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(54) **HIGH STRENGTH L12 ALUMINUM ALLOYS
PRODUCED BY CRYOMILLING**

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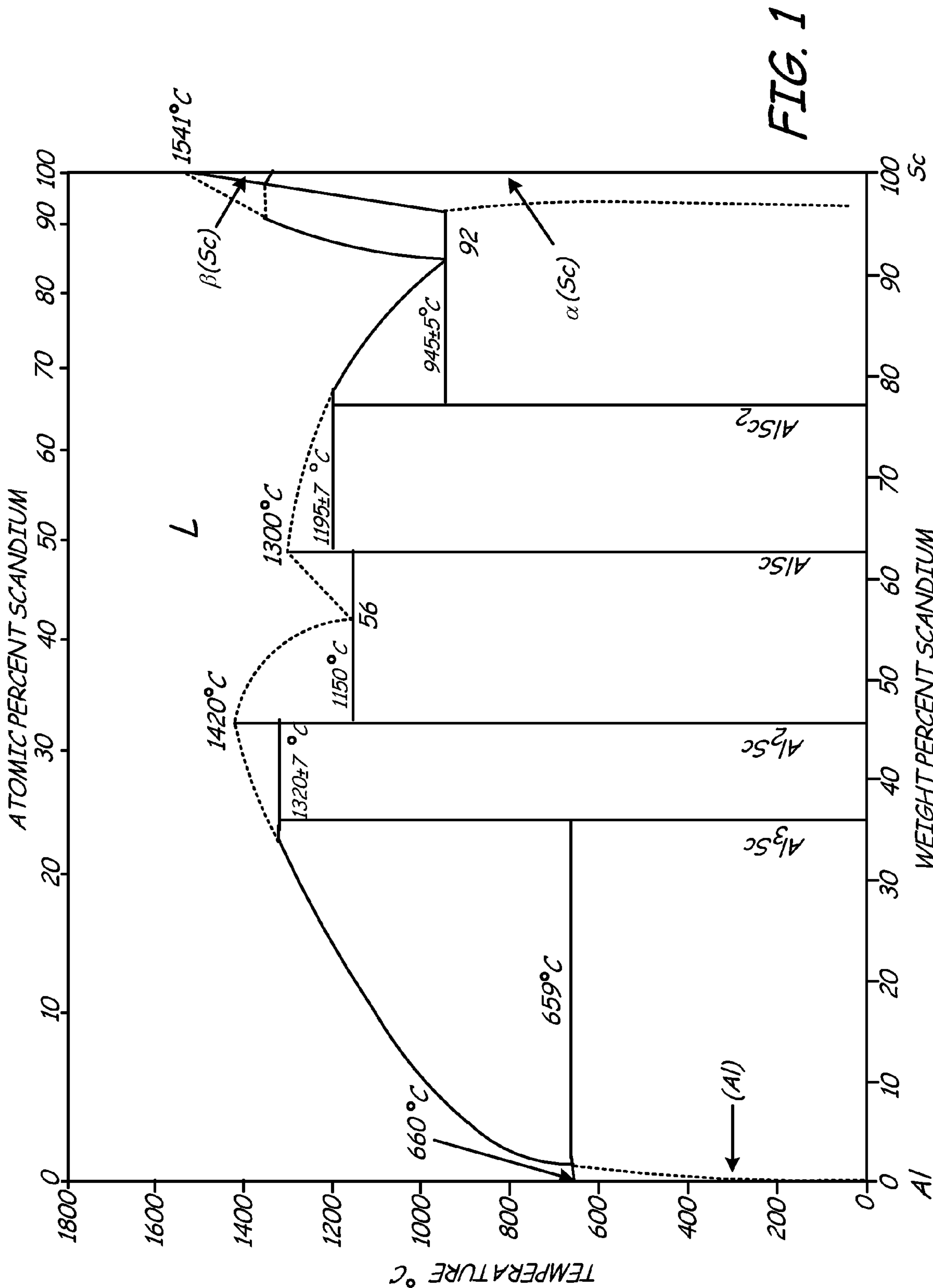
(52) **U.S. Cl. 420/532; 419/23**

(73) Assignee: **UNITED TECHNOLOGIES
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(US)

(57) **ABSTRACT**

(21) Appl. No.: **12/398,712**

A method and apparatus produces high strength aluminum alloys from a cryomilled powder containing L1₂ intermetallic dispersoids. The cryomilled powder is degassed, sealed under vacuum in a container, heated, consolidated by vacuum hot pressing, and extruded.



