Title: A METHOD FOR FORMING HIGH STRENGTH ALUMINUM ALLOYS CONTAINING L1_2 INTERMETALLIC DISPERSOIDS

Abstract: A method and apparatus produces high strength aluminum alloys from a powder containing L1_2 intermetallic dispersoids. The powder is degassed, sealed under vacuum in a container, heated, consolidated by vacuum hot pressing, and extruded.
A METHOD FOR FORMING HIGH STRENGTH ALUMINUM ALLOYS
CONTAINING L12 INTERMETALLIC DISPERSOIDS

BACKGROUND

The present invention relates generally to aluminum alloys and more specifically to a method for forming high strength aluminum alloy powder having L12 dispersoids therein.

The combination of high strength, ductility, and fracture toughness, as well as low density, make aluminum alloys natural candidates for aerospace and space applications. However, their use is typically limited to temperatures below about 300°F (149°C) since most aluminum alloys start to lose strength in that temperature range as a result of coarsening of strengthening precipitates.

The development of aluminum alloys with improved elevated temperature mechanical properties is a continuing process. Some attempts have included aluminum-iron and aluminum-chromium based alloys such as Al-Fe-Ce, Al-Fe-V-Si, Al-Fe-Ce-W, and Al-Cr-Zr-Mn that contain incoherent dispersoids. These alloys, however, also lose strength at elevated temperatures due to particle coarsening. In addition, these alloys exhibit ductility and fracture toughness values lower than other commercially available aluminum alloys.

Other attempts have included the development of mechanically alloyed Al-Mg and Al-Ti alloys containing ceramic dispersoids. These alloys exhibit improved high temperature strength due to the particle dispersion, but the ductility and fracture toughness are not improved.

U.S. Patent No. 6,248,453 owned by the assignee of the present invention discloses aluminum alloys strengthened by dispersed Al3X L12 intermetallic phases where X is selected from the group consisting of Sc, Er, Lu, Yb, Tm, and Lu. The Al3X particles are coherent with the aluminum alloy matrix and are resistant to coarsening at elevated temperatures. The improved mechanical properties of the disclosed dispersion strengthened L12 aluminum alloys are stable up to 572°F (300°C). U.S. Patent Application Publication No. 2006/0269437 Al also commonly owned discloses a high strength aluminum alloy that contains scandium and other elements that is strengthened by L12 dispersoids.

L12 strengthened aluminum alloys have high strength and improved fatigue properties compared to commercially available aluminum alloys. Fine grain size results
in improved mechanical properties of materials. Hall-Petch strengthening has been known for decades where strength increases as grain size decreases. An optimum grain size for optimum strength is in the nanometer range of about 30 to 100 nm. These alloys also have lower ductility.

SUMMARY

The present invention is a method for consolidating aluminum alloy powders into useful components with high temperature strength and fracture toughness. In embodiments, powders include an aluminum alloy having coherent Al$_2$X dispersoids where X is at least one first element selected from scandium, erbium, thulium, ytterbium, and lutetium, and at least one second element selected from gadolinium, yttrium, zirconium, titanium, hafnium, and niobium. The balance is substantially aluminum containing at least one alloying element selected from silicon, magnesium, lithium, copper, zinc, and nickel.

The powders are classified by sieving and blended to improve homogeneity. The powders are then vacuum degassed in a container that is then sealed. The sealed container (i.e. can) is vacuum hot pressed to densify the powder charge and then compacted further by blind die compaction or other suitable method. The can is removed and the billet is extruded, forged and/or rolled into useful shapes with high temperature strength and fracture toughness.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is an aluminum scandium phase diagram.
FIG. 2 is an aluminum erbium phase diagram.
FIG. 3 is an aluminum thulium phase diagram.
FIG. 4 is an aluminum ytterbium phase diagram.
FIG. 5 is an aluminum lutetium phase diagram.
FIG. 6A and 6B are SEM photos of gas atomized Al$_2$ aluminum alloy powder.
FIG. 7A and 7B are photomicrographs of cross-sections showing the cellular microstructure of the gas atomized inventive Al$_2$ aluminum alloy powder.
FIG. 8 is a diagram showing the processing steps to consolidate Al$_2$ aluminum alloy powder.
FIG. 9 is a photo of a 3-inch diameter copper jacketed Al$_2$ aluminum alloy billet.
FIG. 10 is a photo of extrusion dies for 3-inch diameter billet.
FIG. 11 is a photo of extruded Al$_2$ aluminum alloy rods from 3-inch diameter billets.
FIG. 12 is a photo of machined L1_2 aluminum alloy billets.
FIG. 13 is a photo of a machined three-piece L1_2 aluminum alloy billet assembly for 6-inch copper jacketed extrusion billet.
FIG. 14 is a photo of extruded L1_2 aluminum alloy rods from 6-inch diameter billets.
FIG. 15 are photos of microstructures of extruded bars in longitudinal and transverse directions.
FIG. 16 shows X-ray diffracto grams of powder and extrusions made from these powders.
FIG. 17 shows the effect of degassing the temperature on hydrogen content in extrusion.
FIG. 18 are photos showing fracture surfaces of tensile tested samples showing ductile fracture.

DETAILED DESCRIPTION

1. L1_2 Aluminum Alloys

Alloy powders of this invention are formed from aluminum based alloys with high strength and fracture toughness for applications at temperatures from about -420°F (-251°C) up to about 650°F (343°C). The aluminum alloy comprises a solid solution of aluminum and at least one element selected from silicon, magnesium, lithium, copper, zinc, and nickel strengthened by L1_2 Al_3X coherent precipitates where X is at least one first element selected from scandium, erbium, thulium, ytterbium, and lutetium, and at least one second element selected from gadolinium, yttrium, zirconium, titanium, hafnium, and niobium.

The aluminum silicon system is a simple eutectic alloy system with a eutectic reaction at 12.5 weight percent silicon and 1077°F (577°C). There is little solubility of silicon in aluminum at temperatures up to 930°F (500°C) and none of aluminum in silicon. However, the solubility can be extended significantly by utilizing rapid solidification techniques.

The binary aluminum magnesium system is a simple eutectic at 36 weight percent magnesium and 842°F (450°C). There is complete solubility of magnesium and aluminum in the rapidly solidified inventive alloys discussed herein.

