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(54) Ceracon forging of L12 aluminum alloys

(57) A method for producing high strength aluminum alloy consolidated billets containing $L1_2$ dispersoids by Ceracon forging is disclosed. The method comprises forming an aluminum alloy powder compact preform containing $L1_2$ dispersoid forming elements therein and encompassing the preform in a flowable pressure transmitting medium in a die in a hydraulic press. The die, pressure transmitting medium and preform are then heated and the preform is forged by applying pressure to the pressure transmitting medium by the ram of the hydraulic press. The unequal axial and radial strain resulting from this type of forging results in improved mechanical properties of $L1_2$ aluminum alloys.

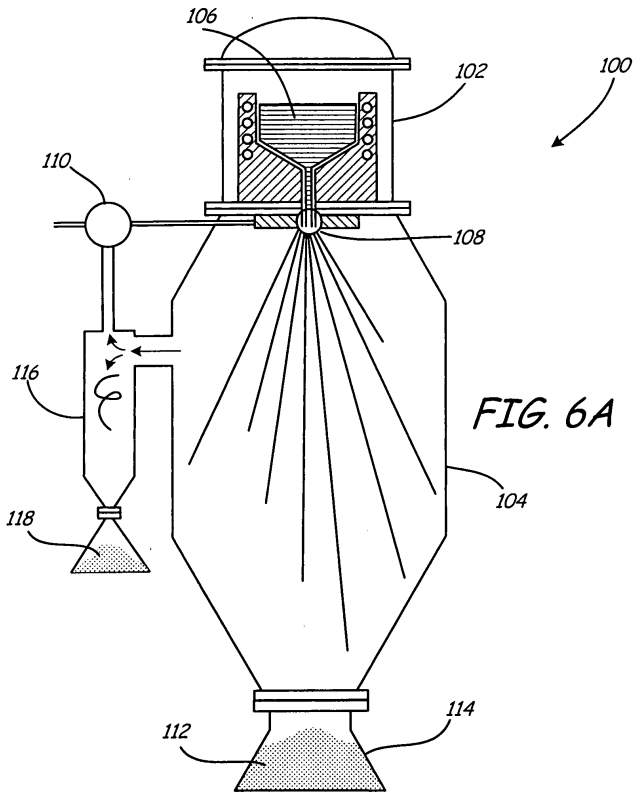


FIG. 6A

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Description

[0001] The present invention relates generally to aluminum alloys and more specifically to a method for forming high strength aluminum alloy billets having $L1_2$ dispersoids therein.

[0002] 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.

[0003] 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.

[0004] 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.

[0005] U.S. Patent No. 6,248,453 owned by the assignee of the present invention discloses aluminum alloys strengthened by dispersed Al_3X $L1_2$ intermetallic phases where X is selected from the group consisting of Sc, Er, Lu, Yb, Tm, and Lu. The Al_3X 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 $L1_2$ aluminum alloys are stable up to 572°F (300°C). U.S. Patent Application Publication No. 2006/0269437 Al also owned commonly discloses a high strength aluminum alloy that contains scandium and other elements that is strengthened by $L1_2$ dispersoids.

[0006] $L1_2$ 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 nano range of about 30 to 100 nm. These alloys also have lower ductility.

[0007] The present invention is a method for forming aluminum alloy billets with high strength and acceptable fracture toughness.

[0008] In a first aspect of the invention there is provided a method for producing high strength aluminum alloy consolidated billets containing $L1_2$ dispersoids, comprising the steps of: forming an aluminum alloy powder containing $L1_2$ dispersoid forming elements, wherein the $L1_2$ dispersoid forming elements form $A1_3X$ 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; placing the powder in a container; vacuum degassing the container at about 500°F (260°C) to about 900°F (482°C) for about 12 hours to about 8 days; sealing the container; creating a preform by compressing the container by closed die forging or by quasi-isostatic forging to consolidate the powder; encompassing the aluminum alloy powder preform with a flowable pressure transmitting medium and heating the encompassed alloy powder; and uniaxially compressing the medium to thereby consolidate the aluminum powder; and removing the consolidated powder billet.

[0009] In embodiments, the alloys have coherent $L1_2$ $A1_3X$ 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.

[0010] The alloys are formed by encompassing a powder preform of an aluminum alloy body containing $L1_2$ dispersoid forming elements in a heated, flowable pressure transmitting medium, and rapidly compressing the powder to consolidate the perform to form the billet. Use of graphite or a ceramic as the pressure transfer medium causes a non-isostatic pressure field to form in the chamber. During consolidation, the powder preform undergoes an axial compression that exceeds radial expansion. The resulting shear strain cleans the surface oxide from the particles and increases metal-to-metal contact improving forging density.

[0011] In a second aspect of the invention there is provided a high strength aluminum alloy consolidated billet containing $L1_2$ dispersoids in an aluminum alloy matrix wherein: the $L1_2$ dispersoid forming elements form $A1_3X$ 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 gadolin-

ium, 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, wherein: the aluminum alloy consolidated billet containing $L1_2$ dispersoids is formed by the steps comprising: placing the powder in a container; vacuum degassing the container at about 500°F (260°C) to about 900°F (482°C) for about 12 hours to about 8 days; sealing the container; creating a preform by compressing the container by closed die forging or by quasi-isostatic forging to consolidate the powder; encompassing the aluminum alloy powder preform with a flowable pressure transmitting medium and heating the encompassed alloy powder; and uniaxially compressing the medium to thereby consolidate the aluminum powder; and removing the consolidated powder billet.

[0012] This method is known in the industry as Ceracon forging. It has not been attempted using aluminum alloy containing $L1_2$ dispersoids. Examples of the Ceracon forging process are shown in U.S. Patent No. 4,667,497, U.S. Patent Application Publication No. 2005/0147520 and U.S. Patent No.7,097,807 and are included in their entirety by reference.

[0013] The compression takes place in a closed container and results in billets of aluminum alloy containing $L1_2$ dispersoids with a density of essentially 100%.

[0014] Certain preferred embodiments of the invention will now be described by way of example only with reference to the accompanying 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. 6 is a schematic diagram of a vertical gas atomizer.
FIG. 7 is a scanning electron micrograph of the gas atomized inventive $L1_2$ aluminum alloy powder.
FIG. 8 is a diagram showing the processing steps to consolidate $L1_2$ aluminum alloy powder.
FIG. 9 is a schematic diagram illustrating non-isostatic forging.
FIG. 10 is a diagram showing the processing steps to Ceracon forge an $L1_2$ aluminum alloy powder preform.

1. $L1_2$ Aluminum Alloys

[0015] The alloy products 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 $L1_2$ 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.

[0016] 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/

[0017] 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.

[0018] 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 can be complete solubility of lithium in the rapid solidified inventive alloys discussed herein.

[0019] The binary aluminum copper system is a simple eutectic at 32 weight percent copper and 1018°F (548°C). There can be complete solubility of copper in the rapidly solidified inventive alloys discussed herein.

[0020] 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 I 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.

[0021] 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 $L1_2$ intermetallic $A1_3Ni$.

[0022] 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 $A1_3X$ intermetallic dispersoids where X is at least one of scandium, erbium, thulium, ytterbium, and lutetium, that have an $L1_2$ 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.

[0023] Scandium forms Al_3Sc dispersoids that are fine and coherent with the aluminum matrix. Lattice parameters of aluminum and Al_3Sc 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 Al_3Sc dispersoids. This low interfacial energy makes the Al_3Sc 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_3Sc to coarsening. Additions of zinc, copper, lithium, silicon, and nickel provide solid solution and precipitation strengthening in the aluminum alloys. In the alloys of this invention these Al_3Sc 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_3Sc in solution.

[0024] Erbium forms $A1_3Er$ dispersoids in the aluminum matrix that are fine and coherent with the aluminum matrix. The lattice parameters of aluminum and $A1_3Er$ are close (0.405 nm and 0.417 nm respectively), indicating there is minimal driving force for causing growth of the $A1_3Er$ dispersoids. This low interfacial energy makes the $A1_3Er$ 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 $A1_3Er$ to coarsening. Additions of zinc, copper, lithium, silicon, and nickel provide solid solution and precipitation strengthening in the aluminum alloys. In the alloys of this invention, these Al_3Er 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_3Er in solution.

[0025] Thulium forms metastable Al_3Tm dispersoids in the aluminum matrix that are fine and coherent with the aluminum matrix. The lattice parameters of aluminum and Al_3Tm are close (0.405 nm and 0.420 nm respectively), indicating there is minimal driving force for causing growth of the Al_3Tm dispersoids. This low interfacial energy makes the Al_3Tm 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_3Tm to coarsening. Additions of zinc, copper, lithium, silicon, and nickel provide solid solution and precipitation strengthening in the aluminum alloys. In the alloys of this invention these Al_3Tm 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_3Tm in solution.

