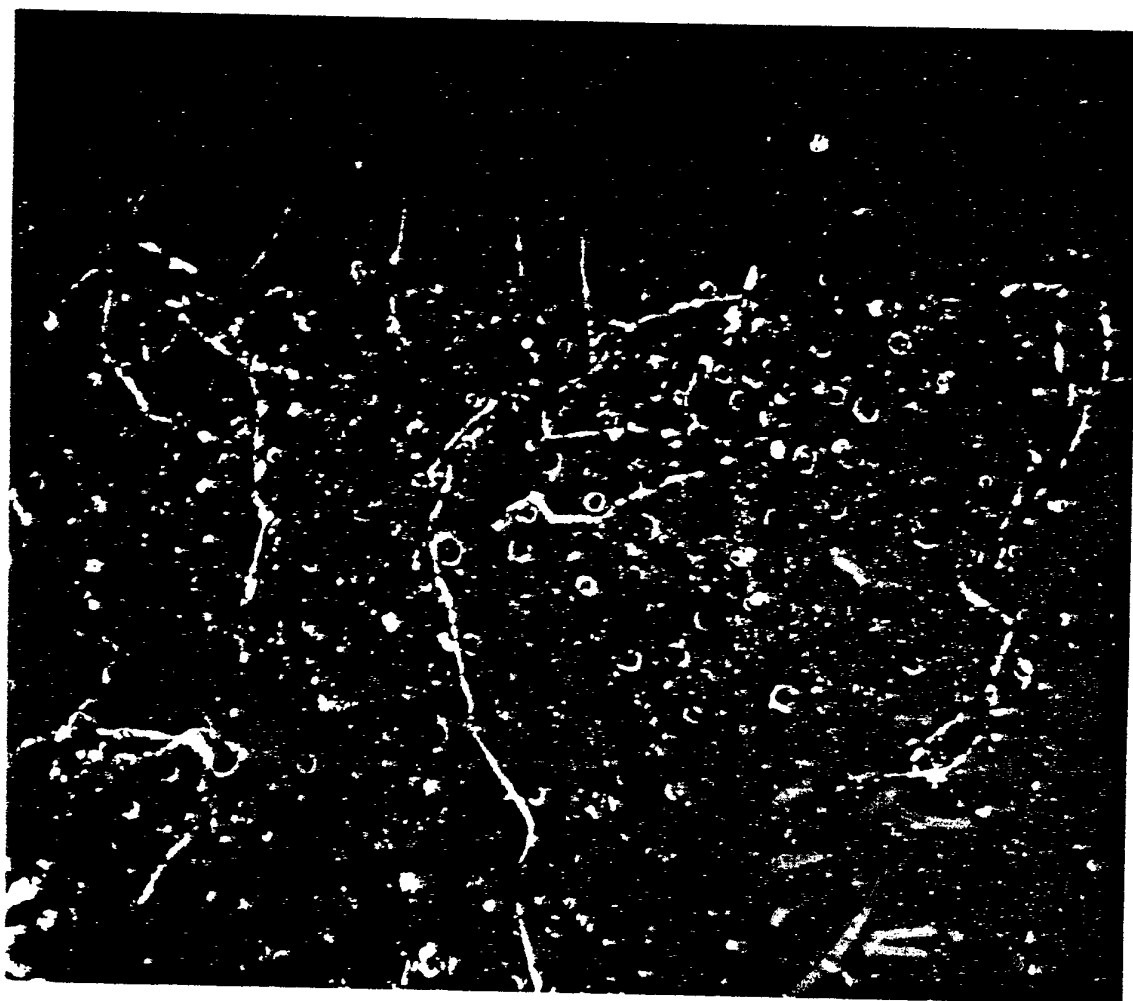

0.1 μm

Fig. 1

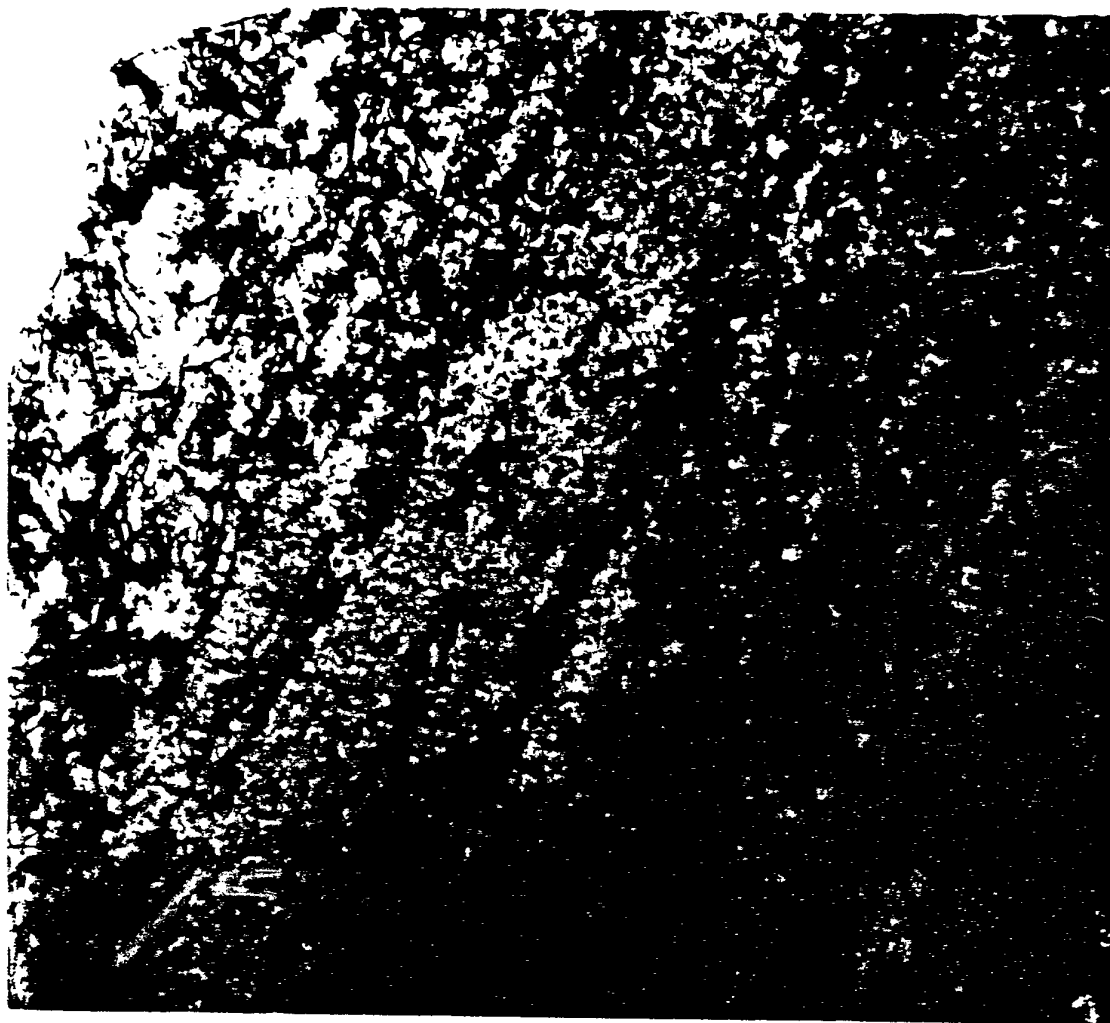
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