The binary aluminum lithium system is a simple eutectic at 8 weight percent lithium and 1105°F (596°C). The equilibrium solubility of 4 weight percent lithium can
be extended significantly by rapid solidification techniques. There is complete solubility of lithium in the rapid solidified inventive alloys discussed herein.

The binary aluminum copper system is a simple eutectic at 32 weight percent copper and 1018°F (548°C). There is complete solubility of copper in the rapidly solidified inventive alloys discussed herein.

The aluminum zinc binary system is a eutectic alloy system involving a monotectoid reaction and a miscibility gap in the solid state. There is a eutectic reaction at 94 weight percent zinc and 718°F (381°C). Zinc has maximum solid solubility of 83.1 weight percent in aluminum at 717.8°F (381°C), which can be extended by rapid solidification processes. Decomposition of the super saturated solid solution of zinc in aluminum gives rise to spherical and ellipsoidal GP zones, which are coherent with the matrix and act to strengthen the alloy.

The aluminum nickel binary system is a simple eutectic at 5.7 weight percent nickel and 1183.8°F (639.9°C). There is little solubility of nickel in aluminum. However, the solubility can be extended significantly by utilizing rapid solidification processes. The equilibrium phase in the aluminum nickel eutectic system is L12 intermetallic Al3Ni.

In the aluminum based alloys disclosed herein, scandium, erbium, thulium, ytterbium, and lutetium are potent strengtheners that have low diffusivity and low solubility in aluminum. All these elements form equilibrium Al3X intermetallic dispersoids where X is at least one of scandium, erbium, thulium, ytterbium, and lutetium, that have an L12 structure that is an ordered face centered cubic structure with the X atoms located at the corners and aluminum atoms located on the cube faces of the unit cell.

Scandium forms Al3Sc dispersoids that are fine and coherent with the aluminum matrix. Lattice parameters of aluminum and Al3Sc are very close (0.405 nm and 0.410 nm respectively), indicating that there is minimal or no driving force for causing growth of the Al3Sc dispersoids. This low interfacial energy makes the Al3Sc dispersoids thermally stable and resistant to coarsening up to temperatures as high as about 842°F (450°C). Additions of magnesium in aluminum increase the lattice parameter of the aluminum matrix, and decrease the lattice parameter mismatch further increasing the resistance of the Al3Sc to coarsening. Additions of zinc, copper, lithium, silicon, and nickel provide solid solution and precipitation strengthening in the aluminum alloys. These Al3Sc dispersoids are made stronger and more resistant to coarsening at elevated
temperatures by adding suitable alloying elements such as gadolinium, yttrium, zirconium, titanium, hafnium, niobium, or combinations thereof, that enter Al$_3$Sc in solution.

Erbium forms Al$_3$Er dispersoids in the aluminum matrix that are fine and coherent with the aluminum matrix. The lattice parameters of aluminum and Al$_3$Er are close (0.405 nm and 0.417 nm respectively), indicating there is minimal driving force for causing growth of the Al$_3$Er dispersoids. This low interfacial energy makes the Al$_3$Er dispersoids thermally stable and resistant to coarsening up to temperatures as high as about 842°F (450°C). Additions of magnesium in aluminum increase the lattice parameter of the aluminum matrix, and decrease the lattice parameter mismatch further increasing the resistance of the Al$_3$Er to coarsening. Additions of zinc, copper, lithium, silicon, and nickel provide solid solution and precipitation strengthening in the aluminum alloys. These Al$_3$Er dispersoids are made stronger and more resistant to coarsening at elevated temperatures by adding suitable alloying elements such as gadolinium, yttrium, zirconium, titanium, hafnium, niobium, or combinations thereof that enter Al$_3$Er in solution.

Thulium forms metastable Al$_3$Tm dispersoids in the aluminum matrix that are fine and coherent with the aluminum matrix. The lattice parameters of aluminum and Al$_3$Tm are close (0.405 nm and 0.420 nm respectively), indicating there is minimal driving force for causing growth of the Al$_3$Tm dispersoids. This low interfacial energy makes the Al$_3$Tm dispersoids thermally stable and resistant to coarsening up to temperatures as high as about 842°F (450°C). Additions of magnesium in aluminum increase the lattice parameter of the aluminum matrix, and decrease the lattice parameter mismatch further increasing the resistance of the Al$_3$Tm to coarsening. Additions of zinc, copper, lithium, silicon, and nickel provide solid solution and precipitation strengthening in the aluminum alloys. These Al$_3$Tm dispersoids are made stronger and more resistant to coarsening at elevated temperatures by adding suitable alloying elements such as gadolinium, yttrium, zirconium, titanium, hafnium, niobium, or combinations thereof that enter Al$_3$Tm in solution.

Ytterbium forms Al$_3$Yb dispersoids in the aluminum matrix that are fine and coherent with the aluminum matrix. The lattice parameters of Al and Al$_3$Yb are close (0.405 nm and 0.420 nm respectively), indicating there is minimal driving force for causing growth of the Al$_3$Yb dispersoids. This low interfacial energy makes the Al$_3$Yb dispersoids thermally stable and resistant to coarsening up to temperatures as high as
about 842°F (450°C). Additions of magnesium in aluminum increase the lattice parameter of the aluminum matrix, and decrease the lattice parameter mismatch further increasing the resistance of the Al₃Yb to coarsening. Additions of zinc, copper, lithium, silicon, and nickel provide solid solution and precipitation strengthening in the aluminum alloys. These Al₃Yb dispersoids are made stronger and more resistant to coarsening at elevated temperatures by adding suitable alloying elements such as gadolinium, yttrium, zirconium, titanium, hafnium, niobium, or combinations thereof that enter Al₃Yb in solution.

Lutetium forms Al₃Lu dispersoids in the aluminum matrix that are fine and coherent with the aluminum matrix. The lattice parameters of Al and Al₃Lu are close (0.405 nm and 0.419 nm respectively), indicating there is minimal driving force for causing growth of the Al₃Lu dispersoids. This low interfacial energy makes the Al₃Lu dispersoids thermally stable and resistant to coarsening up to temperatures as high as about 842°F (450°C). Additions of magnesium in aluminum increase the lattice parameter of the aluminum matrix, and decrease the lattice parameter mismatch further increasing the resistance of the Al₃Lu to coarsening. Additions of zinc, copper, lithium, silicon, and nickel provide solid solution and precipitation strengthening in the aluminum alloys. These Al₃Lu dispersoids are made stronger and more resistant to coarsening at elevated temperatures by adding suitable alloying elements such as gadolinium, yttrium, zirconium, titanium, hafnium, niobium, or mixtures thereof that enter Al₃Lu in solution.