[0026] Ytterbium forms Al_3Yb dispersoids in the aluminum matrix that are fine and coherent with the aluminum matrix. The lattice parameters of Al and $A1_3Yb$ are close (0.405 nm and 0.420 nm respectively), indicating there is minimal driving force for causing growth of the $A1_3Yb$ dispersoids. This low interfacial energy makes the $A1_3Yb$ 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_3Yb to coarsening. Additions of zinc, copper, lithium, silicon, and nickel provide solid solution and precipitation strengthening in the aluminum alloys. In the alloys of this invention, these Al_3Yb 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_3Yb in solution.

[0027] Lutetium forms Al_3Lu dispersoids in the aluminum matrix that are fine and coherent with the aluminum matrix. The lattice parameters of Al and Al_3Lu are close (0.405 nm and 0.419 nm respectively), indicating there is minimal driving force for causing growth of the Al_3Lu dispersoids. This low interfacial energy makes the Al_3Lu 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_3Lu to coarsening. Additions of zinc, copper, lithium, silicon, and nickel provide solid solution and precipitation strengthening in the aluminum alloys. In the alloys of this invention, these Al_3Lu 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 Al_3Lu in solution.

[0028] Gadolinium forms metastable Al_3Gd 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 Al_3Gd dispersoids have a $D0_{19}$ structure in the equilibrium condition. Despite its large atomic size, gadolinium has fairly high solubility in the Al_3X intermetallic dispersoids (where X is.scandium, erbium, thulium, ytterbium or lutetium). Gadolinium can substitute for the X atoms in Al_3X intermetallic, thereby forming an ordered $L1_2$ phase which results in improved thermal and structural stability.

[0029] Yttrium forms metastable Al_3Y dispersoids in the aluminum matrix that have an $L1_2$ structure in the metastable condition and a $D0_{19}$ structure in the equilibrium condition. The metastable Al_3Y dispersoids have a low diffusion coefficient which makes them thermally stable and highly resistant to coarsening. Yttrium has a high solubility in the Al_3X intermetallic dispersoids allowing large amounts of yttrium to substitute for X in the Al_3X $L1_2$ dispersoids which results in improved thermal and structural stability.

[0030] Zirconium forms Al_3Zr dispersoids in the aluminum matrix that have an $L1_2$ structure in the metastable condition and $D0_{23}$ structure in the equilibrium condition. The metastable Al_3Zr dispersoids have a low diffusion coefficient which makes them thermally stable and highly resistant to coarsening. Zirconium has a high solubility in the Al_3X dispersoids allowing large amounts of zirconium to substitute for X in the Al_3X dispersoids, which results in improved thermal and structural stability.

[0031] Titanium forms Al_3Ti 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_3Ti despersoids have a low diffusion coefficient which makes them thermally stable and highly resistant to coarsening. Titanium has a high solubility in the Al_3X dispersoids allowing large amounts of titanium to substitute for X in the Al_3X dispersoids, which result in improved thermal and structural stability.

[0032] Hafnium forms metastable Al_3 Hf dispersoids in the aluminum matrix that have an $L1_2$ structure in the metastable condition and a $D0_{23}$ structure in the equilibrium condition. The Al_3Hf dispersoids have a low diffusion coefficient, which makes them thermally stable and highly resistant to coarsening. Hafnium has a high solubility in the Al_3X dispersoids allowing large amounts of hafnium to substitute for scandium, erbium, thulium, ytterbium, and lutetium in the above mentioned Al_3X dispersoids, which results in stronger and more thermally stable dispersoids.

[0033] Niobium forms metastable Al_3Nb dispersoids in the aluminum matrix that have an $L1_2$ structure in the metastable condition and a $D0_{22}$ structure in the equilibrium condition. Niobium has a lower solubility in the $A1_3X$ dispersoids than hafnium or yttrium, allowing relatively lower amounts of niobium than hafnium or yttrium to substitute for X in the $A1_3X$ dispersoids. Nonetheless, niobium can be very effective in slowing down the coarsening kinetics of the $A1_3X$ dispersoids because the Al_3Nb dispersoids are thermally stable. The substitution of niobium for X in the above mentioned Al_3X dispersoids results in stronger and more thermally stable dispersoids.

[0034] Al_3X $L1_2$ 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.

[0035] $L1_2$ 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.

[0036] Exemplary aluminum alloys for the bimodal system alloys of this invention include, but are not limited to (in weight percent unless otherwise specified):

about Al-M-(0.1-4)Sc-(0.1-20)Gd;
about Al-M-(0.1-20)Er-(0.1-20)Gd;
about Al-M-(0.1-15)Tm-(0.1-20)Gd;
about Al-M-(0.1-25)Yb-(0.1-20)Gd;
about Al-M-(0.1-25)Lu-(0.1-20)Gd;
about Al-M-(0.1-4)Sc-(0.1-20)Y;
about Al-M-(0.1-20)Er-(0.1-20)Y;
about Al-M-(0.1-15)Tm-(0.1-20)Y;
about Al-M-(0.1-25)Yb-(0.1-20)Y;
about Al-M-(0.1-25)Lu-(0.1-20)Y;
about Al-M-(0.1-4)Sc-(0.05-4)Zr;
about Al-M-(0.1-20)Er-(0.05-4)Zr;
about Al-M-(0.1-15)Tm-(0.054)Zr;
about Al-M-(0.1-25)Yb-(0.05-4)Zr;
about Al-M-(0.1-25)Lu-(0.05-4)Zr;
about Al-M-(0.1-4)Sc-(0.05-10)Ti;
about Al-M-(0.1-20)Er-(0.05-10)Ti;
about Al-M-(0.1-15)Tm-(0.05-10)Ti;
about Al-M- (0.1-25)Yb-(0.05-10)Ti;
about Al-M-(0.1-25)Lu-(0.05-10)Ti;
about Al-M-(0.1-4)Sc-(0.05-10)Hf;
about Al-M-(0.1-20)Er-(0.05-10)Hf;
about Al-M-(0.1-15)Tm-(0.05-10)Hf;
about Al-M-(0.1-25)Yb-(0.05-10)Hf;
about Al-M-(0.1-25)Lu-(0.05-10)Hf;
about Al-M-(0.1-4)Sc-(0.05-5)Nb;
about Al-M-(0.1-20)Er-(0.05-5)Nb;
about Al-M-(0.1-15)Tm-(0.05-5)Nb;
about Al-M-(0.1-25)Yb-(0.05-5)Nb; and
about Al-M-(0.1-25)Lu-(0.05-5)Nb.

[0037] M is at least one of about (4-25) weight percent silicon, (1-8) weight percent magnesium, (0.5-3) weight percent lithium, (0.2-6.5) weight percent copper, (3-12) weight percent zinc, and (1-12) weight percent nickel.

[0038] The amount of silicon present in the fine grain

matrix of this invention 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.

[0039] The amount of magnesium present in the fine grain matrix of this invention 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.

[0040] The amount of lithium present in the fine grain matrix of this invention 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.

[0041] The amount of copper present in the fine grain matrix of this invention if any may vary from about 0.2 to about 6.5 weight percent, more preferably from about 0.5 to about 5.0 weight percent, and even more preferably from about 2 to about 4.5 weight percent.

[0042] The amount of zinc present in the fine grain matrix of this invention 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.

[0043] The amount of nickel present in the fine grain matrix of this invention 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.

[0044] The amount of scandium present in the fine grain matrix of this invention 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 Al_3Sc 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 $L1_2$ intermetallic Al_3Sc 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 10^3°C/second.

[0045] The amount of erbium present in the fine grain matrix of this invention, 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 $L1_2$ intermetallic Al_3Er 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 10^3°C/second.

[0046] The amount of thulium present in the alloys of this invention, 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 Al_3Tm dispersoids in the aluminum matrix that have an $L1_2$ structure in the equilibrium condition. The Al_3Tm 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 $L1_2$ intermetallic Al_3Tm 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 10^3°C/second.

[0047] The amount of ytterbium present in the alloys of this invention, 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 $L1_2$ intermetallic $A1_3Yb$ 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 10^3°C/second.

[0048] The amount of lutetium present in the alloys of this invention, 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 $L1_2$ intermetallic Al_3Lu 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 10^3°C/second.

[0049] The amount of gadolinium present in the alloys of this invention, 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.

[0050] The amount of yttrium present in the alloys of

this invention, 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.

[0051] The amount of zirconium present in the alloys of this invention, 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.

[0052] The amount of titanium present in the alloys of this invention, 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.

[0053] The amount of hafnium present in the alloys of this invention, 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.

[0054] The amount of niobium present in the alloys of this invention, 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.

[0055] In order to have the best properties for the fine grain matrix of this invention, 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. Ceracon Forging of $L1_2$ Aluminum Alloys

[0056] It is advantageous to form $L1_2$ strengthened aluminum alloy product from powder. The major reason is that the rapid cooling rate experienced during powder formation from the melt results in high supersaturation of intermetallic $L1_2$ phase forming elements in the powder. The high supersaturation leads to a maximum amount of the strengthening phase dispersed throughout the structure in the final consolidated part.