0.1 μ m

Fig.2

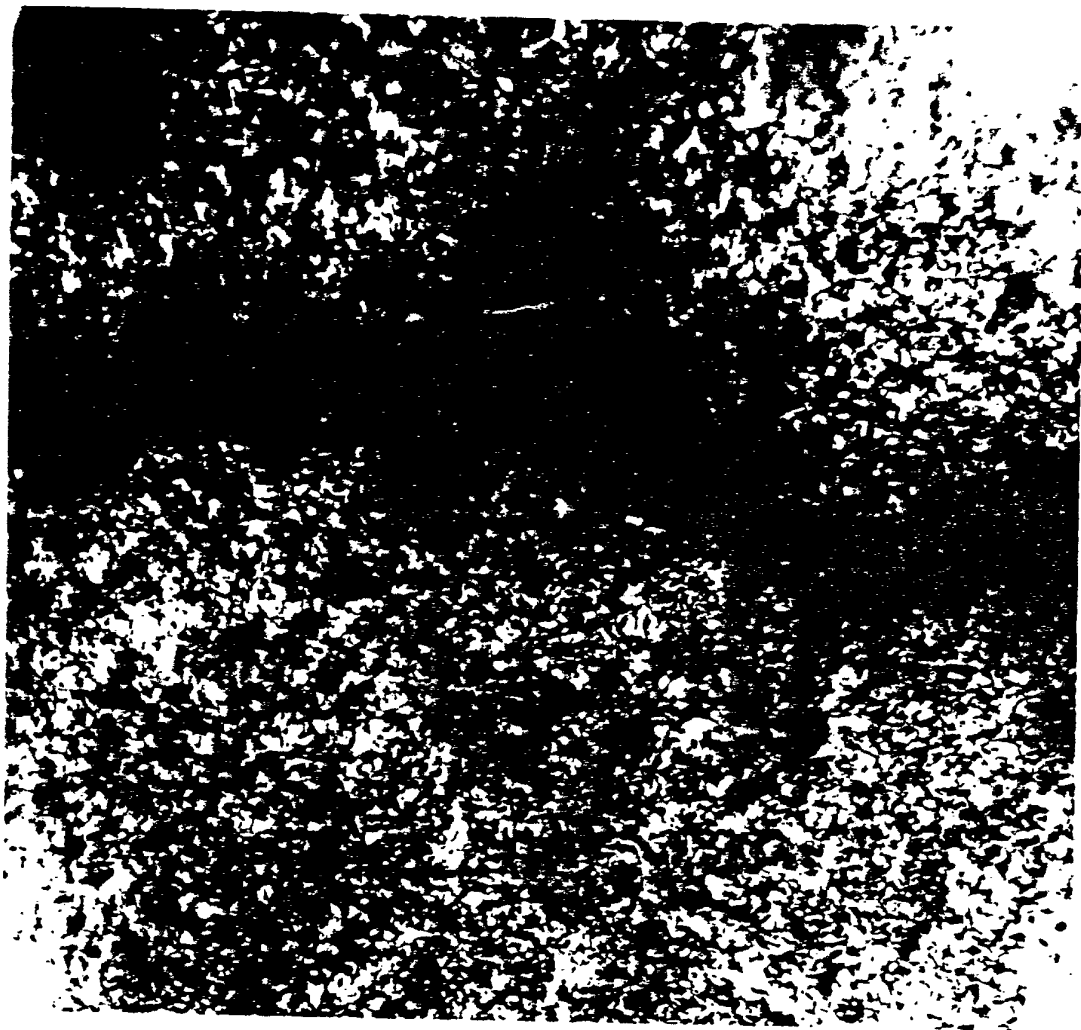
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0.2 μ m