Gadolinium forms metastable Al₃Gd dispersoids in the aluminum matrix that are stable up to temperatures as high as about 842°F (450°C) due to their low diffusivity in aluminum. The Al₃Gd dispersoids have a DO₉ structure in the equilibrium condition. Despite its large atomic size, gadolinium has fairly high solubility in the Al₃X intermetallic dispersoids (where X is scandium, erbium, thulium, ytterbium or lutetium). Gadolinium can substitute for the X atoms in Al₃X intermetallic, thereby forming an ordered L₁₂ phase which results in improved thermal and structural stability.

Yttrium forms metastable Al₃Y dispersoids in the aluminum matrix that have an L₁₂ structure in the metastable condition and a DO₁₉ structure in the equilibrium condition. The metastable Al₃Y dispersoids have a low diffusion coefficient, which makes them thermally stable and highly resistant to coarsening. Yttrium has a high solubility in the Al₃X intermetallic dispersoids allowing large amounts of yttrium to substitute for X in the Al₃X L₁₂ dispersoids, which results in improved thermal and structural stability.
Zirconium forms Al$_3$Zr dispersoids in the aluminum matrix that have an L1$_2$ structure in the metastable condition and DO$_{23}$ structure in the equilibrium condition. The metastable Al$_3$Zr dispersoids have a low diffusion coefficient, which makes them thermally stable and highly resistant to coarsening. Zirconium has a high solubility in the Al$_3$X dispersoids allowing large amounts of zirconium to substitute for X in the Al$_3$X dispersoids, which results in improved thermal and structural stability.

Titanium forms Al$_3$Ti dispersoids in the aluminum matrix that have an L1$_2$ structure in the metastable condition and DO$_{22}$ structure in the equilibrium condition. The metastable Al$_3$Ti dispersoids have a low diffusion coefficient, which makes them thermally stable and highly resistant to coarsening. Titanium has a high solubility in the Al$_3$X dispersoids allowing large amounts of titanium to substitute for X in the Al$_3$X dispersoids, which result in improved thermal and structural stability.

Hafnium forms metastable Al$_3$Hf dispersoids in the aluminum matrix that have an L1$_2$ structure in the metastable condition and a DO$_{23}$ structure in the equilibrium condition. The Al$_3$Hf dispersoids have a low diffusion coefficient, which makes them thermally stable and highly resistant to coarsening. Hafnium has a high solubility in the Al$_3$X dispersoids allowing large amounts of hafnium to substitute for scandium, erbium, thulium, ytterbium, and lutetium in the above-mentioned Al$_3$X dispersoids, which results in stronger and more thermally stable dispersoids.

Niobium forms metastable Al$_3$Nb dispersoids in the aluminum matrix that have an L1$_2$ structure in the metastable condition and a DO$_{23}$ structure in the equilibrium condition. Niobium has a lower solubility in the Al$_3$X dispersoids than hafnium or yttrium, allowing relatively lower amounts of niobium than hafnium or yttrium to substitute for X in the Al$_3$X dispersoids. Nonetheless, niobium can be very effective in slowing down the coarsening kinetics of the Al$_3$X dispersoids because the Al$_3$Nb dispersoids are thermally stable. The substitution of niobium for X in the above mentioned Al$_3$X dispersoids results in stronger and more thermally stable dispersoids.

Al$_3$X L1$_2$ precipitates improve elevated temperature mechanical properties in aluminum alloys for two reasons. First, the precipitates are ordered intermetallic compounds. As a result, when the particles are sheared by glide dislocations during deformation, the dislocations separate into two partial dislocations separated by an anti-phase boundary on the glide plane. The energy to create the anti-phase boundary is the origin of the strengthening. Second, the cubic L1$_2$ crystal structure and lattice parameter of the precipitates are closely matched to the aluminum solid solution matrix. This
results in a lattice coherency at the precipitate/matrix boundary that resists coarsening. The lack of an interphase boundary results in a low driving force for particle growth and resulting elevated temperature stability. Alloying elements in solid solution in the dispersed strengthening particles and in the aluminum matrix that tend to decrease the lattice mismatch between the matrix and particles will tend to increase the strengthening and elevated temperature stability of the alloy.

\(L1_2\) phase strengthened aluminum alloys are important structural materials because of their excellent mechanical properties and the stability of these properties at elevated temperature due to the resistance of the coherent dispersoids in the microstructure to particle coarsening. The mechanical properties are optimized by maintaining a high volume fraction of \(L1_2\) dispersoids in the microstructure. The \(L1_2\) dispersoid concentration following aging scales as the amount of \(L1_2\) phase forming elements in solid solution in the aluminum alloy following quenching. Examples of \(L1_2\) phase forming elements include but are not limited to Sc, Er, Th, Yb, and Lu. The concentration of alloying elements in solid solution in alloys cooled from the melt is directly proportional to the cooling rate.

Exemplary aluminum alloys for this invention include, but are not limited to (in weight percent unless otherwise specified):