[0057] The highest cooling rates observed in commercially viable processes are achieved by gas atomization of molten metals to produce powder. Gas atomization is a two fluid process wherein a stream of molten metal is disintegrated by a high velocity gas stream. The end result is that the particles of molten metal eventually become spherical due to surface tension and finely solidify in powder form. Heat from the liquid droplets is transferred to the atomization gas by convection. The solidification rates, depending on the gas and the surrounding environment, can be very high and can exceed 10^{6}°C/second. Cooling rates greater than 10^{3}°C/second are typically specified to ensure supersaturation of alloying elements in gas atomized $L1_2$ aluminum alloy powder in the inventive process described herein.

[0058] A schematic of typical vertical gas atomizer 100 is shown in FIG. 6A. FIG. 6A is taken from R. Germain, Powder Metallurgy Science Second Edition MPIF (1994) (chapter 3, p. 101) and is incorporated herein by reference. Vacuum or inert gas induction melter 102 is positioned at the top of free flight chamber 104. Vacuum induction melter 102 contains melt 106 which flows by gravity or gas overpressure through nozzle 108. A close up view of nozzle 108 is shown in FIG. 6B. Melt 106 enters nozzle 108 and flows downward till it meets high pressure gas stream from gas source 110 where it is transformed into a spray of droplets. The droplets eventually become spherical due to surface tension and rapidly solidify into spherical powder 112 which collects in collection chamber 114. The gas recirculates through cyclone collector 116 which collects fine powder 118 before returning to the input gas stream. As can be seen from FIG. 6A, the surroundings to which the melt and eventual powder are exposed are completely controlled.

[0059] There are many effective nozzle designs known in the art to produce spherical metal powder. Nozzle designs with short gas-to-melt separation distances produce finer powders. Confined nozzle designs where gas meets the molten stream at a short distance just after it leaves the atomization nozzle are preferred for the production of the inventive $L1_2$ aluminum alloy powders disclosed herein. Higher superheat temperatures cause lower melt viscosity and longer cooling times. Both result in smaller spherical particles.

[0060] A large number of processing parameters are associated with gas atomization that affect the final product. Examples include melt superheat, gas pressure, metal flow rate, gas type, and gas purity. In gas atomization, the particle size is related to the energy input to the metal. Higher gas pressures, higher superheat temperatures and lower metal flow rates result in smaller particle sizes. Higher gas pressures provide higher gas velocities for a given atomization nozzle design.

[0061] To maintain purity, inert gases are used, such as helium, argon, and nitrogen. Helium is preferred for rapid solidification because the high heat transfer coefficient of the gas leads to high quenching rates and high supersaturation of alloying elements.

[0062] Lower metal flow rates and higher gas flow rates favor production of finer powders. The particle size of gas atomized melts typically has a log normal distribution. An example of spherical $L1_2$ aluminum alloy powder is shown in the scanning electron micrograph (SEM) of FIG. 7.

[0063] Oxygen and hydrogen in the powder can degrade the mechanical properties of the final part. It is preferred to limit the oxygen in the $L1_2$ alloy powder to about 1 ppm to 2000 ppm. Oxygen is intentionally introduced as a component of the helium gas during atomization. A thin oxide coating on the $L1_2$ aluminum powder is beneficial for two reasons. First, the coating prevents

agglomeration by contact sintering and secondly, the coating inhibits the chance of explosion of the powder. A controlled amount of oxygen is important in order to provide good ductility and fracture toughness in the final consolidated material. Hydrogen content in the powder is controlled by ensuring the dew point of the helium gas is low. A dew point of about minus 50°F (minus 45.5°C) to minus 100°F (minus 73.3°C) is preferred.

[0064] In preparation for final processing, the powder is classified according to size by sieving. To prepare the powder for sieving, if the powder has zero percent oxygen content, the powder may be exposed to nitrogen gas which passivates the powder surface and prevents agglomeration. Finer powder sizes result in improved mechanical properties of the end product. While minus 325 mesh (about 45 microns) powder can be used, minus. 450 mesh (about 30 microns) powder is a preferred size in order to provide good mechanical properties in the end product. During the atomization process, powder is collected in collection chambers in order to prevent oxidation of the powder. Collection chambers are used at the bottom of atomization chamber 104 as well as at the bottom of cyclone collector 116. The powder is transported and stored in the collection chambers also. Collection chambers are maintained under positive pressure with nitrogen gas which prevents oxidation of the powder.

[0065] The process of consolidating the inventive alloy powders into useful forms is schematically illustrated in FIG. 8. $L1_2$ aluminum alloy powders 210 are first classified according to size by sieving (step 220). Fine particle sizes are required for optimum mechanical properties in the final part. Next, the classified powders are blended (step 230) in order to maintain microstructural homogeneity in the final part. Blending is necessary because different atomization batches produce powders with varying particle size distributions. Other benefits of blending will be discussed later. Powders may be optionally cryomilled (step 240) to minimize grain size and improve strength. Cryomilling is carried out in a high-energy ball mill under liquid nitrogen, and offers several benefits that will be discussed later.

[0066] The sieved, blended and (optionally) cryomilled powders are then put in a can (step 250) and vacuum degassed (step 260). Following vacuum degassing, the can is sealed (step 270) under vacuum and forged (step 280) to produce a densified preform. Finally, the preform is Ceracon forged (step 290) to produce a product with improved mechanical properties useful for subsequent service as a high temperature $L1_2$ strengthened aluminum alloy. Non-isostatic Ceracon forging will be described later.

[0067] Sieving (step 220) is a critical step in consolidation because the final mechanical properties relate directly to the particle size. Finer particle size results in finer $L1_2$ particle dispersion and finer grain size. Optimum mechanical properties have been observed with -450 mesh (30 micron) powder. Sieving (step 220) also limits the defect size in the powder. Before sieving, the powder is passivated with nitrogen gas in order to prevent agglomeration. Ultrasonic sieving is preferred for its efficiency.

[0068] Blending (step 230) is another critical 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 230) is also necessary when separate metal and/or ceramic powders are added to the $L1_2$ base powder to form bimodal and trimodal consolidated alloy microstructures.

[0069] Cryomilling (step 240) can be used to refine the grain size of gas atomized $L1_2$ aluminum alloy powder as well as the final consolidated alloy microstructure. Cryomilling is described in U.S. Patent No. 6,902,699, Fritzemeier et al. and in U.S. Patent No. 7,344,675, Van Daam et al. and are incorporated herein in their entirety by reference. Cryomilling involves high-energy ball milling under liquid nitrogen. The liquid nitrogen environment prevents oxidation and prevents frictional heating of the powder and the resulting grain coarsening. During the process, the powder particles are repeatedly sheared, fractured and cold welded which results in a severely deformed microstructure containing a high dislocation density that, with continued deformation, evolves into a cellular structure consisting of extremely small dislocation free grains separated by high angle grain boundaries with high dislocation density. The grain size of the cellular microstructure is typically less than 100 nm and the microstructure is considered a nanostructure.

[0070] In addition, the nitrogen environment results in the formation of nitride particles that reside at the grain boundaries and in the grain interiors and resist coarsening at higher temperatures. Stearic acid is preferably added to the powder charge to prevent excessive agglomeration and to promote fracturing and rewelding of the $L1_2$ aluminum alloy particles during milling.

[0071] Following sieving (step 220), blending (step 230) and (optionally) cryomilling (Step 240), the powders are transferred to a can (step 250) where the powder is vacuum degassed (step 260) for about 12 hours to over 8 days at elevated temperatures. A temperature range of about 500°F (260°C) to about 900°F (482°C) is preferred and about 750°F (399°C) is more preferred. Dynamic degassing large amounts of powder are preferred to static degassing to expose all of the powder to a uniform temperature. Degassing removes the stearic acid lubricant as well as oxygen and hydrogen from the charge.

[0072] Following vacuum degassing (step 260), the vacuum line is crimped and welded shut. The powder is then consolidated into a dense preform by closed die forging or by quasi-isostatic forging (step 280).

[0073] An exemplary embodiment of this invention is to consolidate a canned $L1_2$ aluminum alloy powder preform into a substantially 100% dense billet by a quasi-

isostatic Ceracon-type forging process. The forging process consists of uniaxially pressing the canned powder preform or solid part perform in a cylindrical press, wherein the preform is surrounded by pressure transmitting medium during the forging. A schematic of quasi-isostatic forging equipment 300 is shown in FIG. 9. The equipment consists of cylindrical die 310 on base 320 with ram 325 inserted in dye cavity 330. Quasi-isostatic forging is carried out at elevated temperatures schematically illustrated by heating coils 340.

[0074] The steps to Ceracon forge an $L1_2$ aluminum alloy powder preform are schematically illustrated in FIG. 10. First die 310 is (optionally) preheated (step 410). The preheat temperature is preferably about 40°F (4.5°C) to about 70°F (21°C) higher than the forging temperature to compensate for cooling that occurs during the loading process. Next, die 310 is partially filled with pressure transmitting medium (PTM) 360 (step 420). To minimize cooling during loading, the PTM can be (optionally) preheated, preferably to the same temperature as the die (step 450). Consolidated powder preform 350 is then inserted in die 310. Powder preform 350 can be (optionally) preheated, preferably to the same temperature as die 310 and PTM 360 (step 440). The remainder of die cavity 330 is then filled with PTM 360 (step 460). PTM 360 can be (optionally) heated (step 450). Ram 325 is then inserted in die 310 (step 470). Pressure 370 is applied to Ram 325 to accomplish quasi-isostatic Ceracon type forging.