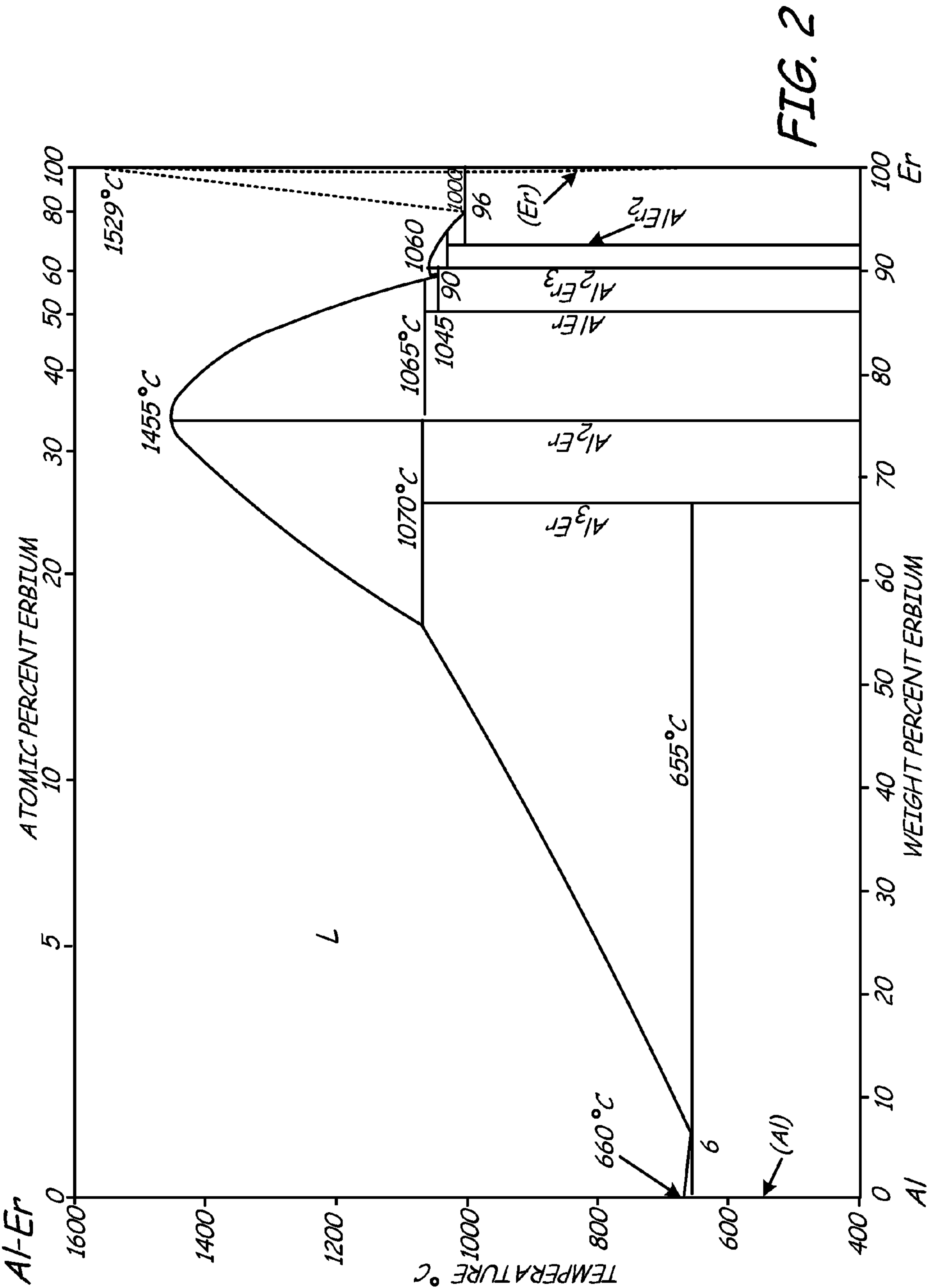
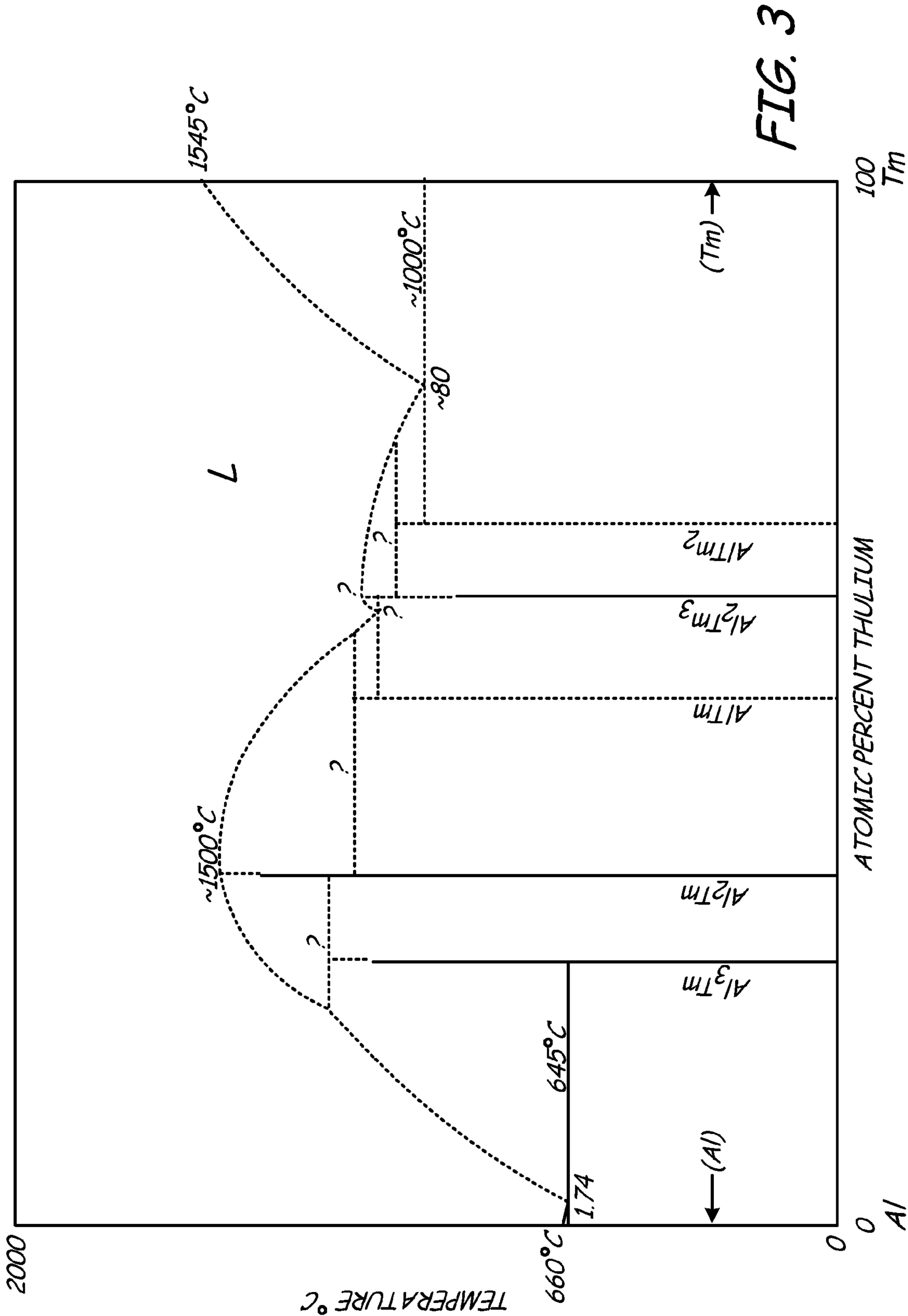
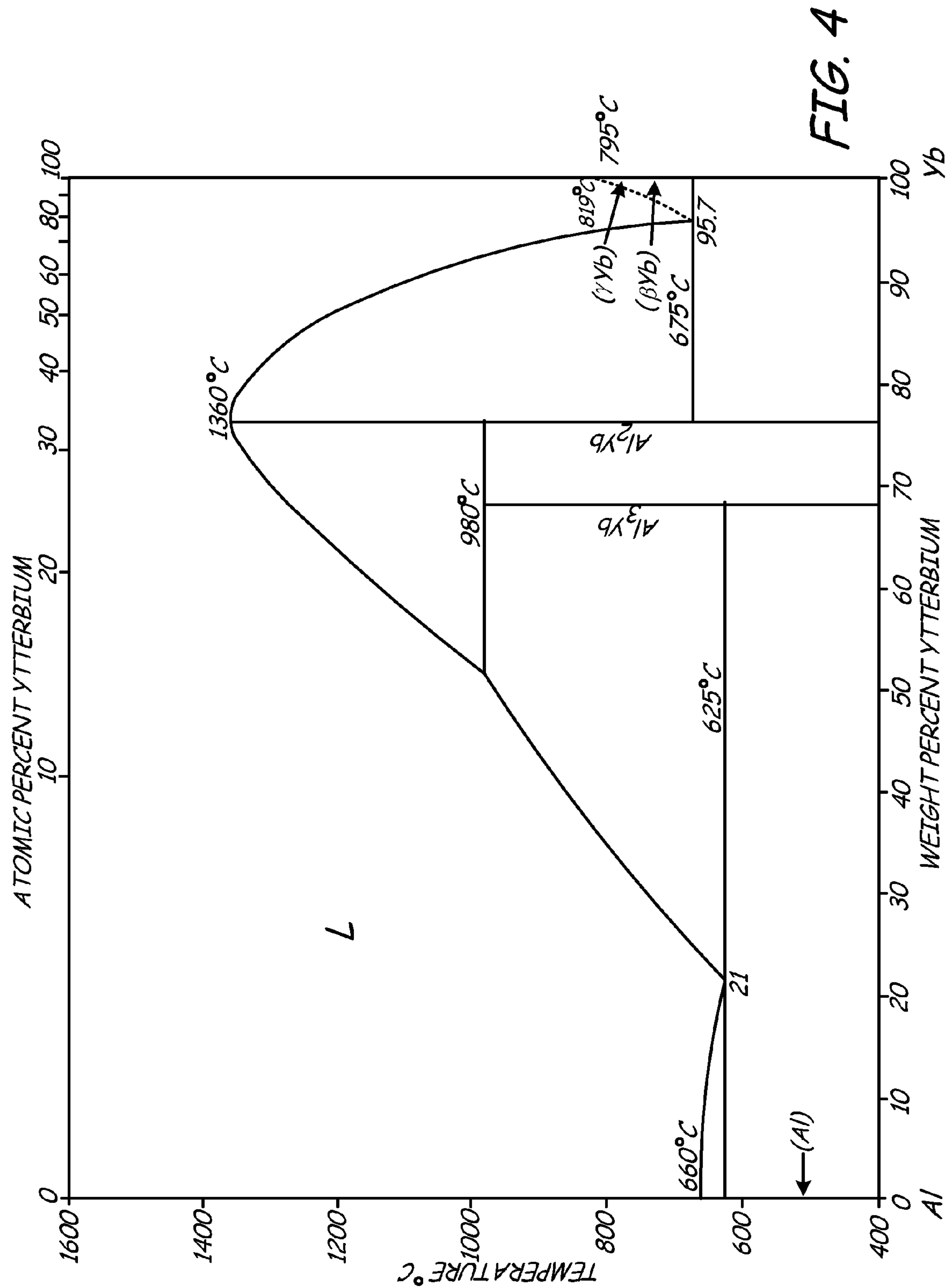
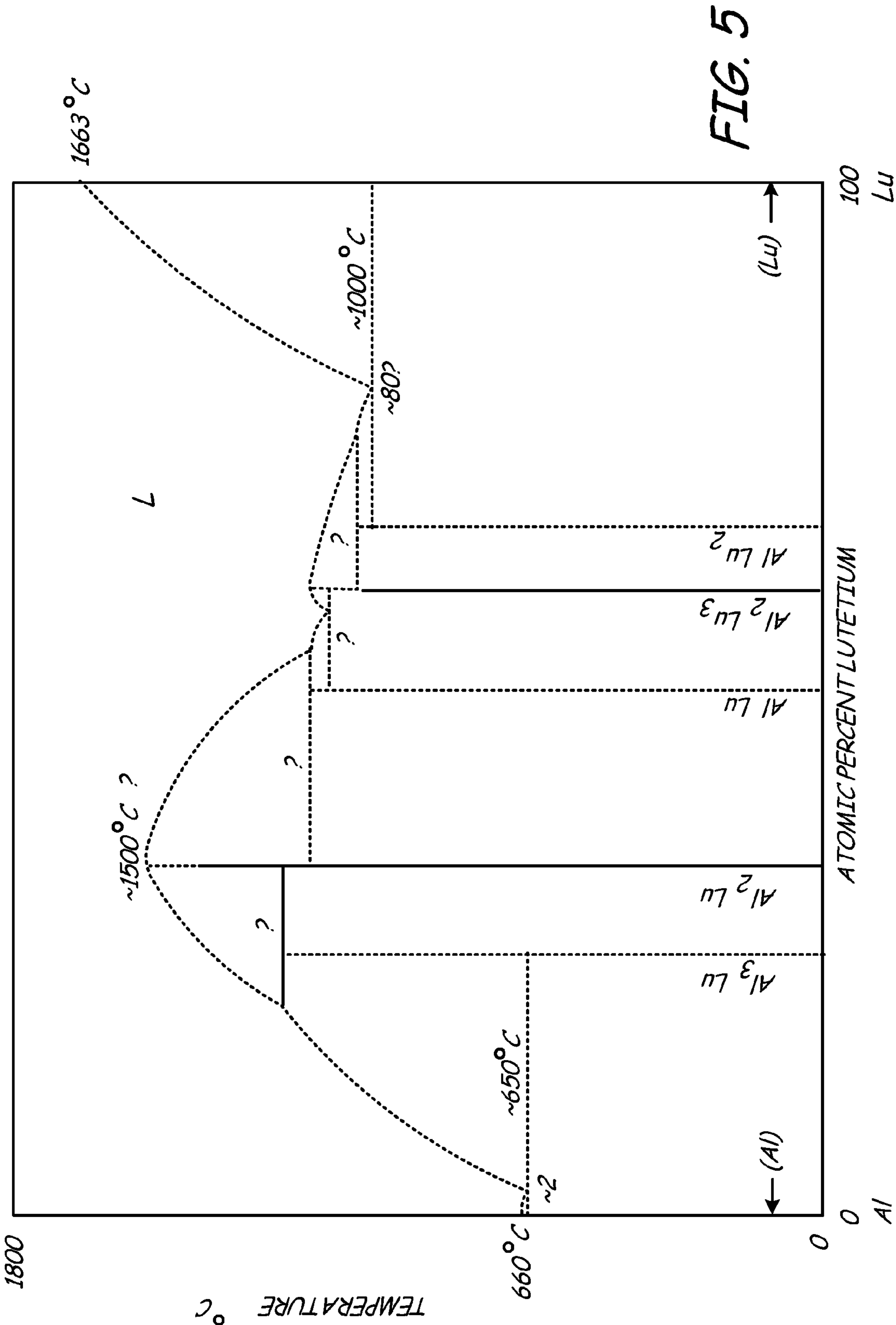