- about Al-M-(O.1-4)Sc-(0.1-20)Gd;
- about Al-M-(0.1-20)Er-(0.1-20)Gd;
- about Al-M-(O.1-15)Tm-(0.1-20)Gd;
- about Al-M-(O.1-25)Yb-(0.1-20)Gd;
- about Al-M-(O.1-25)Lu-(0.1-20)Gd;
- about Al-M-(O.1-4)Sc-(0.1-20)Y;
- about Al-M-(0.1-20)Er-(0.1-20)Y;
- about Al-M-(O.1-15)Tm-(0.1-20)Y;
- about Al-M-(O.1-25)Yb-(0.1-20)Y;
- about Al-M-(O.1-25)Lu-(0.1-20)Y;
- about Al-M-(O.1-4)Sc-(0.05-4)Zr;
- about Al-M-(0.1-20)Er-(0.05-4)Zr;
- about Al-M-(O.1-15)Tm-(0.05-4)Zr;
- about Al-M-(O.1-25)Yb-(0.05-4)Zr;
- about Al-M-(O.1-25)Lu-(0.05-4)Zr;
- about Al-M-(O.1-4)Sc-(0.05-10)Ti;
about Al-M-(O.1-20)Er-(0.05-10)Ti;
about Al-M-(O.1-15)Tm-(0.05-10)Ti;
about Al-M- (0.1-25) Yb-(0.05-10)Ti;
about Al-M-(O.1-25)Lu-(0.05-10)Ti;
about Al-M-(O.1-20)Er-(0.05-10)Hf;
about Al-M-(O.1-15)Tm-(0.05-10)Hf;
about Al-M-(O.1-25)Yb-(0.05- 10)Hf;
about Al-M-(O.1-25)Lu-(0.05-10)Hf;
about Al-M-(O.1-4)Sc-(0.05- 10)Nb;
about Al-M-(O.1-20)Er-(0.05-10)Nb;
about Al-M-(O.1-15)Tm-(0.05-10)Nb;
about Al-M-(O.1-25)Yb-(0.05-10)Nb; and
about Al-M-(O.1-25)Lu-(0.05-10)Nb.

M is at least one of about (4-25) weight percent silicon, (1-8) weight percent magnesium, (0.5-3) weight percent lithium, (0.2-6) weight percent copper, (3-12) weight percent zinc, and (1-12) weight percent nickel.

The amount of silicon present in the fine grain matrix, if any, may vary from about 4 to about 25 weight percent, more preferably from about 4 to about 18 weight percent, and even more preferably from about 5 to about 11 weight percent.

The amount of magnesium present in the fine grain matrix, if any, may vary from about 1 to about 8 weight percent, more preferably from about 3 to about 7.5 weight percent, and even more preferably from about 4 to about 6.5 weight percent.

The amount of lithium present in the fine grain matrix, if any, may vary from about 0.5 to about 3 weight percent, more preferably from about 1 to about 2.5 weight percent, and even more preferably from about 1 to about 2 weight percent.

The amount of copper present in the fine grain matrix, if any, may vary from about 0.2 to about 6 weight percent, more preferably from about 0.5 to about 5 weight percent, and even more preferably from about 2 to about 4.5 weight percent.

The amount of zinc present in the fine grain matrix, if any, may vary from about 3 to about 12 weight percent, more preferably from about 4 to about 10 weight percent, and even more preferably from about 5 to about 9 weight percent.
The amount of nickel present in the fine grain matrix, if any, may vary from about 1 to about 12 weight percent, more preferably from about 2 to about 10 weight percent, and even more preferably from about 4 to about 10 weight percent.

The amount of scandium present in the fine grain matrix, if any, may vary from 0.1 to about 4 weight percent, more preferably from about 0.1 to about 3 weight percent, and even more preferably from about 0.2 to about 2.5 weight percent. The Al-Sc phase diagram shown in FIG. 1 indicates a eutectic reaction at about 0.5 weight percent scandium at about 1219°F (659°C) resulting in a solid solution of scandium and aluminum and Al₃Sc dispersoids. Aluminum alloys with less than 0.5 weight percent scandium can be quenched from the melt to retain scandium in solid solution that may precipitate as dispersed L₁₂ intermetallic Al₃Sc following an aging treatment. Alloys with scandium in excess of the eutectic composition (hypereutectic alloys) can only retain scandium in solid solution by rapid solidification processing (RSP) where cooling rates are in excess of about 10³°C/second.

The amount of erbium present in the fine grain matrix, if any, may vary from about 0.1 to about 20 weight percent, more preferably from about 0.3 to about 15 weight percent, and even more preferably from about 0.5 to about 10 weight percent. The Al-Er phase diagram shown in FIG. 2 indicates a eutectic reaction at about 6 weight percent erbium at about 1211°F (655°C). Aluminum alloys with less than about 6 weight percent erbium can be quenched from the melt to retain erbium in solid solutions that may precipitate as dispersed L₁₂ intermetallic Al₃Er following an aging treatment. Alloys with erbium in excess of the eutectic composition can only retain erbium in solid solution by rapid solidification processing (RSP) where cooling rates are in excess of about 10³°C/second.

The amount of thulium present in the alloys, if any, may vary from about 0.1 to about 15 weight percent, more preferably from about 0.2 to about 10 weight percent, and even more preferably from about 0.4 to about 6 weight percent. The Al-Tm phase diagram shown in FIG. 3 indicates a eutectic reaction at about 10 weight percent thulium at about 1193°F (645°C). Thulium forms metastable Al₃Tm dispersoids in the aluminum matrix that have an L₁₂ structure in the equilibrium condition. The Al₃Tm dispersoids have a low diffusion coefficient, which makes them thermally stable and highly resistant to coarsening. Aluminum alloys with less than 10 weight percent thulium can be quenched from the melt to retain thulium in solid solution that may precipitate as dispersed metastable L₁₂ intermetallic Al₃Tm following an aging treatment. Alloys with
thulium in excess of the eutectic composition can only retain Tm in solid solution by rapid solidification processing (RSP) where cooling rates are in excess of about $10^{30}$C/second.

The amount of ytterbium present in the alloys, if any, may vary from about 0.1 to about 25 weight percent, more preferably from about 0.3 to about 20 weight percent, and even more preferably from about 0.4 to about 10 weight percent. The Al-Yb phase diagram shown in FIG. 4 indicates a eutectic reaction at about 21 weight percent ytterbium at about 1157°F (625°C). Aluminum alloys with less than about 21 weight percent ytterbium can be quenched from the melt to retain ytterbium in solid solution that may precipitate as dispersed L12 intermetallic Al$_3$Yb following an aging treatment. Alloys with ytterbium in excess of the eutectic composition can only retain ytterbium in solid solution by rapid solidification processing (RSP) where cooling rates are in excess of about $10^{30}$C/second.