[0075] One advantage of this process is that, if the preheating steps are followed, the run time is short. As a result, deleterious microstructural changes, such as grain and particle coarsening are minimized. Run times can be as short as a few minutes if automated loading and charging equipment is used. In addition, ductility and fracture toughness of $L1_2$ based aluminum alloys can be improved significantly due to dynamic bimodal pressure generated during quasi-isostatic forging that breaks apart continuous powder surface oxide that prevent metal-metal bonding and randomly distributes them in the material.

[0076] During quasi-isostatic Ceracon type forging, the billet axially deforms about 30% and radially deforms about 10%. Strain rates from about 0.1 min^{-1} to about 6 min^{-1} at forging temperatures from about 400°F (204°C) to about 900°F (482°C) are preferred. Forging pressures from 50 ksi (345 MPa) to 150 ksi (1034 MPa) are preferred.

[0077] The pressure transmitting medium can consist of graphite or other carbon containing powders or ceramic powders or both. By proper fixturing and preheating the pressure transmitting medium and the work piece, densification during quasi-isostatic forging can be extremely rapid on the order of minutes.

[0078] The unequal actual and radial deformation of the powder is schematically illustrated by the dotted line outline of deformed can 352 in FIG. 9.

[0079] Quasi-isostatic forging of a 60 to 80 percent dense aluminum-7.5 weight percent magnesium powder preform by this technique resulted in about a 30 percent axial compression and about a 10 percent radial expansion as taught by Meeks III et al. U.S. Patent No. 7,097,807 and included herein by reference. The quasi-isostatic forging deformation of $L1_2$ aluminum alloy powder of this embodiment has beneficial effects on the microstructure and resulting properties of the consolidated billet. The nonuniform stress and resulting strain field in the powder during forging results in extensive shear deformation. The shear deformation deforms the powder, strips off the surface oxide from the $L1_2$ alloy particles and redistributes it throughout the consolidated $L1_2$ aluminum alloy powder forging as finely divided dispersoids. As a result, there is increased metal to metal contact during forging and resulting increased mechanical integrity. The redistributed surface oxide particles act as additional strengthening agents by resisting dislocation motion by Orowan strengthening.

[0080] In other embodiments, Ceracon type forging can be followed by hot isostatic pressing (HIP), forging, rolling and other deformation processing techniques.

[0081] To summarize, Ceracon type quasi-isostatic forging can be low cost and efficient. The pressure transmitting medium, as well as the vacuum sealed preform can be preheated prior to introduction of the perform into the forging chamber to minimize runtime. In addition, forging can be accomplished at high strain rates resulting in forging runs lasting less than a few minutes. The short run time results in an economical process that also further inhibits grain and $L1_2$ particle growth in the billet during forging due to the limited time at temperature. The pressure transmitting medium can be reused and loading and unloading the preforms and resulting forgings can be an automated procedure.

[0082] 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 scope of the invention which is defined by the attached claims.

[0083] In at least a preferred embodiment of the invention there is provided a high strength aluminum alloy consolidated billet containing $L1_2$ dispersoids in an aluminum alloy matrix wherein: the $L1_2$ dispersoid forming elements form $A1_3X$ 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, wherein: the aluminum

alloy consolidated billet containing $L1_2$ dispersoids is formed by the steps comprising: placing the powder in a container; vacuum degassing the container at about 500°F (260°C) to about 900°F (482°C) for about 12 hours to about 8 days; sealing the container; creating a preform by compressing the container by closed die forging or by quasi-isostatic forging to consolidate the powder; encompassing the aluminum alloy powder preform with a flowable pressure transmitting medium and heating the encompassed alloy powder and medium; and uniaxially compressing the medium at an axial strain rate of from about 0.1 min^{-1} to about 6 min^{-1} at a pressure from about 50 KSi (345 MPa) to about 150 KSi (1034 MPa) to thereby consolidate the aluminum powder; and removing the consolidated powder in a billet.

Claims

1. A method for producing high strength aluminum alloy consolidated billets containing $L1_2$ dispersoids, comprising the steps of:

forming an aluminum alloy powder containing $L1_2$ dispersoid forming elements, wherein the $L1_2$ dispersoid forming elements form Al_3X 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;
placing the powder in a container;
vacuum degassing the container at about 500°F (260°C) to about 900°F (482°C) for about 12 hours to about 8 days;
sealing the container;
creating a preform by compressing the container by closed die forging or by quasi-isostatic forging to consolidate the powder;
encompassing the aluminum alloy powder preform with a flowable pressure transmitting medium and heating the encompassed alloy powder; and uniaxially compressing the medium to thereby consolidate the aluminum powder; and removing the consolidated powder billet.

2. The method of claim 1, wherein the forging temperature is from about 400°F (204°C) to about 900°F (482°C).

3. The method of claim 1 or 2, wherein the axial strain rate during compressing the medium is from about 0.1 min^{-1} to 6 min^{-1}.

4. The method of claim 1, 2 or 3, wherein compressing the medium is at a pressure from about 50 Ksi (345 MPa) to about 150 Ksi (1034 MPa).

5. The method of any preceding claim, wherein the preform is vacuum sealed in an aluminum jacket.

6. The method of any preceding claim, wherein the pressure transmitting medium comprises graphite or other carbon containing powders or ceramic powders or both.

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

8. The method of claim 7, 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 6.5 weight percent copper, about 3 to about 12 weight percent zinc, and about 1 to about 12 weight percent nickel.

9. A high strength aluminum alloy consolidated billet containing $L1_2$ dispersoids in an aluminum alloy matrix wherein:

the $L1_2$ dispersoid forming elements form $A1_3X$ 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, wherein:

the aluminum alloy consolidated billet containing $L1_2$ dispersoids has been formed by a method as claimed in any of claims 1 to 8.