FIG. 2







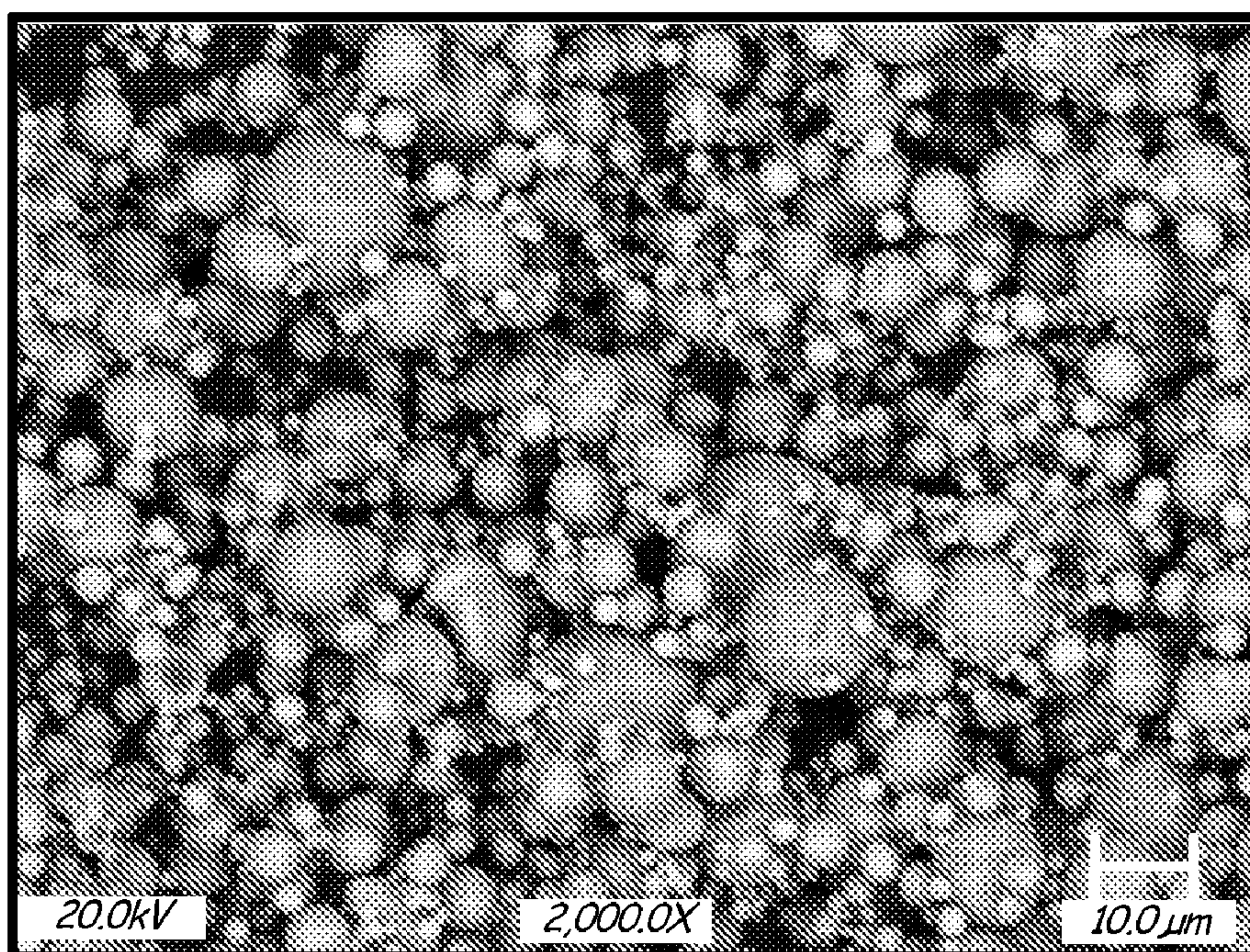


FIG. 6A

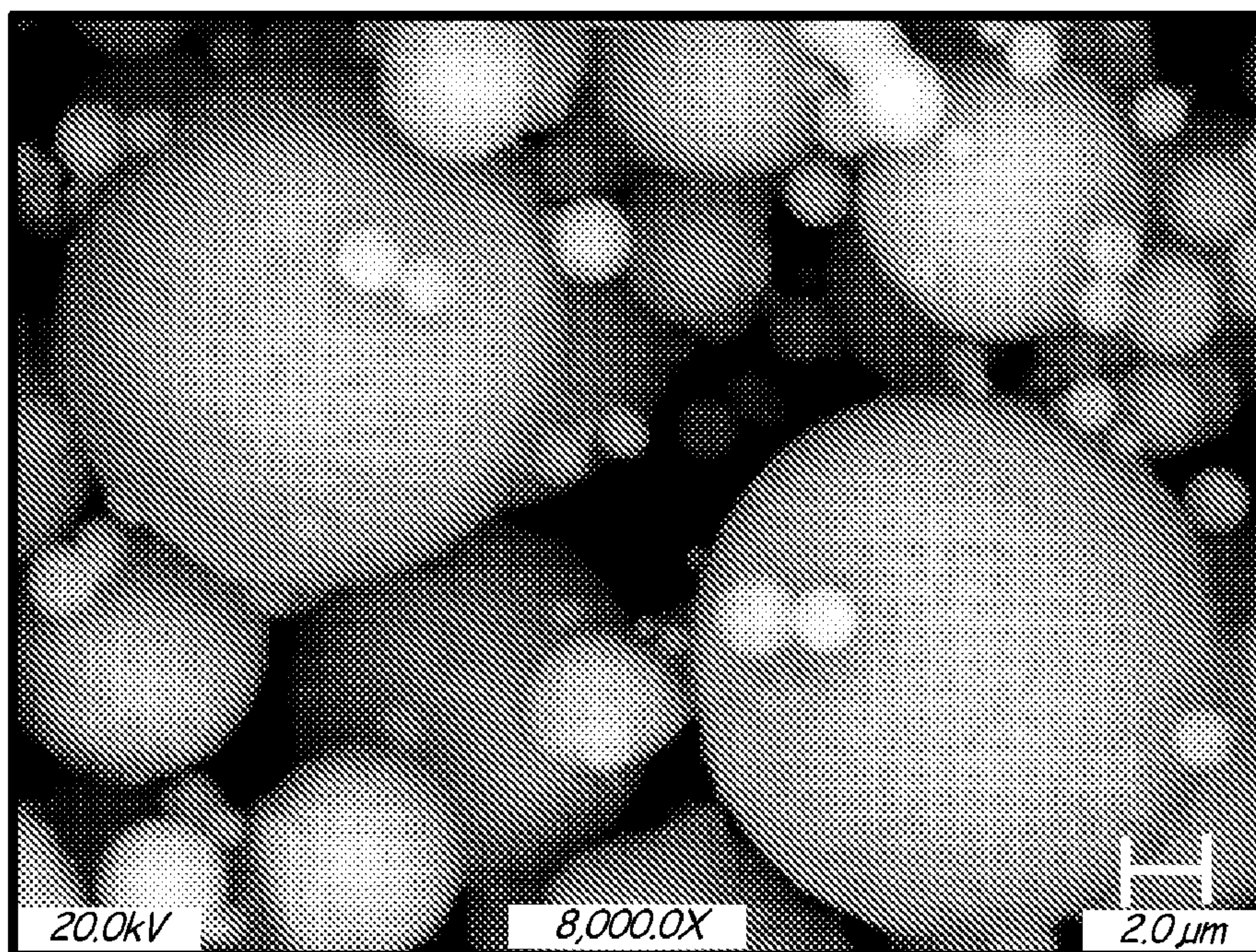


FIG. 6B