The amount of lutetium present in the alloys, if any, may vary from about 0.1 to about 25 weight percent, more preferably from about 0.3 to about 20 weight percent, and even more preferably from about 0.4 to about 10 weight percent. The Al-Lu phase diagram shown in FIG. 5 indicates a eutectic reaction at about 11.7 weight percent Lu at about 1202°F (650°C). Aluminum alloys with less than about 11.7 weight percent lutetium can be quenched from the melt to retain Lu in solid solution that may precipitate as dispersed L12 intermetallic Al$_3$Lu following an aging treatment. Alloys with Lu in excess of the eutectic composition can only retain Lu in solid solution by rapid solidification processing (RSP) where cooling rates are in excess of about $10^{30}$C/second.

The amount of gadolinium present in the alloys, if any, may vary from about 0.1 to about 20 weight percent, more preferably from about 0.3 to about 15 weight percent, and even more preferably from about 0.5 to about 10 weight percent.

The amount of yttrium present in the alloys, if any, may vary from about 0.1 to about 20 weight percent, more preferably from about 0.3 to about 15 weight percent, and even more preferably from about 0.5 to about 10 weight percent.

The amount of zirconium present in the alloys, if any, may vary from about 0.05 to about 4 weight percent, more preferably from about 0.1 to about 3 weight percent, and even more preferably from about 0.3 to about 2 weight percent.

The amount of titanium present in the alloys, if any, may vary from about 0.05 to about 10 weight percent, more preferably from about 0.2 to about 8 weight percent, and even more preferably from about 0.4 to about 4 weight percent.
The amount of hafnium present in the alloys, if any, may vary from about 0.05 to
about 10 weight percent, more preferably from about 0.2 to about 8 weight percent, and
even more preferably from about 0.4 to about 5 weight percent.

The amount of niobium present in the alloys, if any, may vary from about 0.05 to
about 5 weight percent, more preferably from about 0.1 to about 3 weight percent, and
even more preferably from about 0.2 to about 2 weight percent.

In order to have the best properties for the fine grain matrix, it is desirable to limit
the amount of other elements. Specific elements that should be reduced or eliminated
include no more than about 0.1 weight percent iron, 0.1 weight percent chromium, 0.1
weight percent manganese, 0.1 weight percent vanadium, and 0.1 weight percent cobalt.
The total quantity of additional elements should not exceed about 1% by weight,
including the above listed impurities and other elements.

2. Consolidation of Aluminum L1₂ Alloy Powder

Gas atomized high temperature L1₂ aluminum alloy powder needs to be
consolidated into solid-state forms suitable for engineering applications. Scanning
electron micrographs of the inventive gas atomized L1₂ aluminum alloy powder are
shown in FIGS. 6A and 6B. The powder is spherical and capable of high packing
density. As a result of the high solidification rate, e.g. greater than 10³⁰C/second, the
microstructure is a finely divided cellular structure instead of a dendritic structure
common to conventionally cooled alloys. SEM photos illustrating the fine cellular
structure of the L1₂ aluminum powder are shown in FIGS. 7A and 7B. The fine structure
allows for a uniform distribution of alloying elements and resulting even dispersion of
L1₂ strengthening dispersoids in the final consolidated alloy structure. The process of
consolidating the alloy powders into useful forms is schematically illustrated in FIG. 8.

L1₂ aluminum alloy powders 10 are first classified according to size by sieving
(step 20). Fine particle sizes are required for optimum mechanical properties in the final
part.

Sieving (step 20) is a critical step in consolidation because the final mechanical
properties relate directly to the particle size. Finer particle size results in finer L1₂
particle dispersion. Sufficient mechanical properties have been observed with -450
mesh (30 micron) powder. Sieving (step 20) also limits the defect size in the powder.
Before sieving, the powder is passivated with nitrogen gas in order to minimize reaction
of the powder with atmosphere. The powder is stored in a nitrogen atmosphere to prevent
oxidation. However, if the powder is completely free from oxides, it sticks together
reducing the efficiency of sieving. If oxygen in the powder is too high, it has a deleterious effect on mechanical properties. There is an optimal oxygen level which is desired so that it does not create problems with sieving and yields good mechanical properties. The oxygen content of the powder is between about 1 ppm and 2000 ppm, preferred between about 10 ppm to 1000 ppm and most preferred between about 25 ppm to about 500 ppm. Ultrasonic sieving is preferred for its efficiency.

Blending (step 30) is a preferred step in the consolidation process because it results in improved uniformity of particle size distribution. Gas atomized L1$_2$ aluminum alloy powder generally exhibits a bimodal particle size distribution and cross blending of separate powder batches tends to homogenize the particle size distribution. Blending (step 30) is also preferred when separate metal and/or ceramic powders are added to the L1$_2$ base powder to form bimodal or trimodal consolidated alloy microstructures.

Following sieving (step 20) and blending (step 30), the powders are transferred to can (step 50) where the powder is vacuum degassed (step 60) at elevated temperatures. The can (step 50) is an aluminum container having a cylindrical, rectangular or other configuration with a central axis. Vacuum degassing times can range from about 0.5 hours to about 8 days, more preferably it can range from about 4 hours to 7 days, even more preferably it can range from about 8 hours to about 6 days. A temperature range of about 300°F (149°C) to about 900°F (482°C) is preferred and about 600°F (316°C) to about 850°F (454°C) is more preferred and 650°F (343°C) to about 850°F(454°C) is most preferred. Dynamic degassing of large amounts of powder is preferred to static degassing. In dynamic degassing, the can is preferably rotated during degassing to expose all of the powder to a uniform temperature. Degassing removes oxygen and hydrogen from the powder.

The role of dynamic degassing is to remove oxygen and hydrogen more efficiently than static degassing. Dynamic degassing is very important for large billets to reduce time and temperature required for degassing. Static degassing works well for small sizes of billets and small quantity of powder as it does not take long time to degas effectively. For large billets, it can take several days to degas at high temperatures which can coarsen the material microstructure and reduce the strength. In addition, the process efficiency goes down with longer time for degassing.

Following vacuum degassing (step 60), the vacuum line is crimped and welded shut. The powder is then consolidated further by unaxially hot pressing the evacuated can along its central axis while radial movement is restrained in a die or by hot isostatic
pressing (HIP) the can in an isostatic press. The billet can be compressed by blind die compaction (step 90) to further densify the structure if it is not 100% dense. At this point the can may be removed by machining.