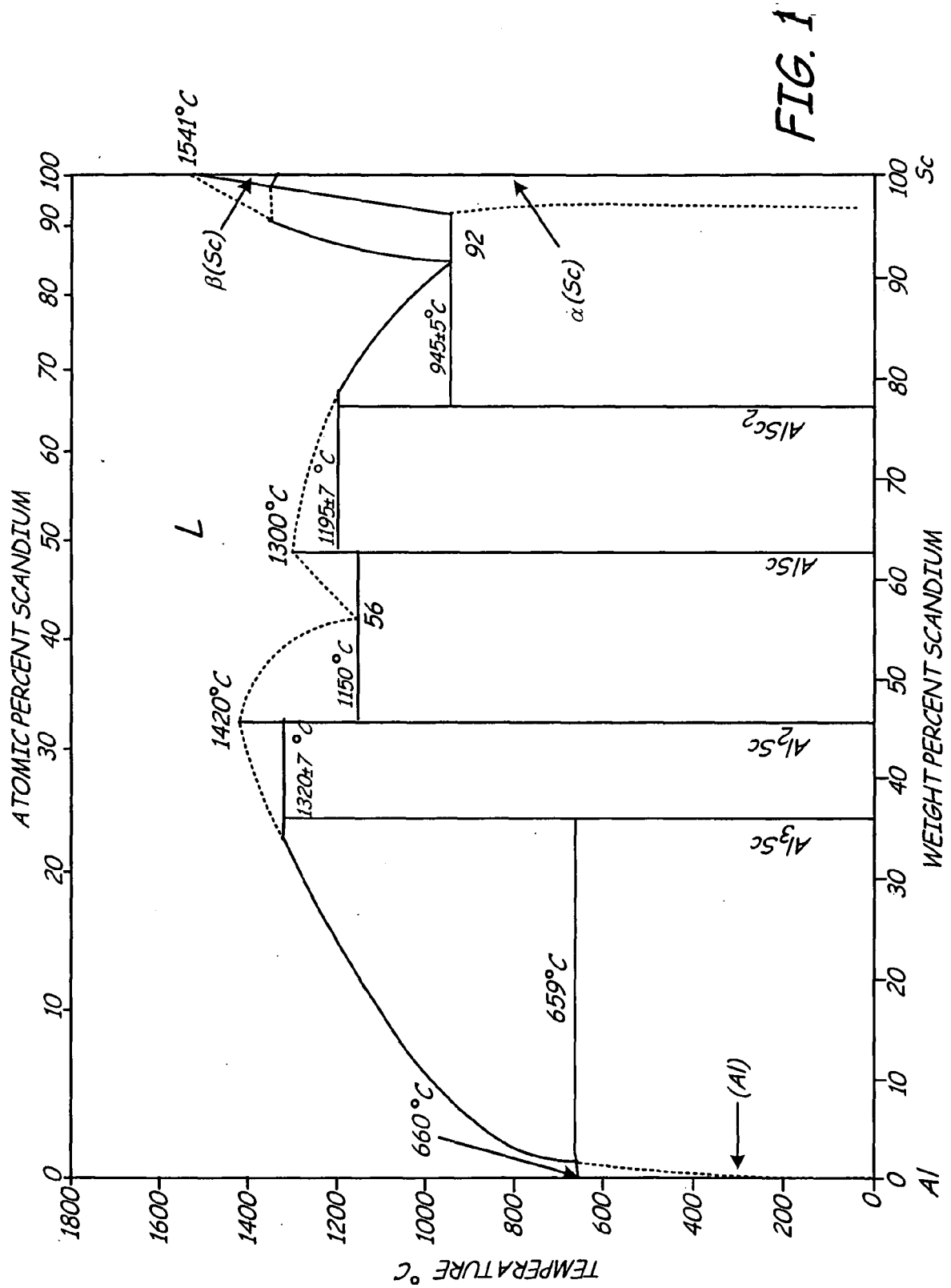

1541°C
β(Sc)
α(Sc)
92
945±5°C
1300°C
1195±7 °C
L
AlSc2
AlSc
56
1150 °C
1420°C
1320±7 °C
Al2Sc
Al3Sc
659°C
660 °C
(Al)
ATOMIC PERCENT SCANDIUM
WEIGHT PERCENT SCANDIUM
TEMPERATURE °C
Al
Sc
0
10
20
30
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1200
1400
1600
1800

FIG. 1

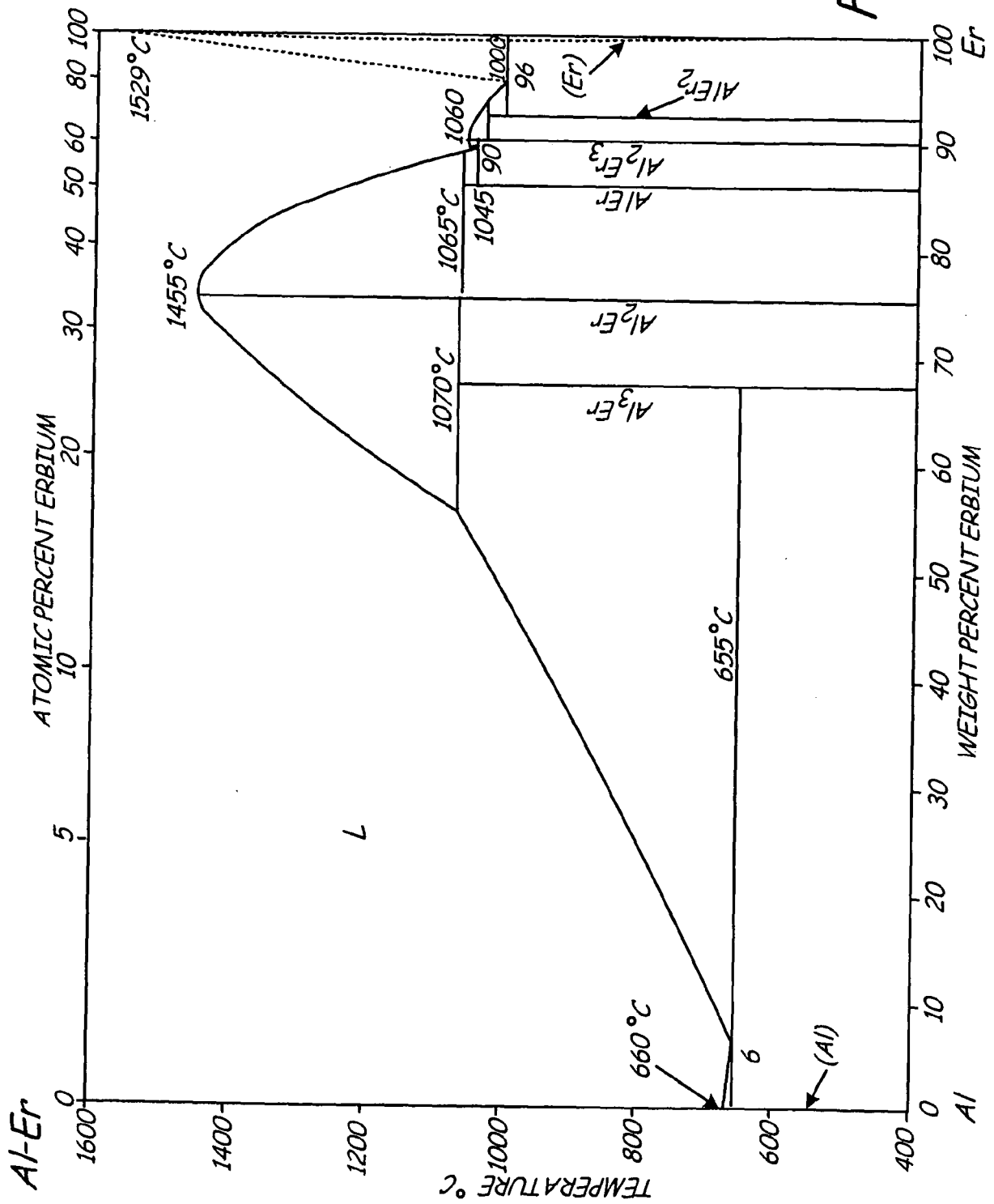

Al-Er
ATOMIC PERCENT ERBIUM
WEIGHT PERCENT ERBIUM
TEMPERATURE °C
1529°C
1455°C
1065°C
1070°C
655°C
660°C
1060
1045
1000
96
90
(Er)
AlEr2
Al2Er3
AlEr
Al2Er
Al3Er
L
δ
(Al)
Al
Er

FIG. 2

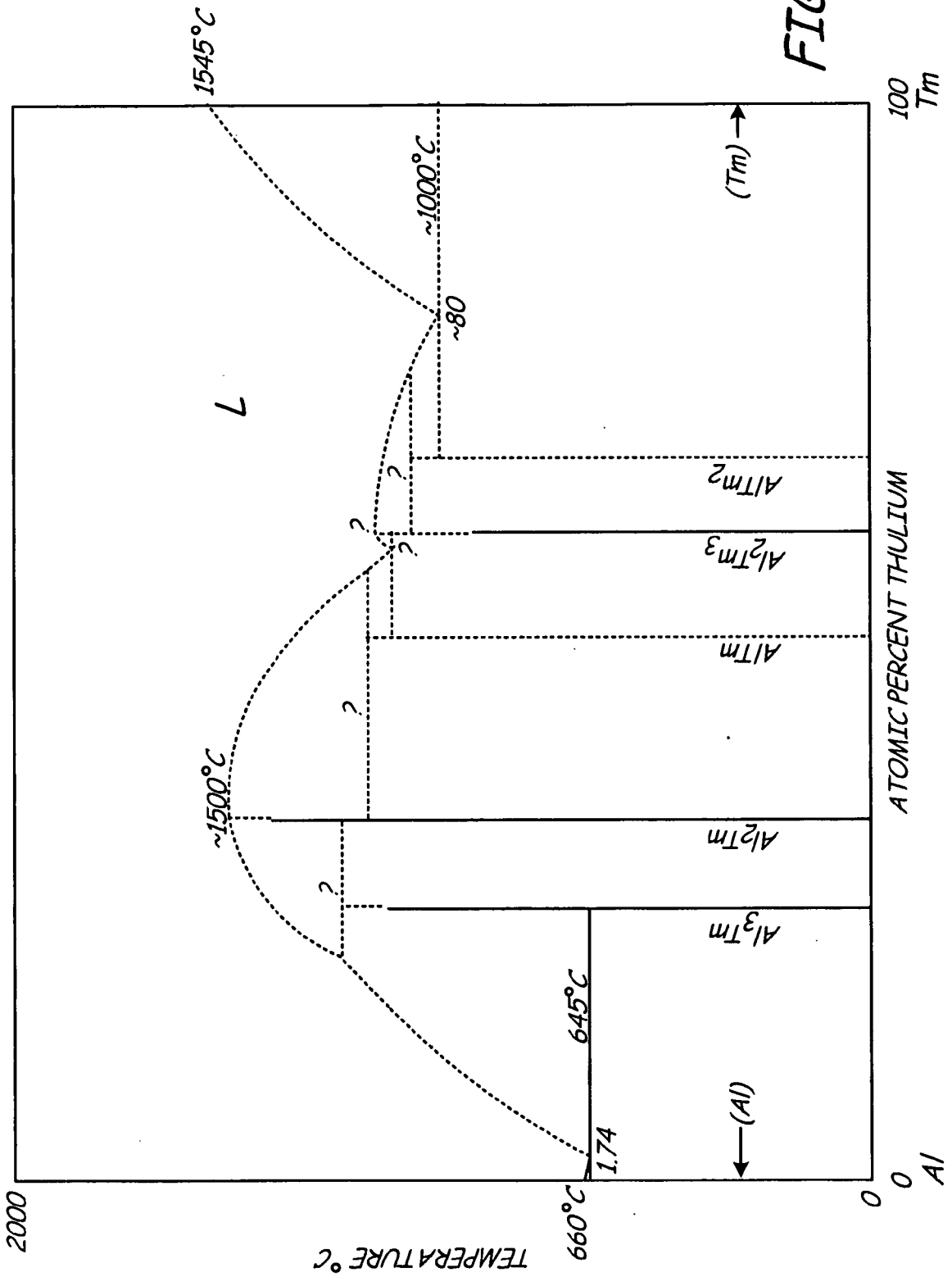

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~1500°C
~1000°C
~80
1545°C
L
Al3Tm
Al2Tm
AlTm
Al2Tm3
AlTm2
?
?
?
?
?
(Al)
(Tm)
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Al
ATOMIC PERCENT THULIUM
100
Tm