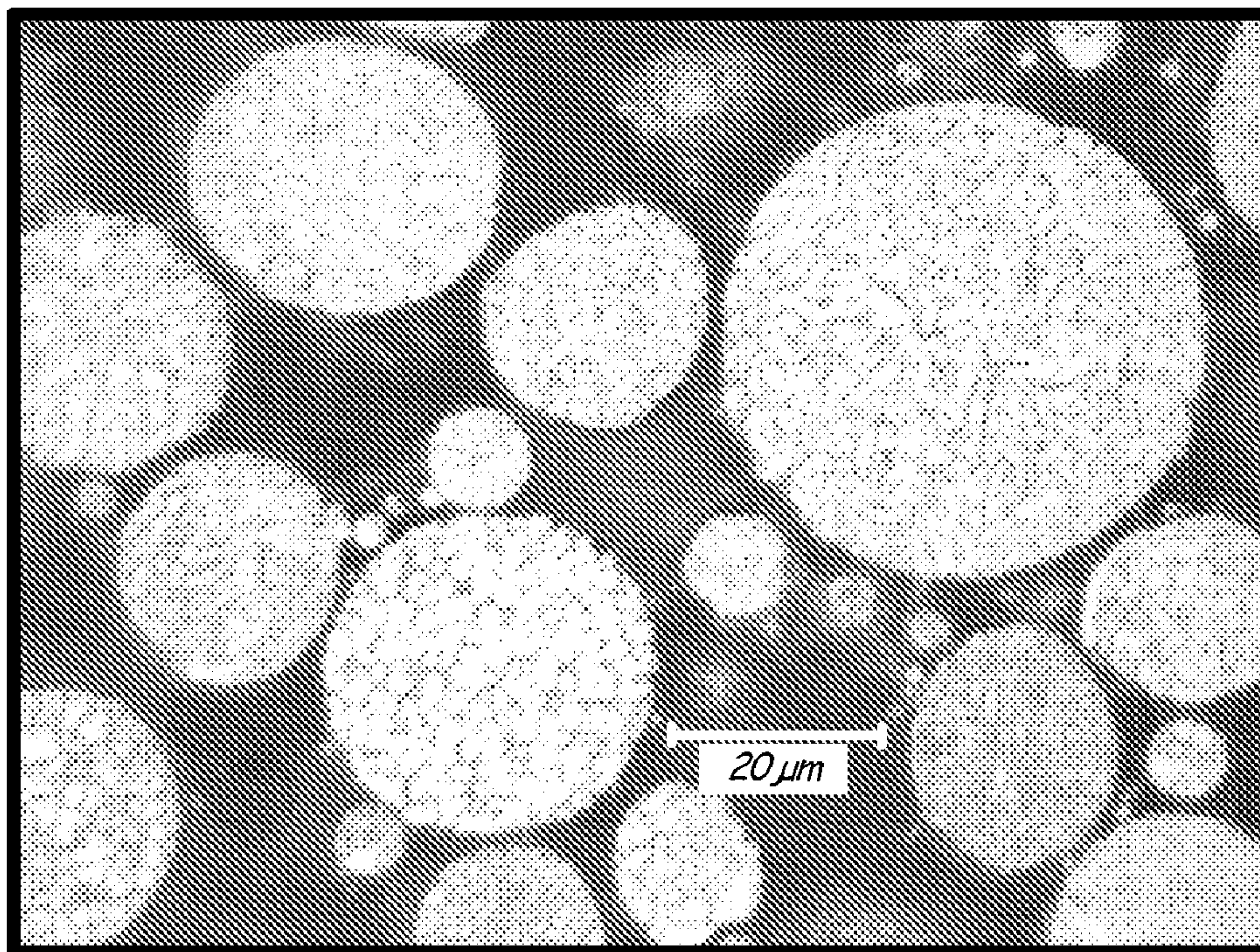


FIG. 7A

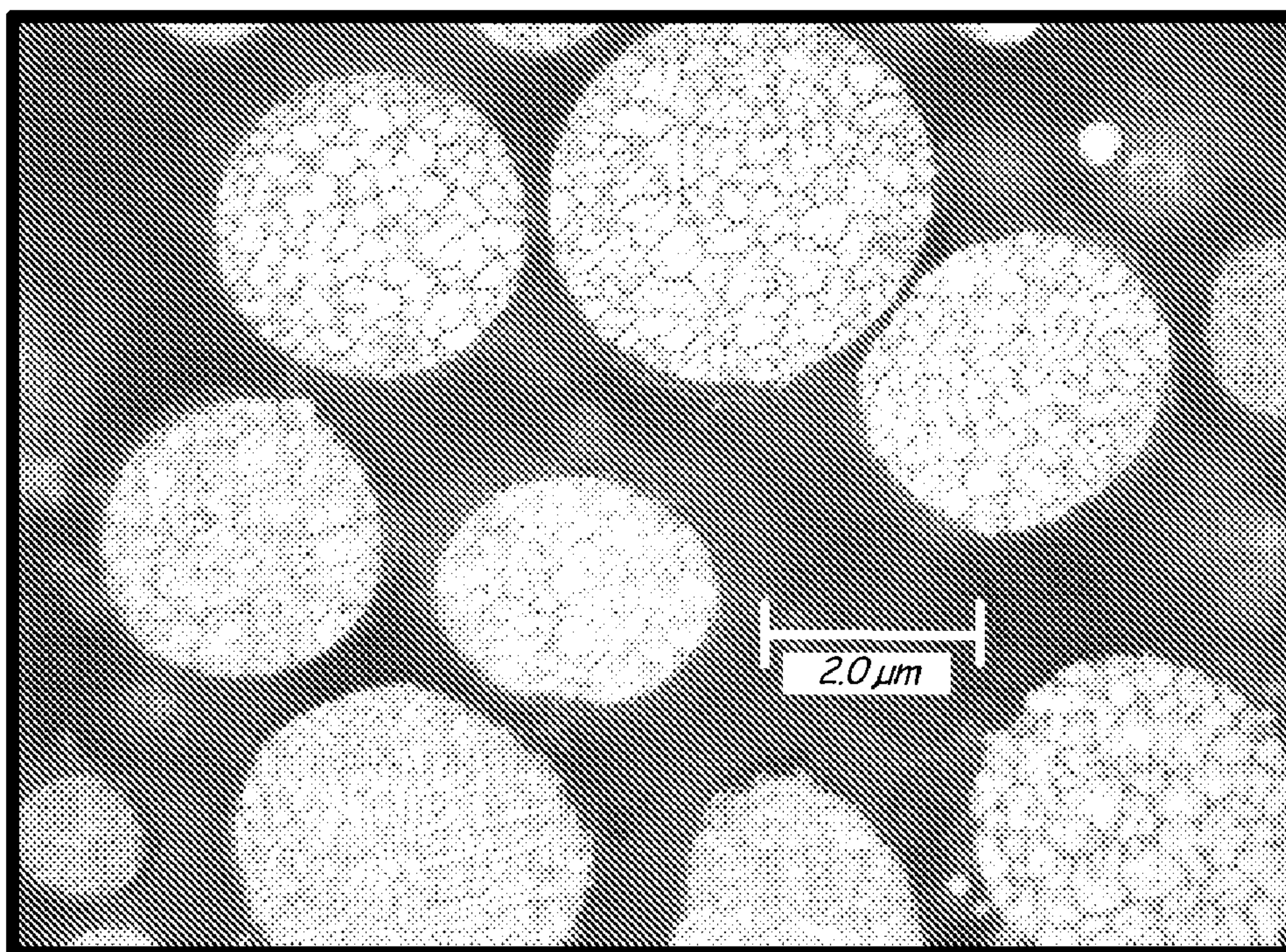


FIG. 7B