Following blind die compaction, the billet is machined into an extrusion billet, copper jacketed and extruded (step 100). Alternatively, the billet can be extruded directly after blind die compaction without machining and without a copper jacket. A copper jacket is preferred to provide improved lubrication. However, it is not essential for extrusion of billets. The extrusion process preferably improves the hardness and improves the tensile ductility. Extrusion imparts directional mechanical properties to the material. Forging and/or rolling (step 110) can improve the transverse mechanical properties leading to isotropic properties.

FIG. 9 shows a 3-inch diameter copper jacketed L12 aluminum alloy billet ready for extrusion. FIG. 10 is a photo of three 3-inch diameter extrusion dies. Representative extrusions using the 3-inch diameter dies are shown in FIG. 11. A 12-inch ruler is included in the photo for size comparison. Larger 6-inch diameter billets were also extruded. Machined 6-inch diameter L12 aluminum alloy extrusion billets are shown in FIG. 12. FIG. 13 is a photo of a machined three-piece copper jacketed 6-inch diameter billet assembly. A 12-inch ruler is included in the photo for size comparison. The upright cylinder behind the three-piece assembly is another machined, copper jacketed L12 aluminum alloy extrusion billet.

Extruded L12 aluminum alloy rods from 6-inch diameter billets are shown in FIG. 14. The top rod is 46 inches long.

Representative processing parameters for 3-inch diameter L12 aluminum alloy billets are listed in Table 1.

Table 1.

<table>
<thead>
<tr>
<th>Billet #</th>
<th>Powder Degassing Temp/Time</th>
<th>Vacuum Hot Pressing Temp/Time</th>
<th>Extrusion Billet Temp/Die and Container Temp</th>
<th>Breakthrough Load</th>
<th>Extrusion Speed</th>
<th>Extrusion Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>500°F/19h</td>
<td>500°F/1h</td>
<td>700°F/650°F</td>
<td>640 Tons</td>
<td>0.5”/min</td>
<td>10:01</td>
</tr>
<tr>
<td>2</td>
<td>550°F/19h</td>
<td>550°F/1h</td>
<td>600°F/660°F</td>
<td>601 Tons</td>
<td>0.75”/min</td>
<td>6:01</td>
</tr>
<tr>
<td>3</td>
<td>600°F/19h</td>
<td>600°F/1h</td>
<td>650°F/650°F</td>
<td>648 Tons</td>
<td>0.5”/min</td>
<td>10:01</td>
</tr>
<tr>
<td>4</td>
<td>650°F/19h</td>
<td>700°F/1h</td>
<td>700°F/650°F</td>
<td>655 Tons</td>
<td>0.5”/min</td>
<td>10:01</td>
</tr>
<tr>
<td>5</td>
<td>700°F/19h</td>
<td>700°F/1h</td>
<td>700°F/650°F</td>
<td>634 Tons</td>
<td>0.5”/min</td>
<td>10:01</td>
</tr>
<tr>
<td>6</td>
<td>700°F/19h</td>
<td>700°F/1h</td>
<td>600°F/660°F</td>
<td>621 Tons</td>
<td>0.5”/min</td>
<td>10:01</td>
</tr>
<tr>
<td>7</td>
<td>750°F/19h</td>
<td>700°F/1h</td>
<td>700°F/650°F</td>
<td>550 Tons</td>
<td>0.5”/min</td>
<td>10:01</td>
</tr>
<tr>
<td>8</td>
<td>600°F/19h</td>
<td>700°F/1h</td>
<td>700°F/650°F</td>
<td>612 Tons</td>
<td>0.5”/min</td>
<td>10:01</td>
</tr>
</tbody>
</table>
Table 1 shows powder processing data that includes degassing temperature, time, consolidation temperature and time, extrusion temperature, ratio and load experienced during extrusion, extrusion die and billet temperatures. These processing parameters were used to degas, consolidate and extrude $L_1$ aluminum alloy powders. The degassing temperature range of $500^0F$ to $750^0F$ ($260^0C$ to $399^0C$) for a constant degas time of 19 hours was evaluated. Vacuum hot pressing at a temperature range of $500^0F$ to $700^0F$ ($260^0C$ to $371^0C$) for a constant time of 1 hour was evaluated. Since the billet does not usually have good ductility to provide sufficient integrity for testing, billets were extruded for providing deformation to impart ductility in the billet. Extrusion billet temperature, die temperature and container temperature varied from $650^0F$ to $700^0F$ ($343^0C$ to $371^0C$). Extrusion speed varied from 0.5 inch per minute to 0.75 inch per minute and extrusion ratio varied from 6:1 to 10:1. Extrusion load varied from 550 tons to 655 tons depending on process parameters used for the powder. Breakthrough load depends on degassing temperature and vacuum hot pressing temperature in addition to extrusion temperature, extrusion speed and extrusion ratio. Breakthrough load decreased with an increase in degassing temperature and vacuum hot pressing temperature. The load has decreased from 640 tons to 550 tons as we increased the degassing temperature from $500^0F$ to $750^0F$ ($260^0C$ to $399^0C$). Breakaway load is important in order to make successful extrusions. If the load requirement is higher than the capacity of the extrusion press, then the press will stall and material will not be extruded. It is very important to select the degassing and vacuum hot pressing temperature in such a way that successful extrusions are produced with good mechanical properties.

Microstructures of extruded bars in longitudinal and transverse directions are shown in FIG. 15. The microstructures in longitudinal direction show deformation bands from the extrusion process. The transverse microstructures show more uniform microstructures without any deformation bands. The grain size cannot be resolved by optical microscopy as it is very fine. Very fine dispersoids are present in the material as shown in FIG. 15.

FIG. 16 shows X-ray diffractograms of powder and extrusion. The powder diffractogram shows only two phases: aluminum and aluminum nickel. Since the lattice parameters of aluminum and $Al_2Sc$ dispersoids are very similar, the peaks for aluminum and $Al_2Sc$ dispersoids cannot be resolved. The extrusion diffractogram shows additional phases based on gadolinium nickel and nickel zirconium. These phases were produced during powder processing.
FIG. 17 shows the hydrogen content in extrusions produced from powders which were degassed at different temperatures from 500°F to 750°F (260°C to 399°C). As clearly evident, the hydrogen content in the extrusions from powder degassed at 500°F to 600°F (260°C to 343°C) were higher than those degassed at 650°F-750°F (343°C to 399°C). 650°F (343°C) is a critical temperature above which degassing is more effective in L12 powder. There is no appreciable benefit in degassing at higher temperature than 650°F (343°C) in terms of hydrogen content for a constant time of 19 hours. If time is varied for degassing, results will change based on diffusion kinetics. For a given temperature, longer time will give better degassing based on diffusion kinetics of L12 powder. It is desired to have low hydrogen in the material as hydrogen has deleterious effects on ductility of the material.