FIG. 3

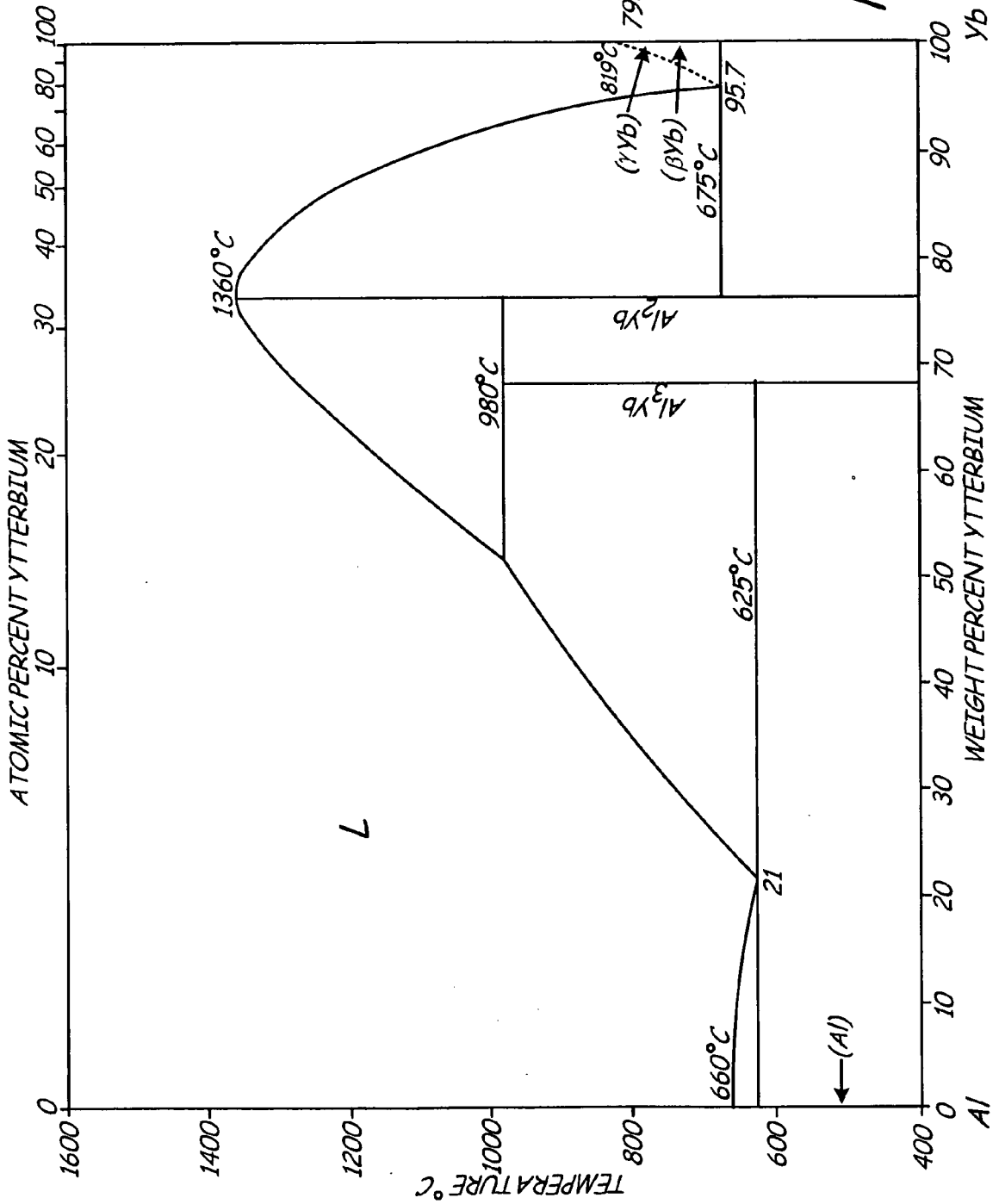

TEMPERATURE °C
ATOMIC PERCENT YTTERBIUM
WEIGHT PERCENT YTTERBIUM
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Yb
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1400
1200
1000
800
600
400
0
10
20
30
40
50
60
70
80
90
100
1360°C
980°C
625°C
660°C
675°C
819°C
795°C
95.7
21
L
(Al)
(γYb)
(βYb)
Al2Yb
Al3Yb

FIG. 4

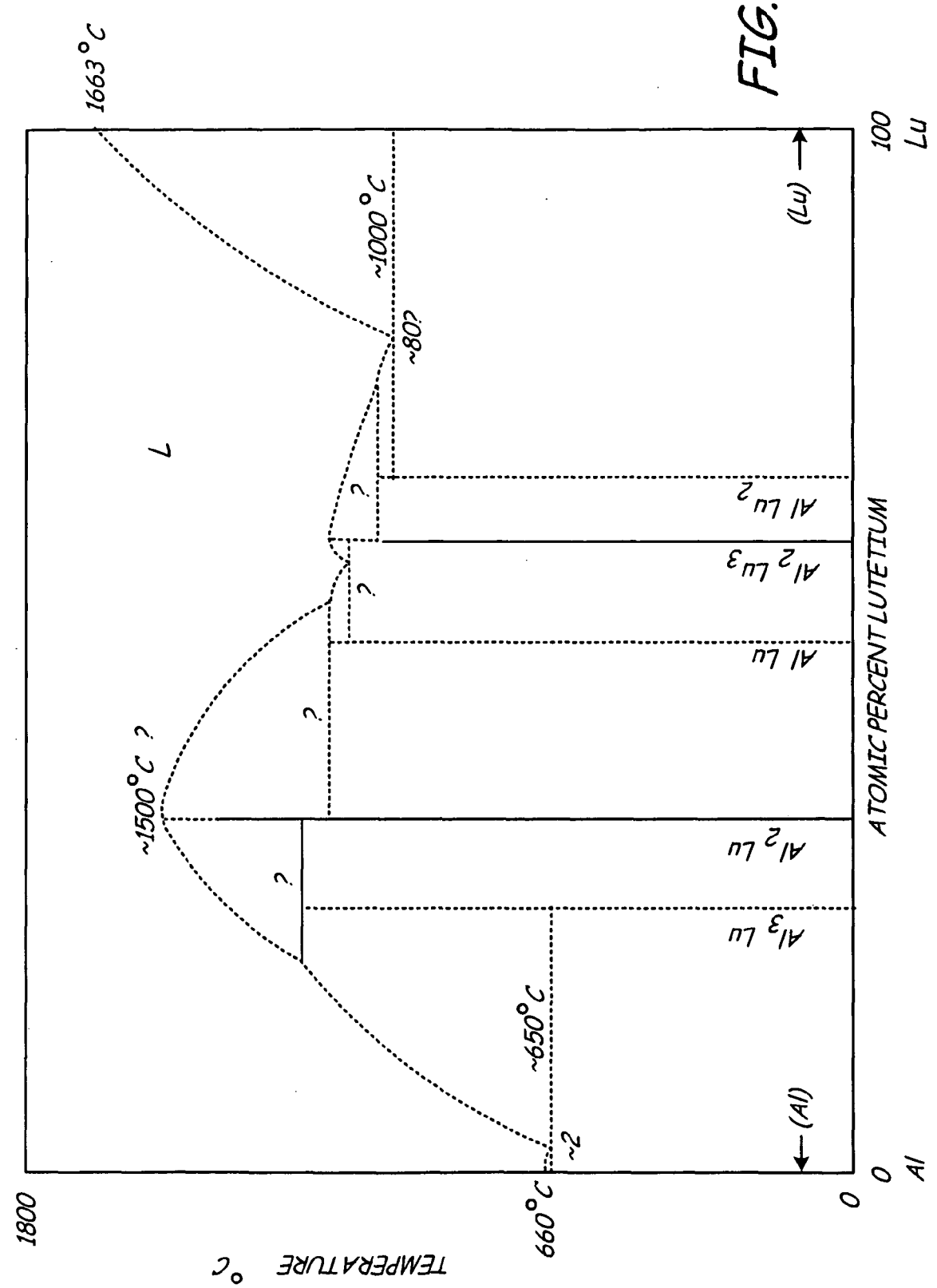

1663 °C
~1000 °C
~80?
L
~1500°C ?
~650°C
~2
660°C
1800
TEMPERATURE °C
0
Al
(Al)
100
Lu
(Lu)
ATOMIC PERCENT LUTETIUM
Al Lu2
Al2 Lu3
Al Lu
Al2 Lu
Al3 Lu
?