Fig.3(a)

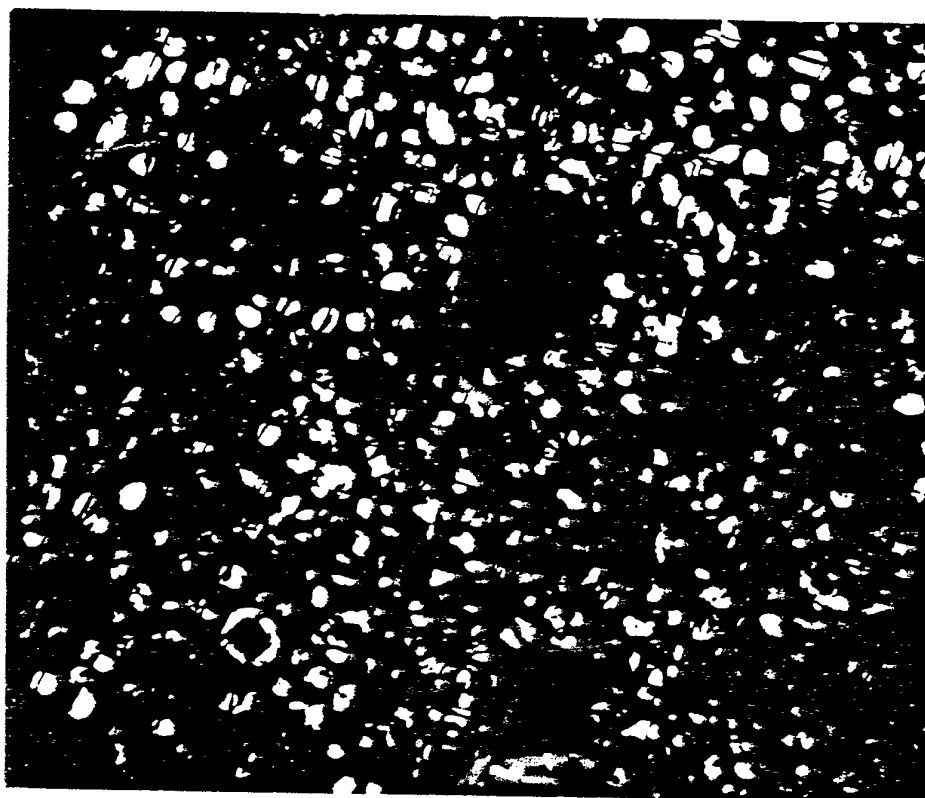
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0.2 μ m

Fig.3(b)