Representative mechanical properties of extruded aluminum alloy billets are listed in Table 2.

Table 2: Tensile properties of extruded L12 aluminum alloy

<table>
<thead>
<tr>
<th>Billet #</th>
<th>Ultimate Tensile Strength, ksi</th>
<th>Yield Strength, ksi</th>
<th>Elongation, %</th>
<th>Reduction in Area, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>115.4</td>
<td>102.4</td>
<td>4</td>
<td>4.3</td>
</tr>
<tr>
<td>2</td>
<td>113.2</td>
<td>100.8</td>
<td>4</td>
<td>13</td>
</tr>
<tr>
<td>3</td>
<td>115.4</td>
<td>104.2</td>
<td>3.7</td>
<td>8.5</td>
</tr>
<tr>
<td>4</td>
<td>113.5</td>
<td>101.9</td>
<td>5.4</td>
<td>7.9</td>
</tr>
<tr>
<td>5</td>
<td>110</td>
<td>101.3</td>
<td>4.7</td>
<td>15</td>
</tr>
<tr>
<td>6</td>
<td>101.2</td>
<td>93.2</td>
<td>10.3</td>
<td>18</td>
</tr>
<tr>
<td>7</td>
<td>107.8</td>
<td>96.1</td>
<td>6.6</td>
<td>17.5</td>
</tr>
<tr>
<td>8</td>
<td>115.9</td>
<td>101.7</td>
<td>5</td>
<td>8.5</td>
</tr>
</tbody>
</table>

Table 2 shows tensile properties of extrusions made out of powders degassed at different temperatures. The yield strength and ultimate tensile strength of these L12 based alloys are excellent. These strength values are much higher than the strengths of commercial aluminum alloys including 6061 Al, 2124 Al and 7075 Al suggesting that processing parameters used for making this material in this invention has worked well. The tensile strength over 100 ksi for L12 aluminum alloy is remarkable as it can provide significant weight savings by replacing high strength aluminum alloy, titanium nickel and steel alloys. In addition, the elongation and reduction in area values for this L12 alloy are also very good. The yield strength remains fairly constant over 100 ksi for degassing and vacuum hot pressing temperature range of 500°F-650°F (260°C to 343°C). The yield
strength decreased slightly for degassing and vacuum hot pressing temperature range of 700°F to 750°F (371°C to 399°C). The ductility measured by elongation and reduction in area, however, increased significantly with an increase in degassing temperature. Reduction in area has increased almost two times for material degassed in temperature range of 700°F-750°F (371°C to 399°C) compared to material that was degassed in temperature range of 500°F-650°F (260°C to 343°C). These results are expected based on strengthening models including an Orowan strengthening model and a Hall-Petch strengthening model. Vacuum degassing is more effective when the powder is degassed at higher temperatures as indicated by lower hydrogen content. Lower hydrogen results in higher ductility of material as measured by elongation and reduction in area. However, strength is expected to decrease with an increase in degassing temperature because strengthening precipitates would start to coarsen with an increase in degassing temperature, which is consistent with observed results from material degassed at 700°F-750°F (371°C to 399°C). These results indicate that properties of L12 alloy can be varied by controlling the degassing and vacuum hot pressing temperature. In order to have a balanced combination of strength and ductility in material, L12 alloy needs to be degassed and vacuum hot pressed at recommended temperatures and times. The results obtained here demonstrate that the present invention worked very well.

Figure 18 shows fracture surfaces of tensile tested samples. The fracture surfaces show presence of dimples indicating ductile fracture where void nucleates, grows and finally coalesces to failure. The fracture surface morphology provides an evidence of ductile failure mode which is consistent with good elongation and reduction in area values.

Although the present invention has been described with reference to preferred embodiments, workers skilled in the art will recognize that changes may be made in form and detail without departing from the spirit and scope of the invention.
CLAIMS:
1. A method for producing a high strength aluminum alloy billet containing \( \text{L}_1\text{L}_2 \) dispersoids, comprising the steps of:
   - placing a quantity of an aluminum alloy powder containing an \( \text{L}_1\text{L}_2 \) dispersoid therein having a mesh size of less than 450 mesh in a container;
   - vacuum degassing the powder at a temperature of about 300\(^\circ\)F (149\(^\circ\)C) to about 900\(^\circ\)F (482\(^\circ\)C) for about 0.5 hours to about 8 days;
   - sealing the degassed powder in the container under vacuum;
   - heating the sealed container at about 300\(^\circ\)F (149\(^\circ\)C) to about 900\(^\circ\)F (482\(^\circ\)C) for about 15 minutes to eight hours;
   - vacuum hot pressing the heated container to form a billet; and removing the container from the formed billet.
2. The method of claim 1, wherein the container is aluminum having a configuration with a central axis, and vacuum hot pressing is done along the axis while restraining radial movement of the container.
3. The method of claim 1, wherein the vacuum hot pressing includes blind die compaction for about 1 minute to about 8 hours at a temperature of 300\(^\circ\)F (149\(^\circ\)C) to about 900\(^\circ\)F (482\(^\circ\)C) under uni-axial pressure of about 5 ksi to about 100 ksi.
4. The method of claim 1, wherein the vacuum hot pressing produces a billet of the aluminum alloy powder having a density of about 100 percent.
5. The method of claim 1, wherein the degassing includes rotating the aluminum alloy powder to heat and expose all the powder to vacuum.
6. The method of claim 1, wherein the thus formed billet is extruded at a load of about 100 tons to about 10,000 tons.
7. The method of claim 6, wherein the extrusion temperature is about 300\(^\circ\)F (149\(^\circ\)C) to about 900\(^\circ\)F (482\(^\circ\)C), the billet soak time is about 15 minutes to about 8 hours at a rate of about 0.2 inch per minute to about 20 inches per minute and an extrusion ratio of about 2:1 to about 500:1.
8. The method of claim 1, wherein the \( \text{L}_1\text{L}_2 \) dispersoids comprise \( \text{Al}_3\text{X} \) dispersoids wherein \( \text{X} \) is at least one first element selected from the group comprising:
   - about 0.1 to about 4.0 weight percent scandium,
   - about 0.1 to about 20.0 weight percent erbium,
   - about 0.1 to about 15.0 weight percent thulium,
   - about 0.1 to about 25.0 weight percent ytterbium, and
   - about 0.1 to about 25.0 weight percent lutetium.
at least one second element selected from the group comprising about 0.1 to about 20.0 weight percent gadolinium, about 0.1 to about 20.0 weight percent yttrium, about 0.05 to about 4.0 weight percent zirconium, about 0.05 to about 10.0 weight percent titanium, about 0.05 to about 10.0 weight percent hafnium, and about 0.05 to about 5.0 weight percent niobium; and the balance substantially aluminum.