FIG. 5

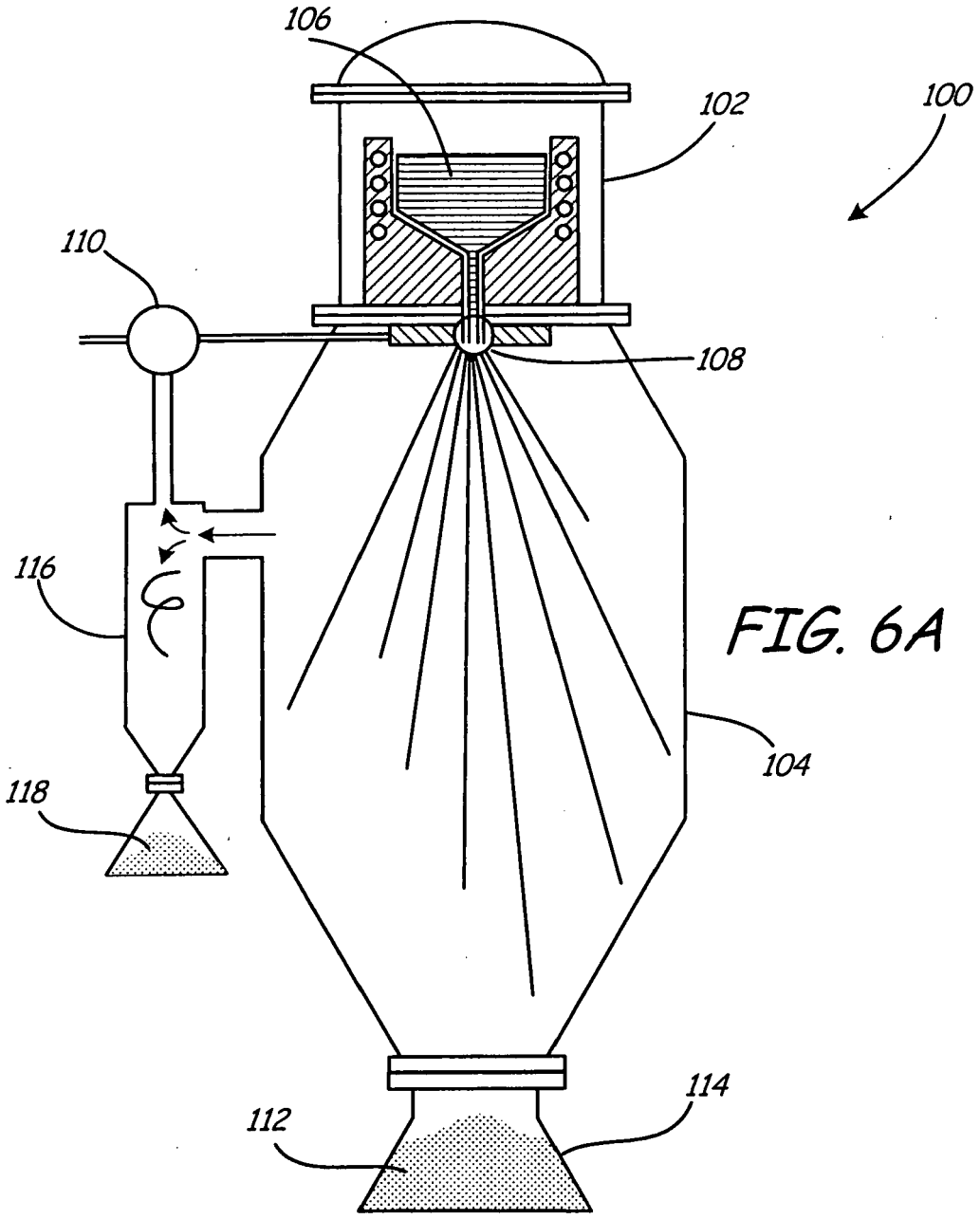


FIG. 6A

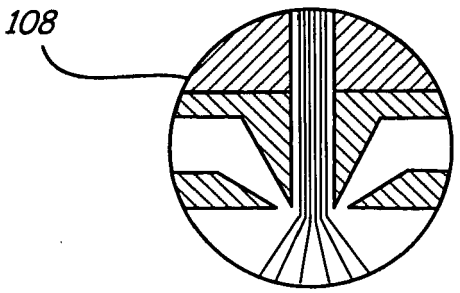


FIG. 6B

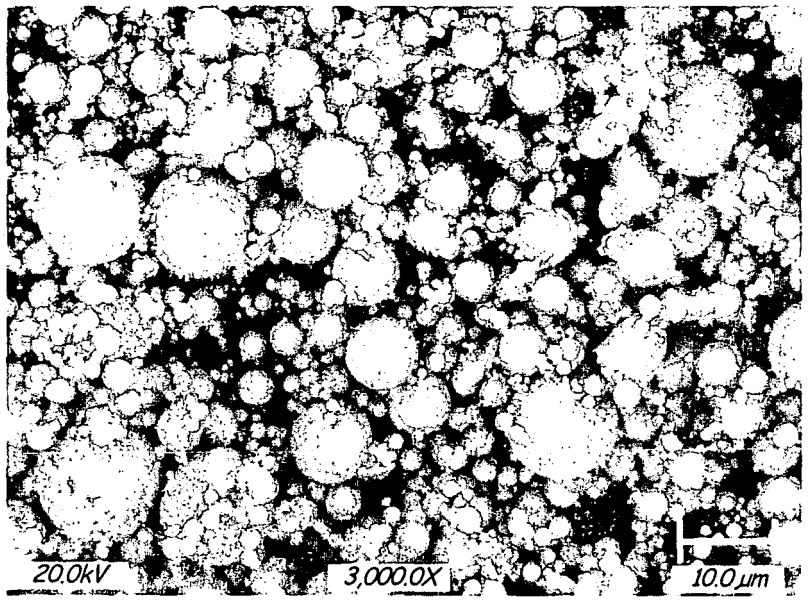


FIG. 7

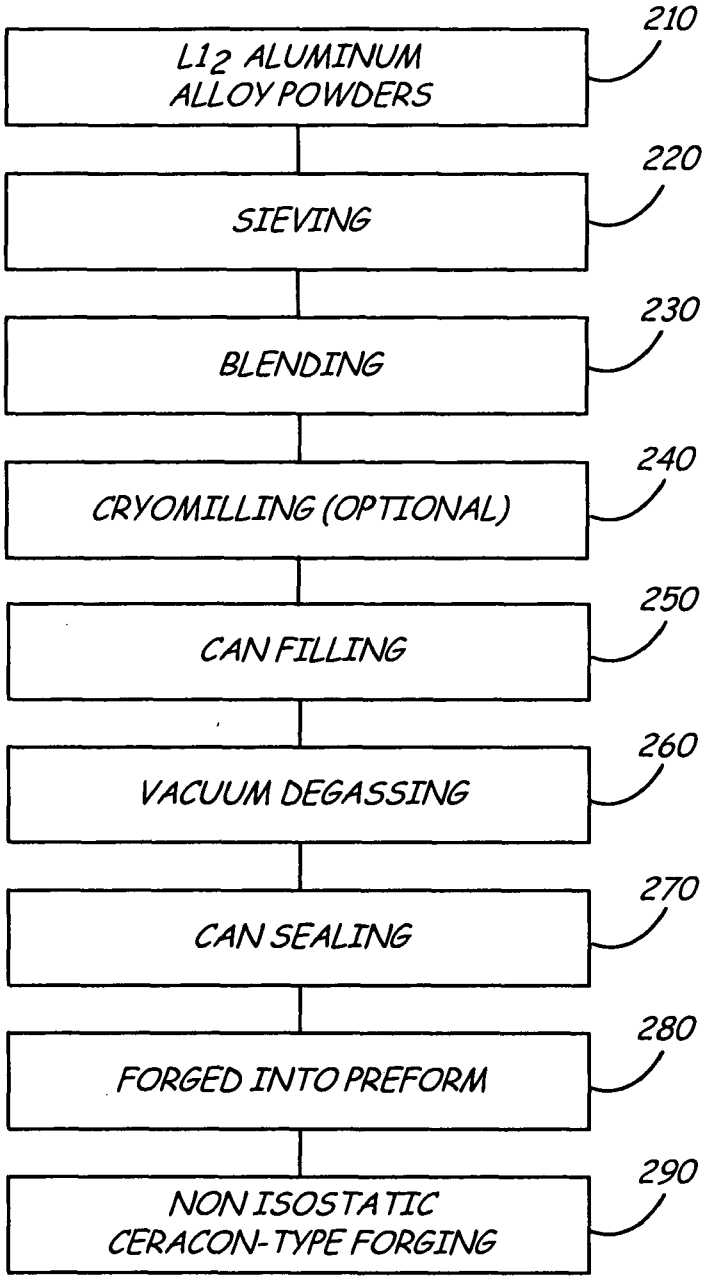

$L1_2$ ALUMINUM ALLOY POWDERS
210
SIEVING
220
BLENDING
230
CRYOMILLING (OPTIONAL)
240
CAN FILLING
250
VACUUM DEGASSING
260
CAN SEALING
270
FORGED INTO PREFORM
280
NON ISOSTATIC CERACON-TYPE FORGING
290