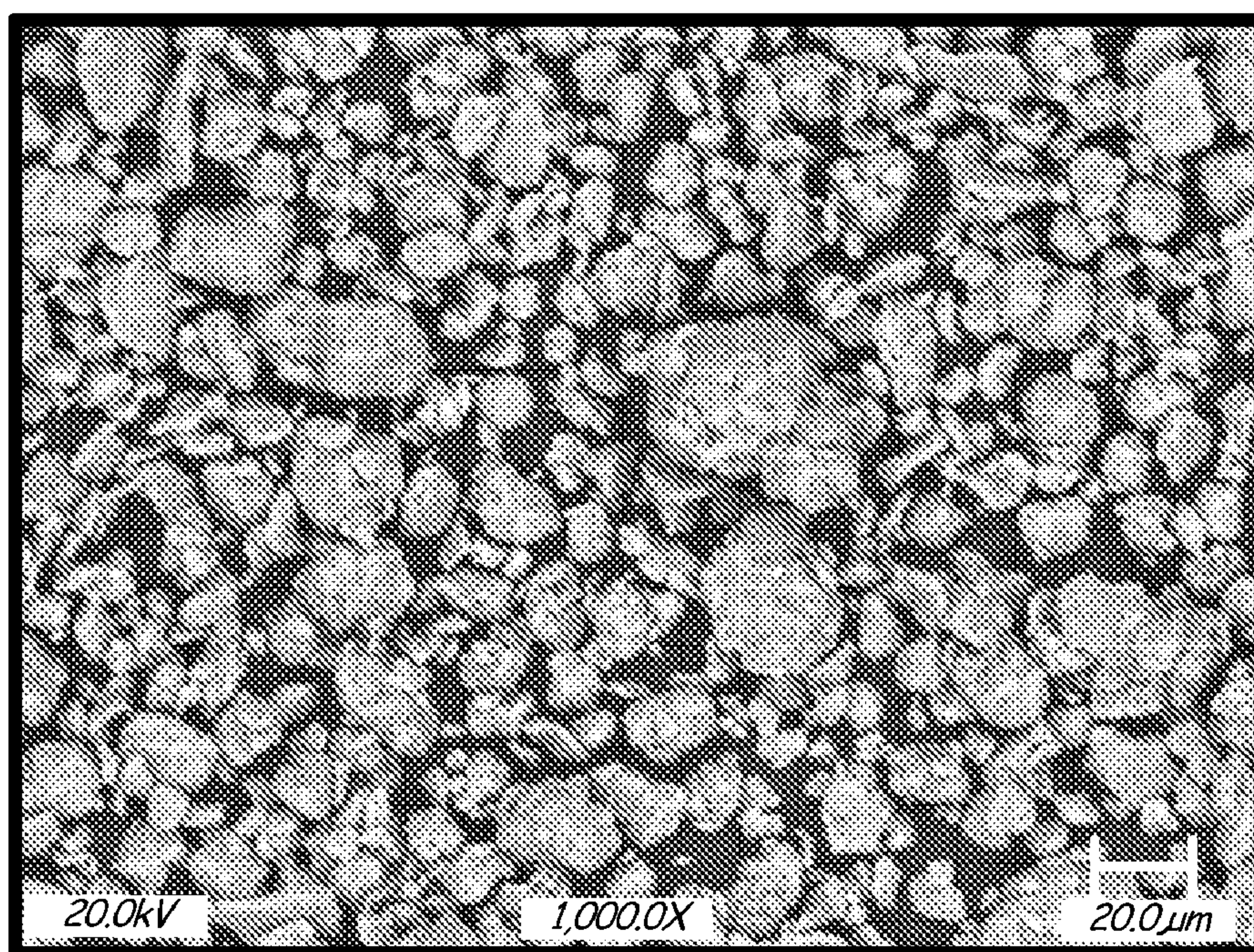


FIG. 8A

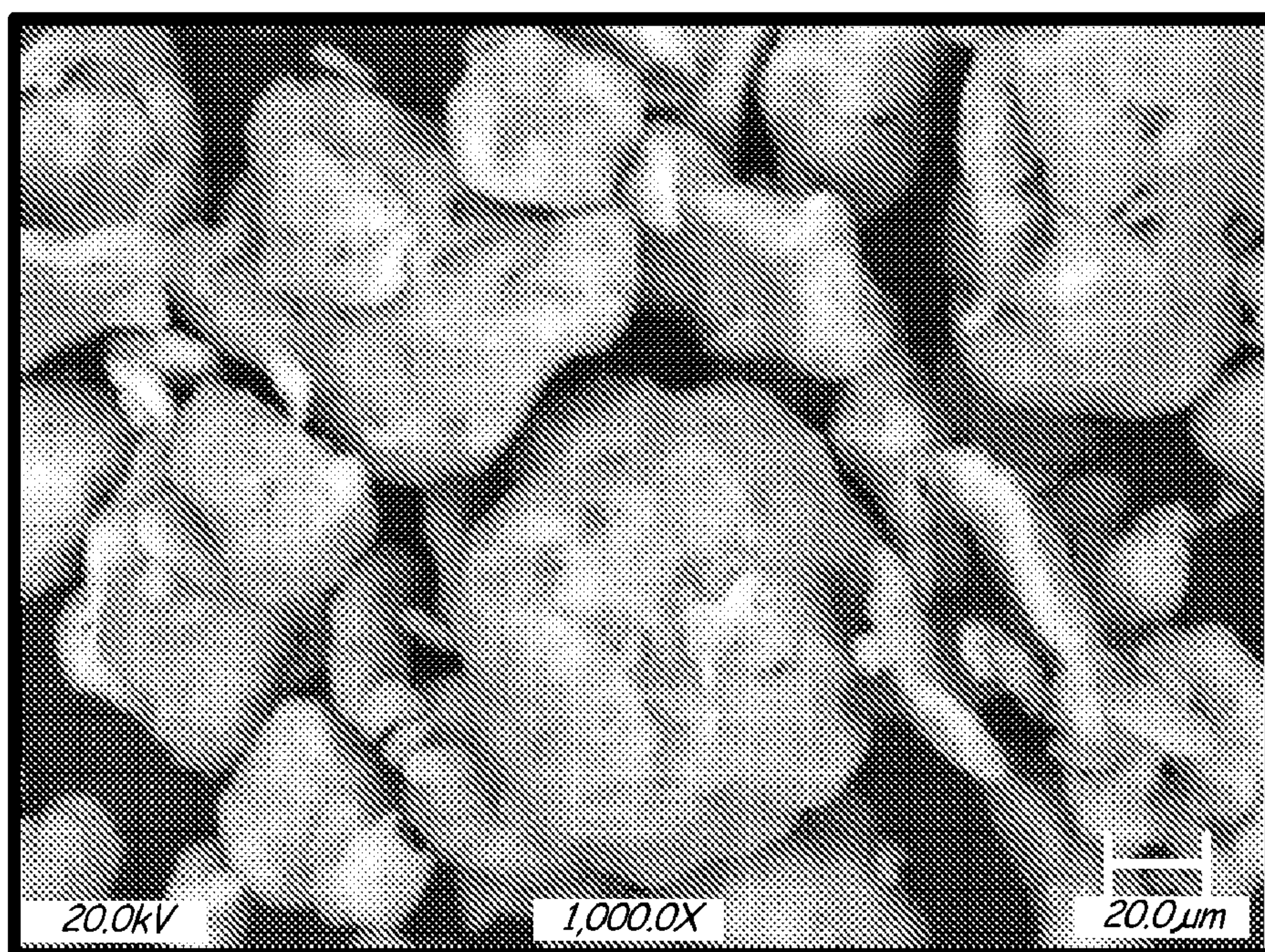


FIG. 8B