9. The method of claim 8, wherein the aluminum alloy powder contains at least one third element selected from the group consisting of silicon, magnesium, lithium, copper, zinc, and nickel.

10. The method of claim 9, wherein the third element comprises at least one of about 4 to about 25 weight percent silicon, about 1 to about 8 weight percent magnesium, about 0.5 to about 3 weight percent lithium, about 0.2 to about 6 weight percent copper, about 3 to about 12 weight percent zinc, about 1 to about 12 weight percent nickel.

11. The method of claim 8, wherein the \( \text{L1}_2 \) dispersoids comprise \( \text{Al}_3\text{X} \) dispersoids wherein \( \text{X} \) is at least one first element selected from the group comprising:

   - about 0.1 to about 0.5 weight percent scandium, about 0.1 to about 6.0 weight percent erbium, about 0.1 to about 10.0 weight percent thulium, about 0.1 to about 15.0 weight percent ytterbium, and about 0.1 to about 12.0 weight percent lutetium;

   at least one second element selected from the group comprising about 0.1 to about 4.0 weight percent gadolinium, about 0.1 to about 4.0 weight percent yttrium, about 0.05 to about 1.0 weight percent zirconium, about 0.05 to about 2.0 weight percent titanium, about 0.05 to about 2.0 weight percent hafnium, and about 0.05 to about 1.0 weight percent niobium; and

   the balance substantially aluminum.

12. Apparatus for producing a high strength aluminum alloy billet containing \( \text{L1}_2 \) dispersoids, comprising:

   - a container for holding a quantity of an aluminum alloy powder containing an \( \text{L1}_2 \) dispersoid therein having a mesh size of less than 450 mesh;

   - a vacuum and heat source for degassing the powder at a temperature of about 300°F (149°C) to about 900°F (482°C) for about 0.5 hours to about 8 days;

   - sealing means for sealing the degassed powder in the container under vacuum;
a heater for heating the sealed container at about 300°F (149°C) to about 900°F (482°C) for about 15 minutes to eight hours;

a vacuum hot press for forming the heated container into a billet; and means for removing the container from the thus formed billet.

13. The apparatus of claim 12, wherein the container is aluminum having a central axis, and vacuum hot pressing is done along the axis while restraining transverse movement of the container.

14. The apparatus of claim 12, wherein the vacuum hot pressing includes blind die compaction for about 1 minute to about 8 hours at a temperature of 300°F (149°C) to about 900°F (482°C) under uni-axial pressure of about 5 ksi to about 100 ksi.

15. The apparatus of claim 12, wherein the vacuum hot pressing produces a billet of the aluminum alloy powder having a density of about 100 percent.

16. The apparatus of claim 12, wherein the degassing includes rotating the aluminum alloy powder to heat and exposing to vacuum all the powder.

17. The apparatus of claim 12, wherein the formed billet is extruded at a load of about 100 tons to about 10,000 tons.

18. The apparatus of claim 17, wherein the extrusion temperature is about 300°F (149°C) to about 900°F (482°C), the billet soak time is about 15 minutes to about 8 hours at a rate of about 0.2 inch per minute to about 20 inches per minute, and an extrusion ratio of about 2:1 to about 500:1.

19. The apparatus of claim 12, wherein the L12 dispersoids comprise Al2X dispersoids wherein X is at least one first element selected from the group comprising:

- about 0.1 to about 4.0 weight percent scandium,
- about 0.1 to about 20.0 weight percent erbium,
- about 0.1 to about 15.0 weight percent thulium,
- about 0.1 to about 25.0 weight percent ytterbium,
- and about 0.1 to about 25.0 weight percent lutetium;

at least one second element selected from the group comprising about 0.1 to about 20.0 weight percent gadolinium,

about 0.1 to about 20.0 weight percent yttrium,

about 0.05 to about 4.0 weight percent zirconium,

about 0.05 to about 10.0 weight percent titanium,

about 0.05 to about 10.0 weight percent hafnium, and

about 0.05 to about 5.0 weight percent niobium; and the balance substantially aluminum.
20. The apparatus of claim 19, wherein the aluminum alloy powder contains at least one third element selected from the group consisting of silicon, magnesium, lithium, copper, zinc, and nickel.

21. The apparatus of claim 20, wherein the third element comprises at least one of about 4 to about 25 weight percent silicon, about 1 to about 8 weight percent magnesium, about 0.5 to about 3 weight percent lithium, about 0.2 to about 6 weight percent copper, about 3 to about 12 weight percent zinc, about 1 to about 12 weight percent nickel.

22. The apparatus of claim 19, wherein the L12 dispersoids comprise AlX dispersoids wherein X is at least one first element selected from the group comprising:

- about 0.1 to about 0.5 weight percent scandium, about 0.1 to about 6.0 weight percent erbium, about 0.1 to about 10.0 weight percent thulium, about 0.1 to about 15.0 weight percent ytterbium, and about 0.1 to about 12.0 weight percent lutetium;

at least one second element selected from the group comprising about 0.1 to about 4.0 weight percent gadolinium, about 0.1 to about 4.0 weight percent yttrium, about 0.05 to about 1.0 weight percent zirconium, about 0.05 to about 2.0 weight percent titanium, about 0.05 to about 2.0 weight percent hafnium, and about 0.05 to about 1.0 weight percent niobium; and the balance substantially aluminum.
FIG. 9

FIG. 10

FIG. 11