FIG. 8

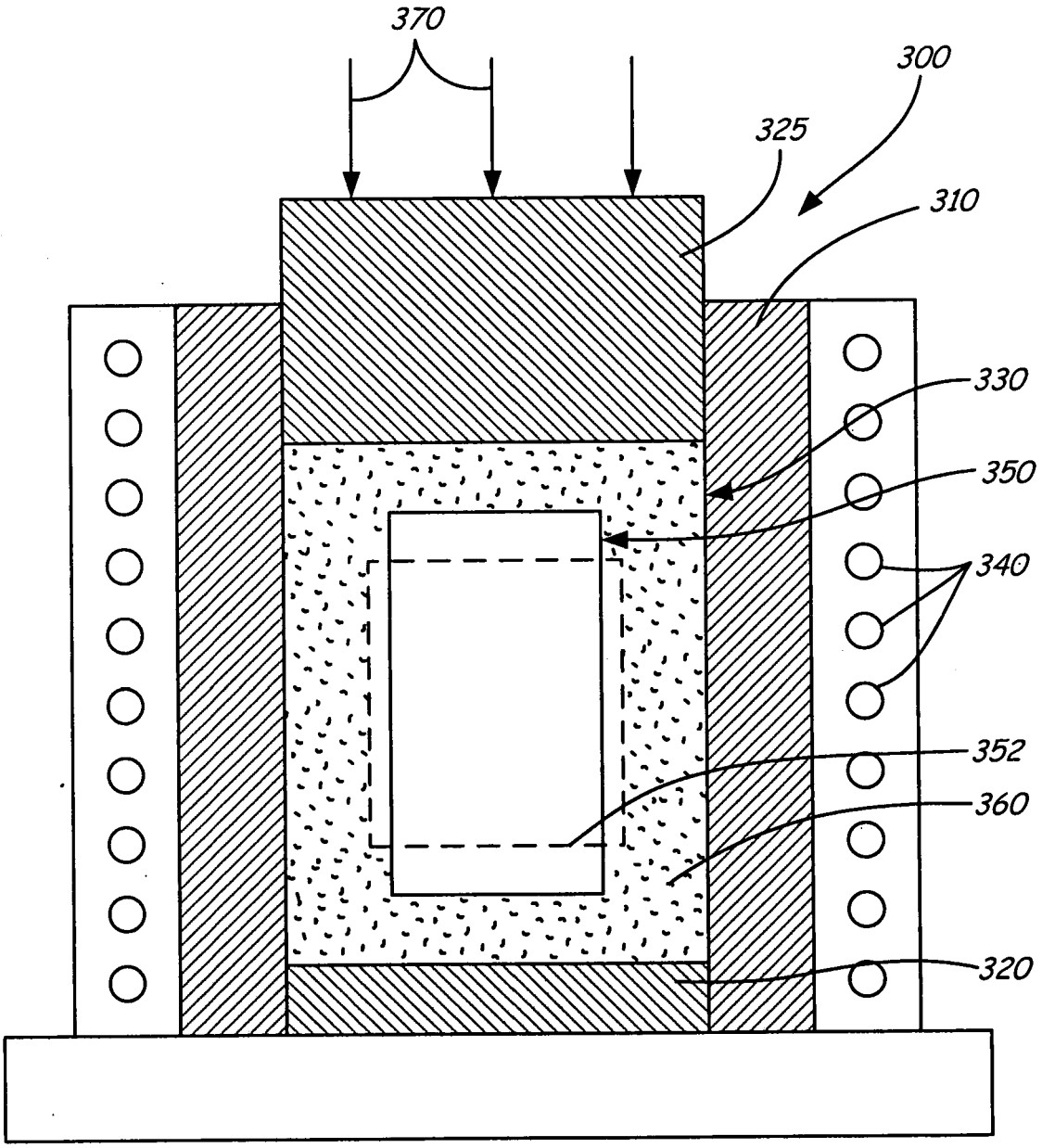


FIG. 9

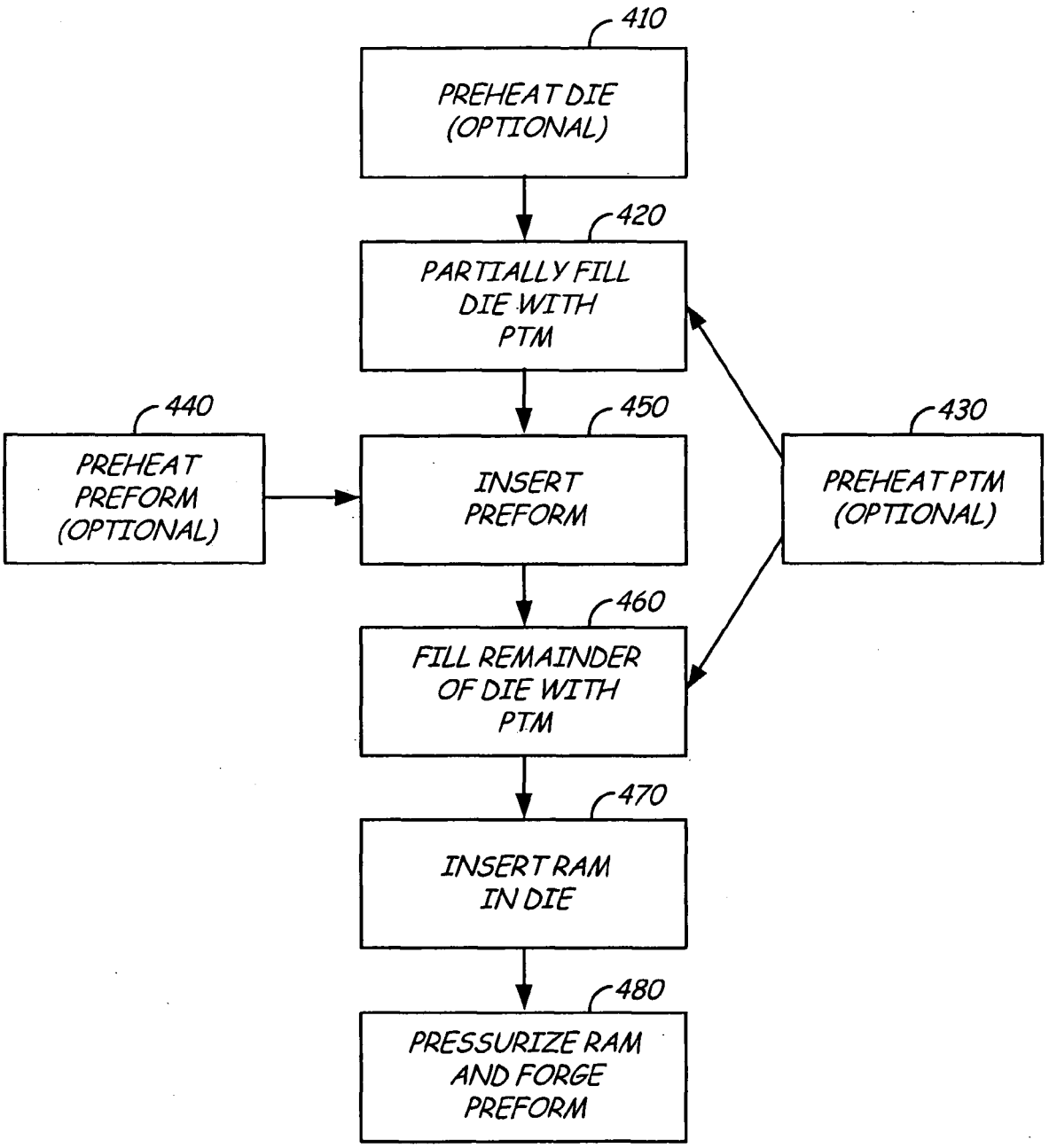

410
PREHEAT DIE (OPTIONAL)
420
PARTIALLY FILL DIE WITH PTM
440
PREHEAT PREFORM (OPTIONAL)
450
INSERT PREFORM
430
PREHEAT PTM (OPTIONAL)
460
FILL REMAINDER OF DIE WITH PTM
470
INSERT RAM IN DIE
480
PRESSURIZE RAM AND FORGE PREFORM

FIG. 10

REFERENCES CITED IN THE DESCRIPTION

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- US 7097807 B **[0012] [0079]**
- US 6902699 B, Fritzemeier **[0069]**
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