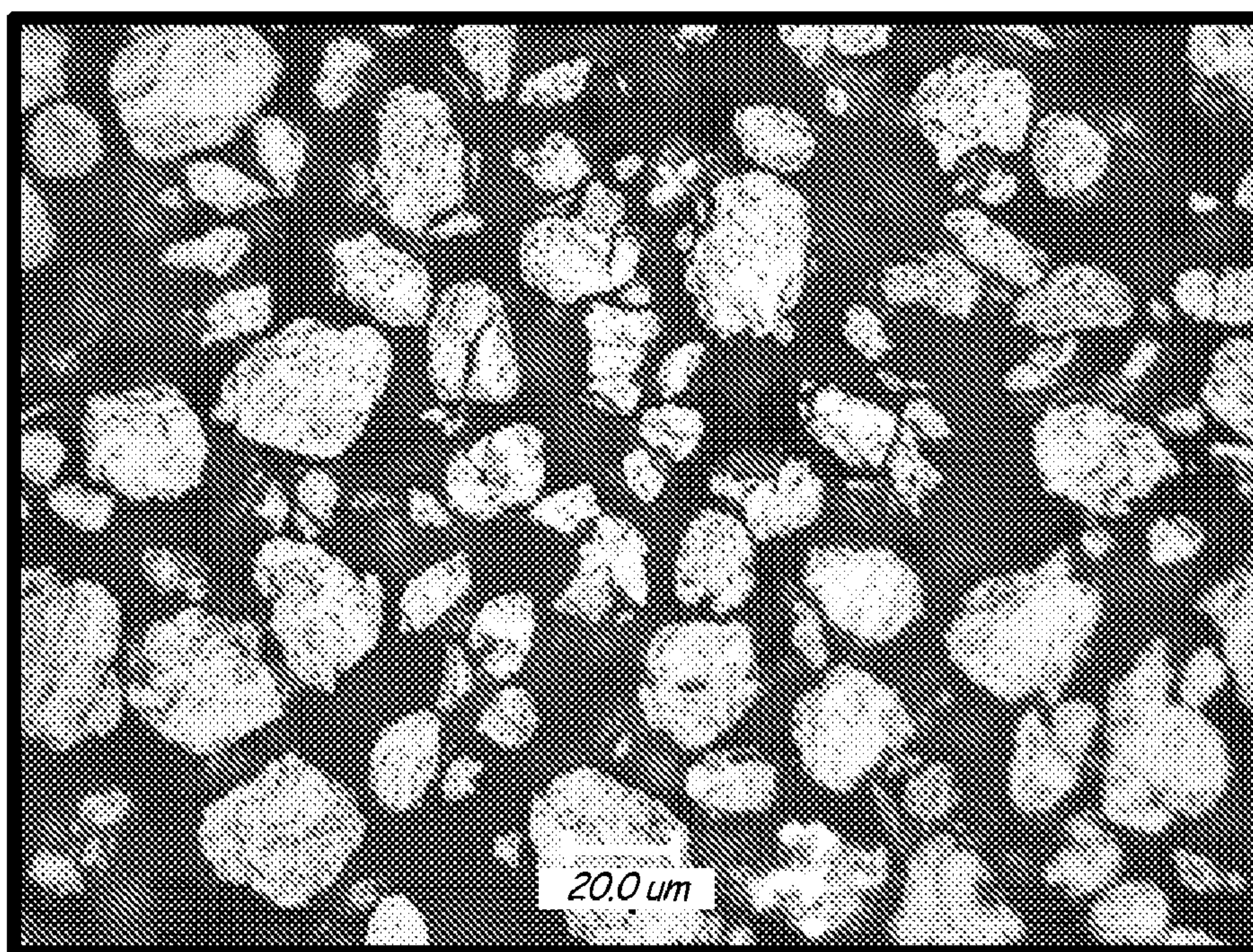


FIG. 9A

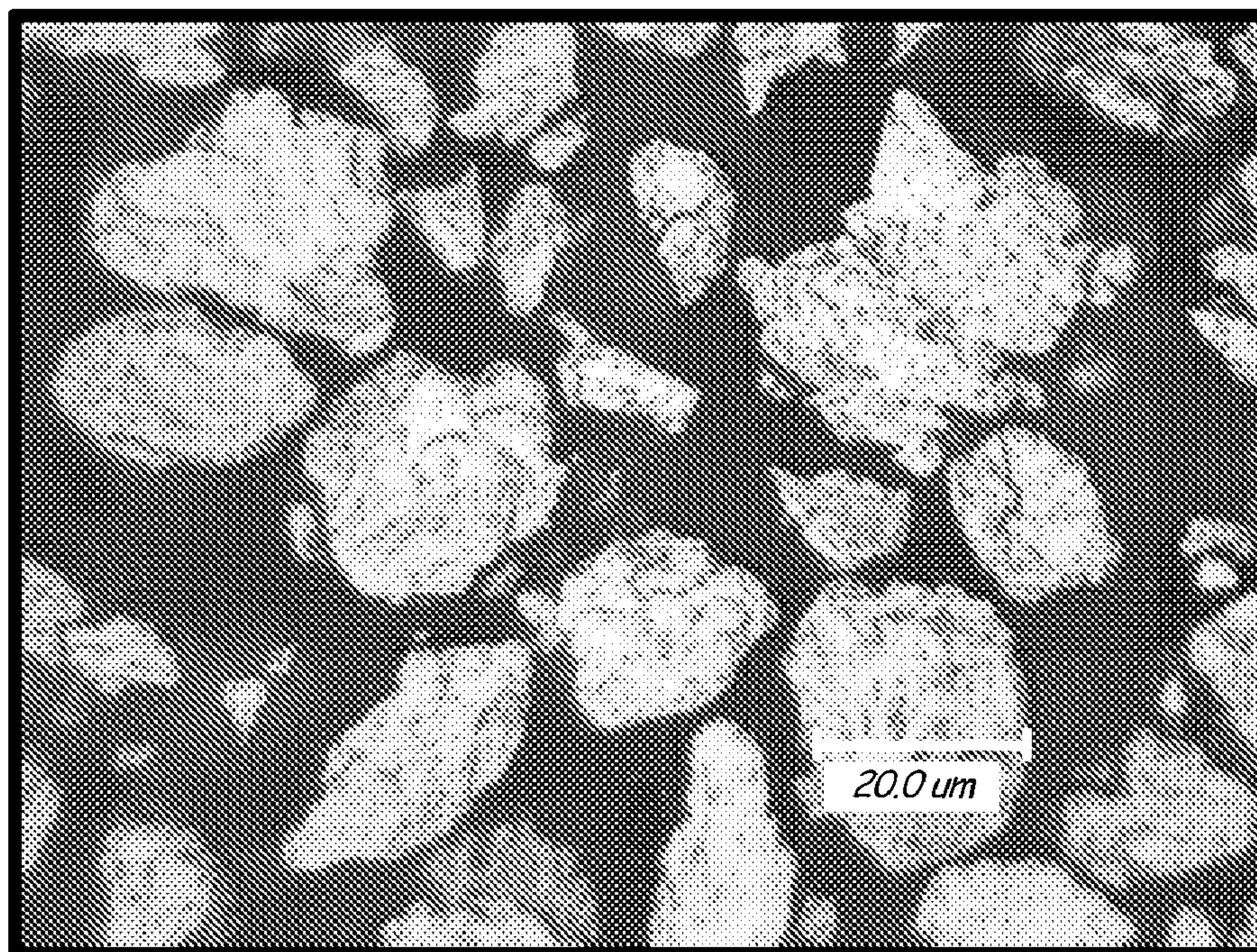
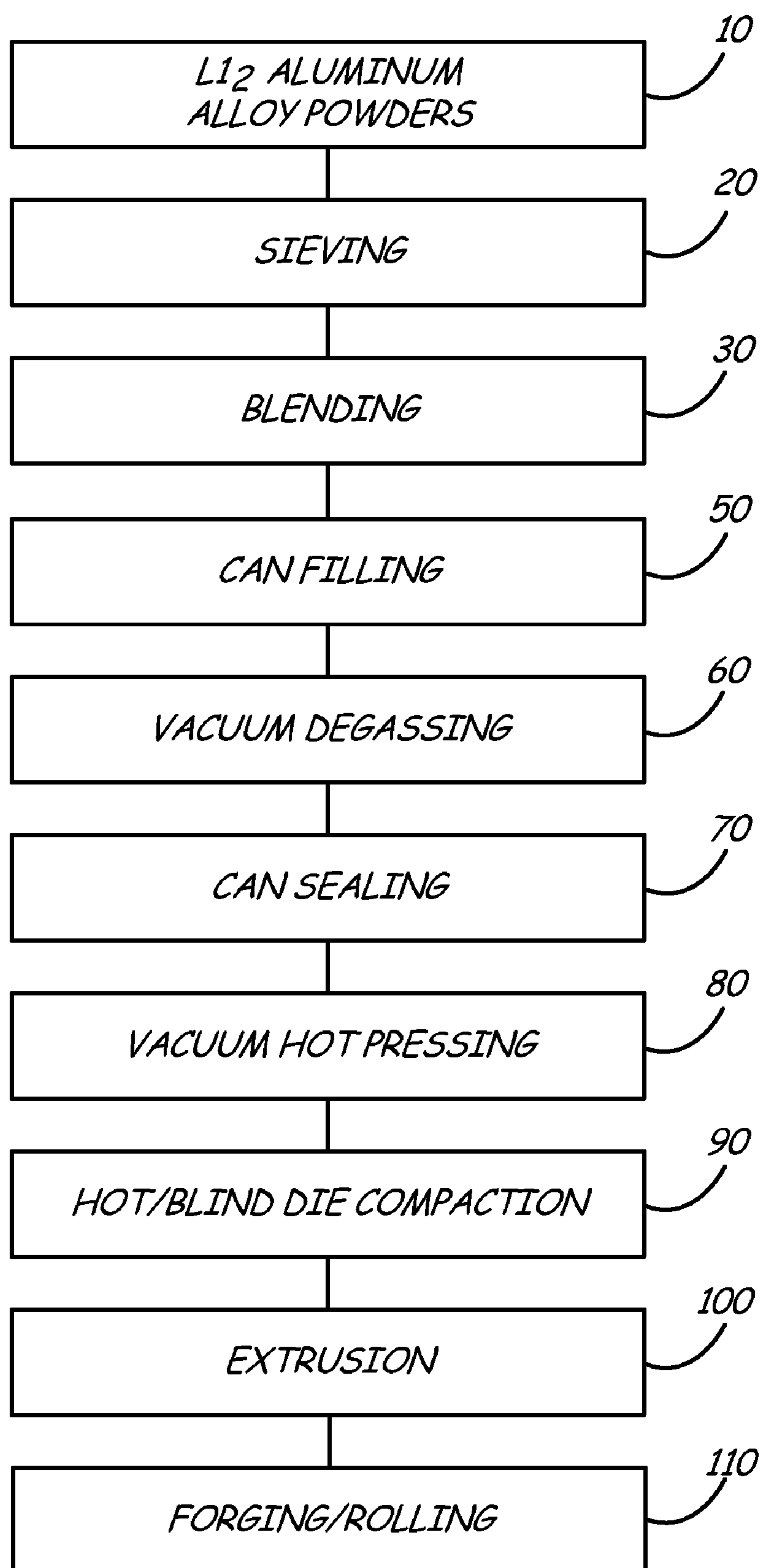