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0.2 μm

Fig. 4

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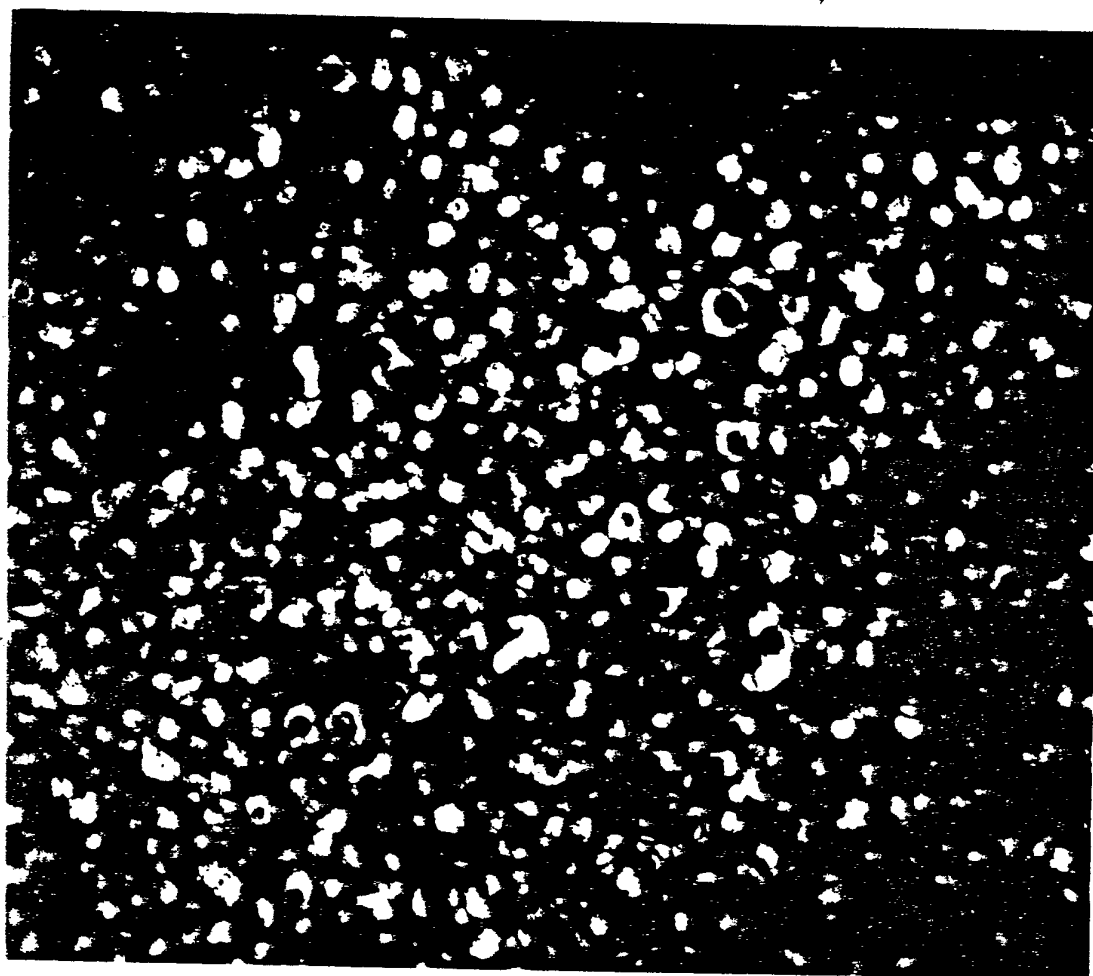
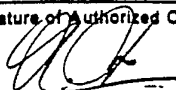


Fig.5

0.1 μ m

INTERNATIONAL SEARCH REPORT

International Application No PCT/US 86/00757

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC		
IPC ⁴ : C 22 C 21/00; C 22 F 1/04		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
IPC ⁴	C 22 C 21/00 C 22 F 1/04	
Documentation Searched other than Minimum Documentation to the extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ⁹		
Category ⁹	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
X, P	EP, A, 0158769 (ALLIED CORPORATION) 23 October 1985, see claims 1,7,8 --	1
A	FR, A, 2538412 (SUMITOMO LIGHT METAL INDUSTRIES) 29 June 1984, see claims 1,2,5; examples; page 11, samples 12,15; page 14, samples 12,15,20 --	1
A	Metallurgical Transactions A, volume 13A, March 1982 Lin et al.: "Microstructure-property relationships of two Al-3Li-2Lu-0,2Zr-X Cd Alloys", pages 401-410, see page 402, : results and Discussion --	1
A	Chemical Abstracts, volume 101, no. 4, 23 July 1984, Columbus, Ohio, (US) Gayle et al.: "Composite" precipitates in an aluminum-lithium-zirconium alloy", see page 214, abstract no. 27114f & Scr. Metall. 1984, 18(5), 473-8 -----	1
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>* Special categories of cited documents: ¹⁰</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"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)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"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</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"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.</p> <p>"&" document member of the same patent family</p> </div> </div>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
24th July 1986	09 SEP 1986	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	 L. ROSSI	

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON

INTERNATIONAL APPLICATION NO.

PCT/US 86/00757 (SA 12980)

This Annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 19/08/86

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A- 0158769	23/10/85	JP-A- 60208445	21/10/85
FR-A- 2538412	29/06/84	JP-A- 59118848	09/07/84
		DE-A- 3346882	28/06/84
		GB-A, B 2134925	22/08/84

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