FIG. 9B

*FIG. 10*

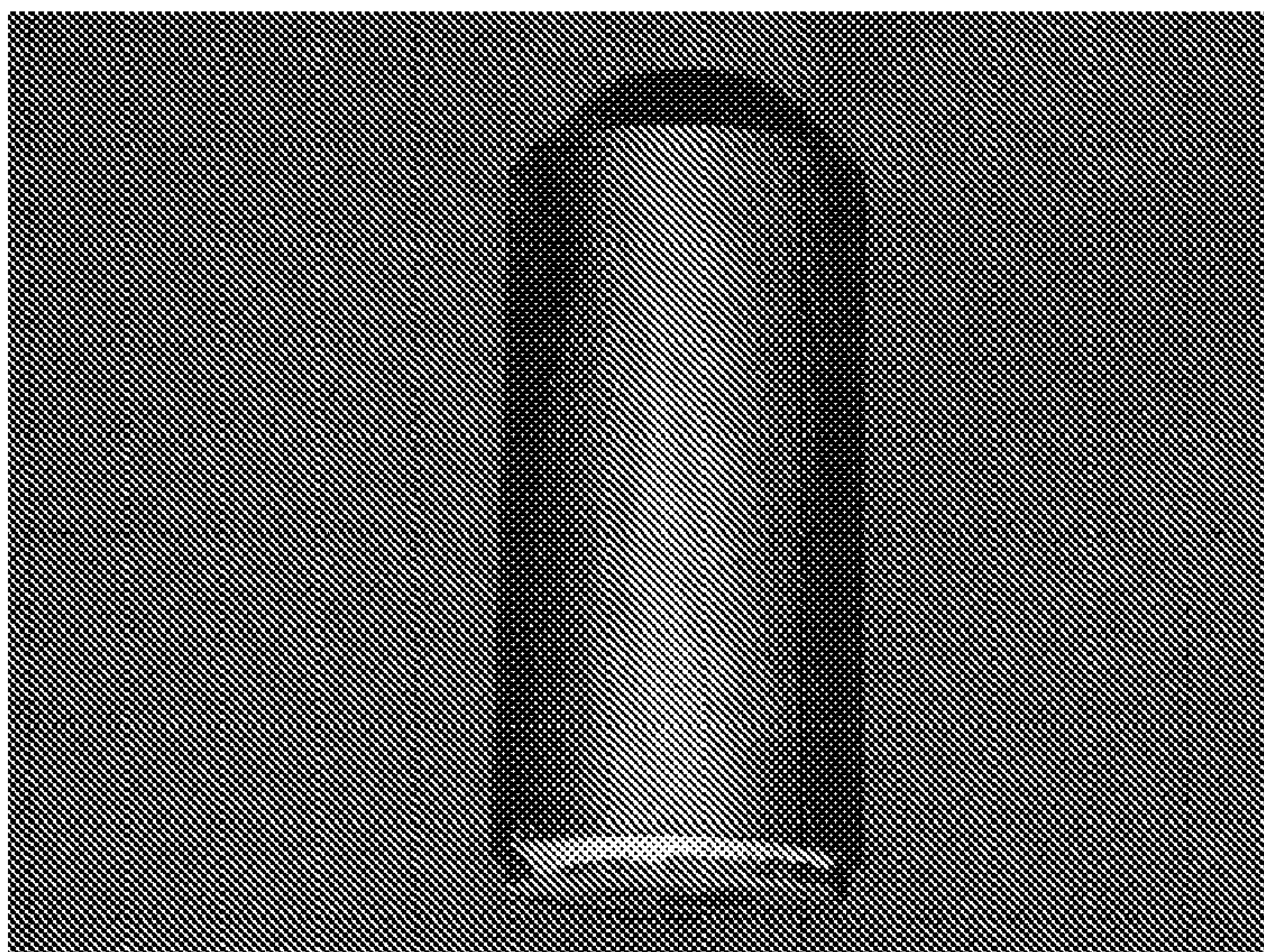


FIG. 11

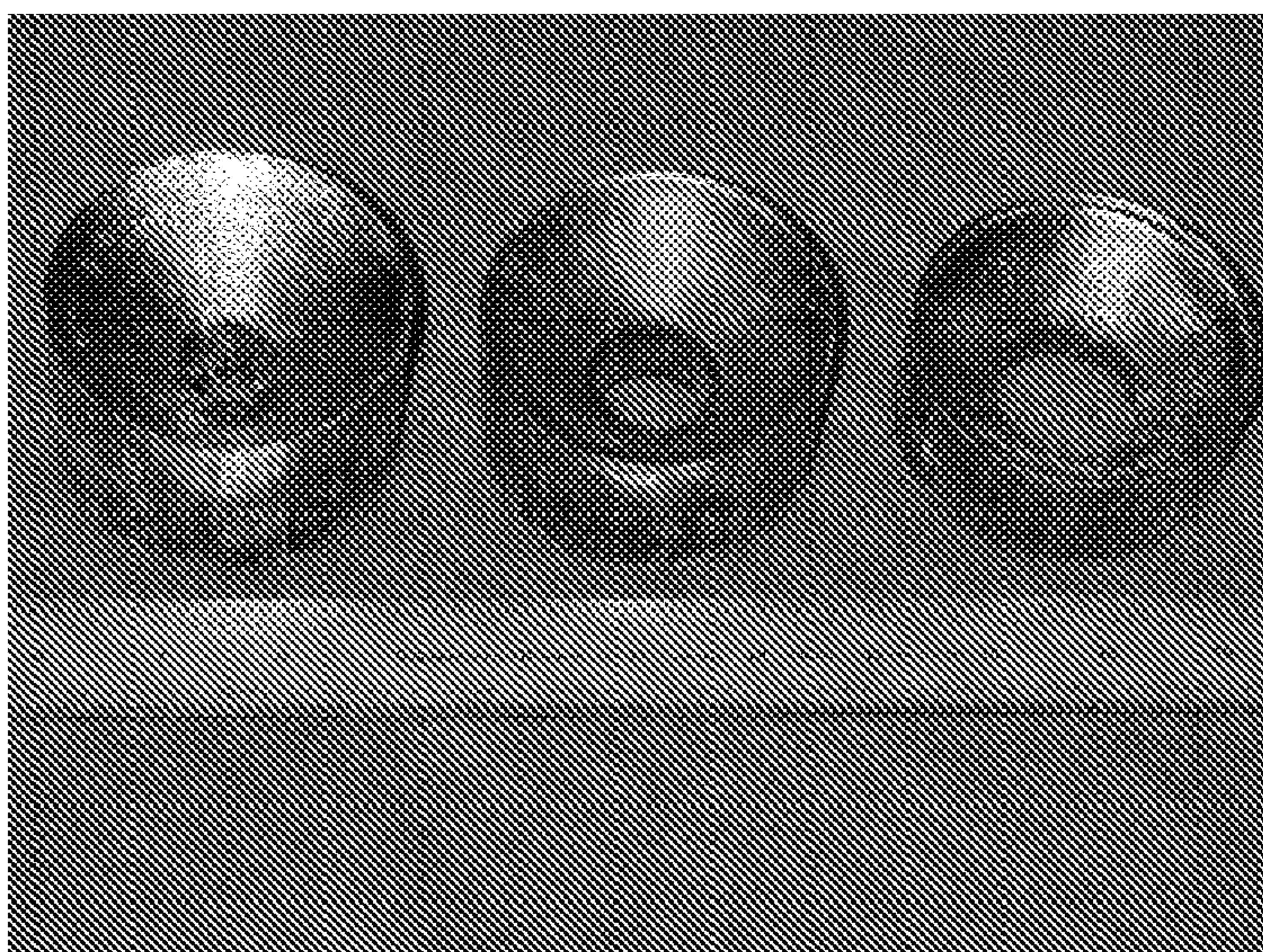


FIG. 12

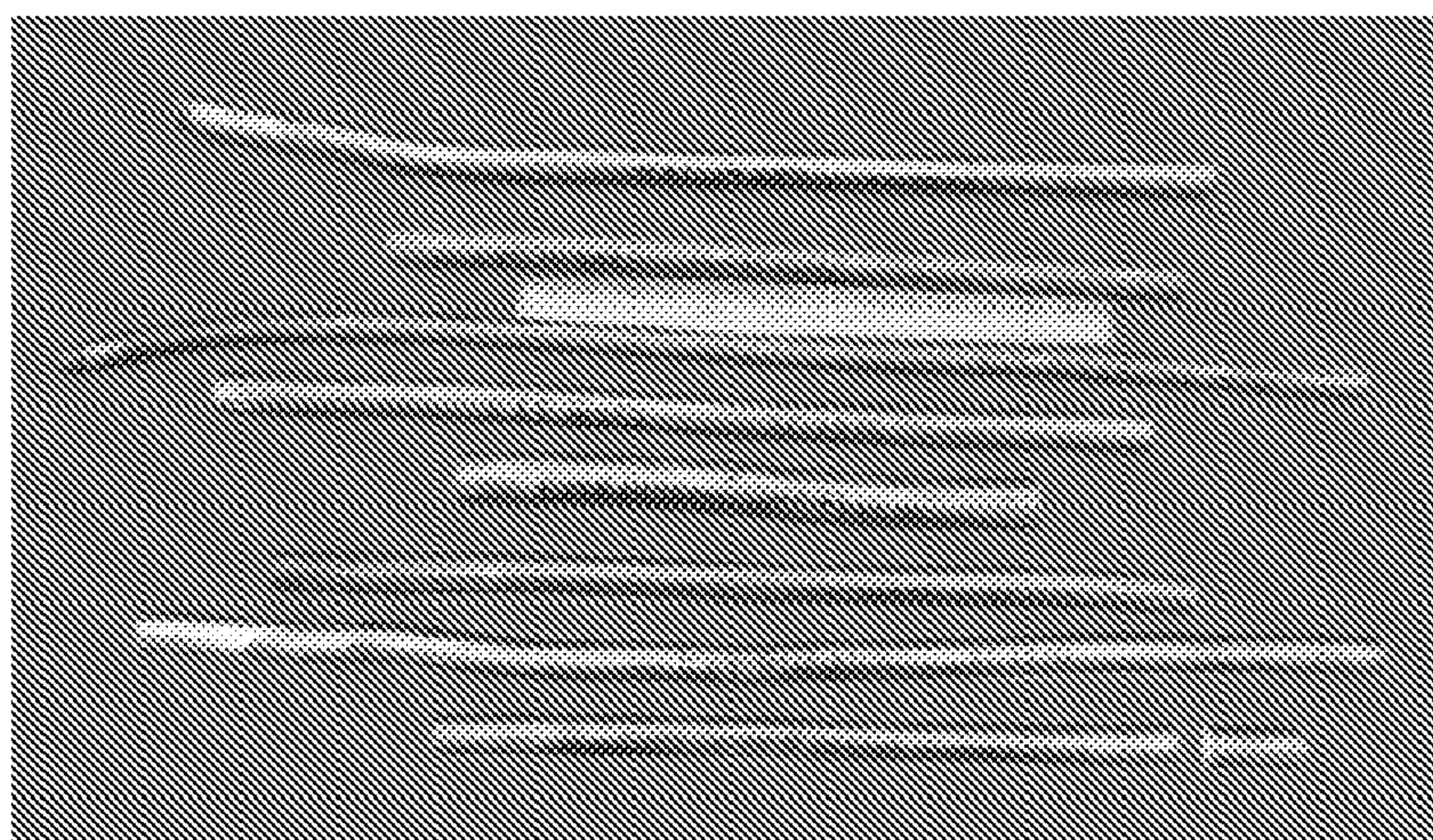


FIG. 13

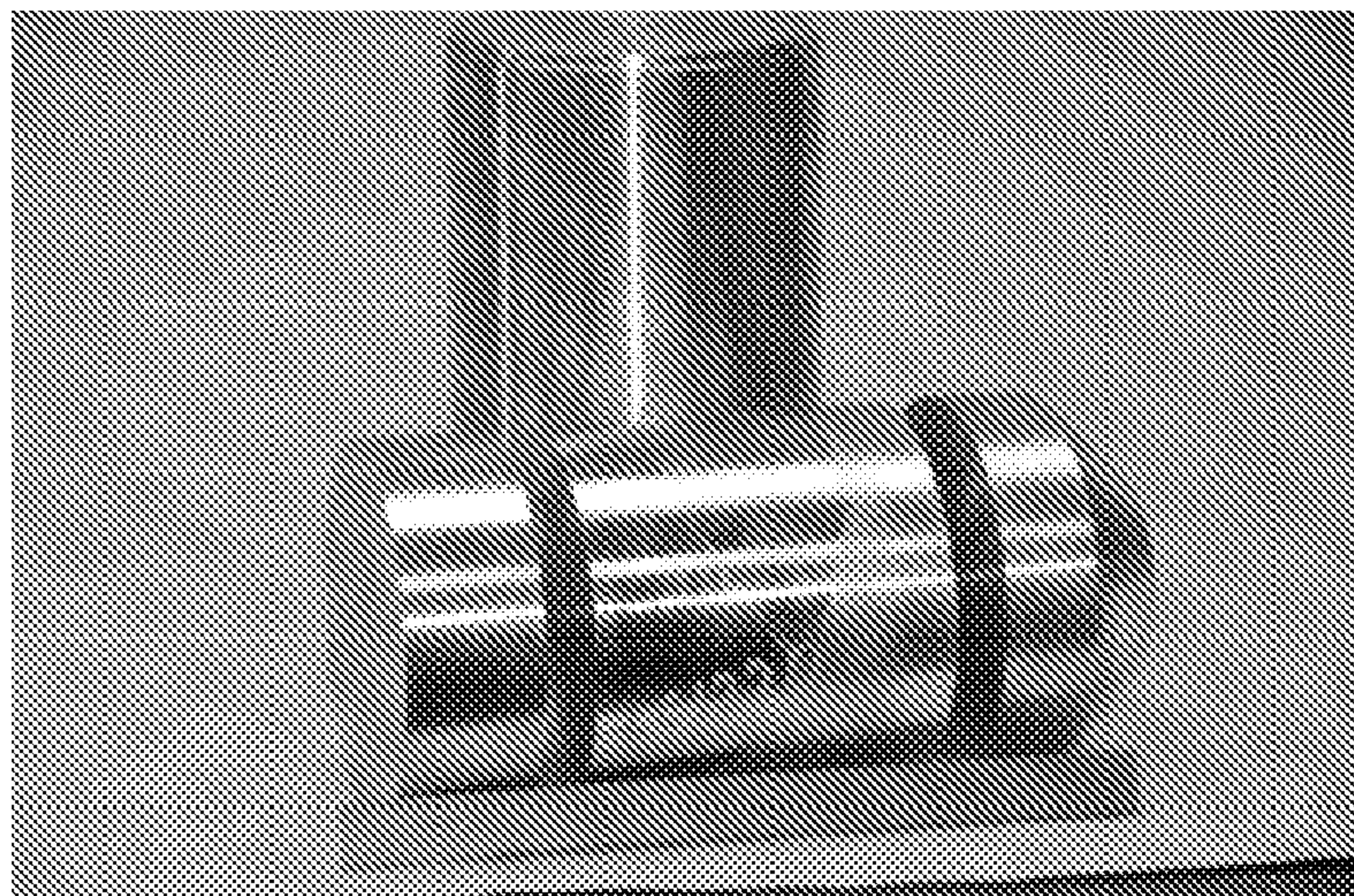


FIG. 14

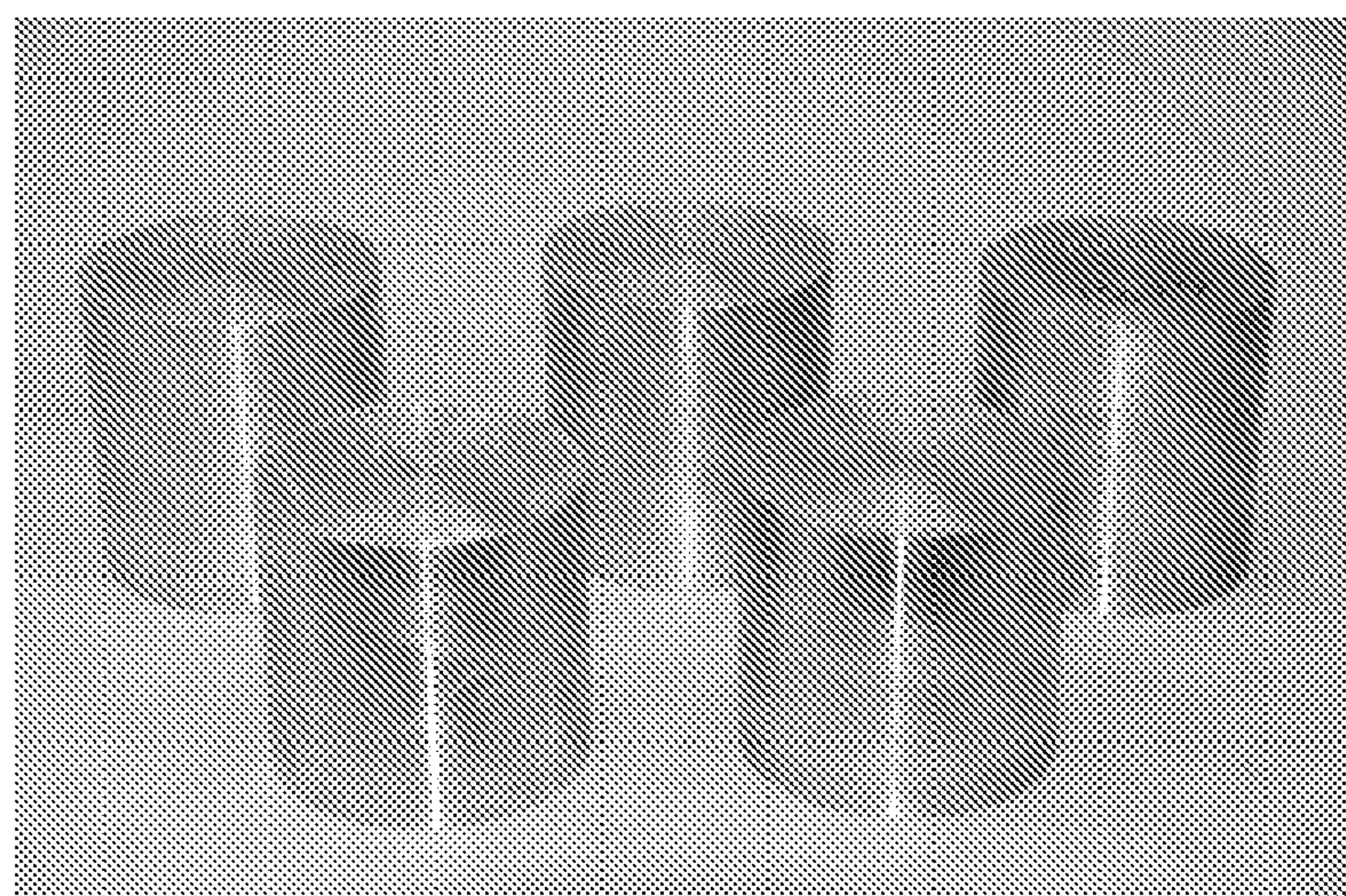


FIG. 15

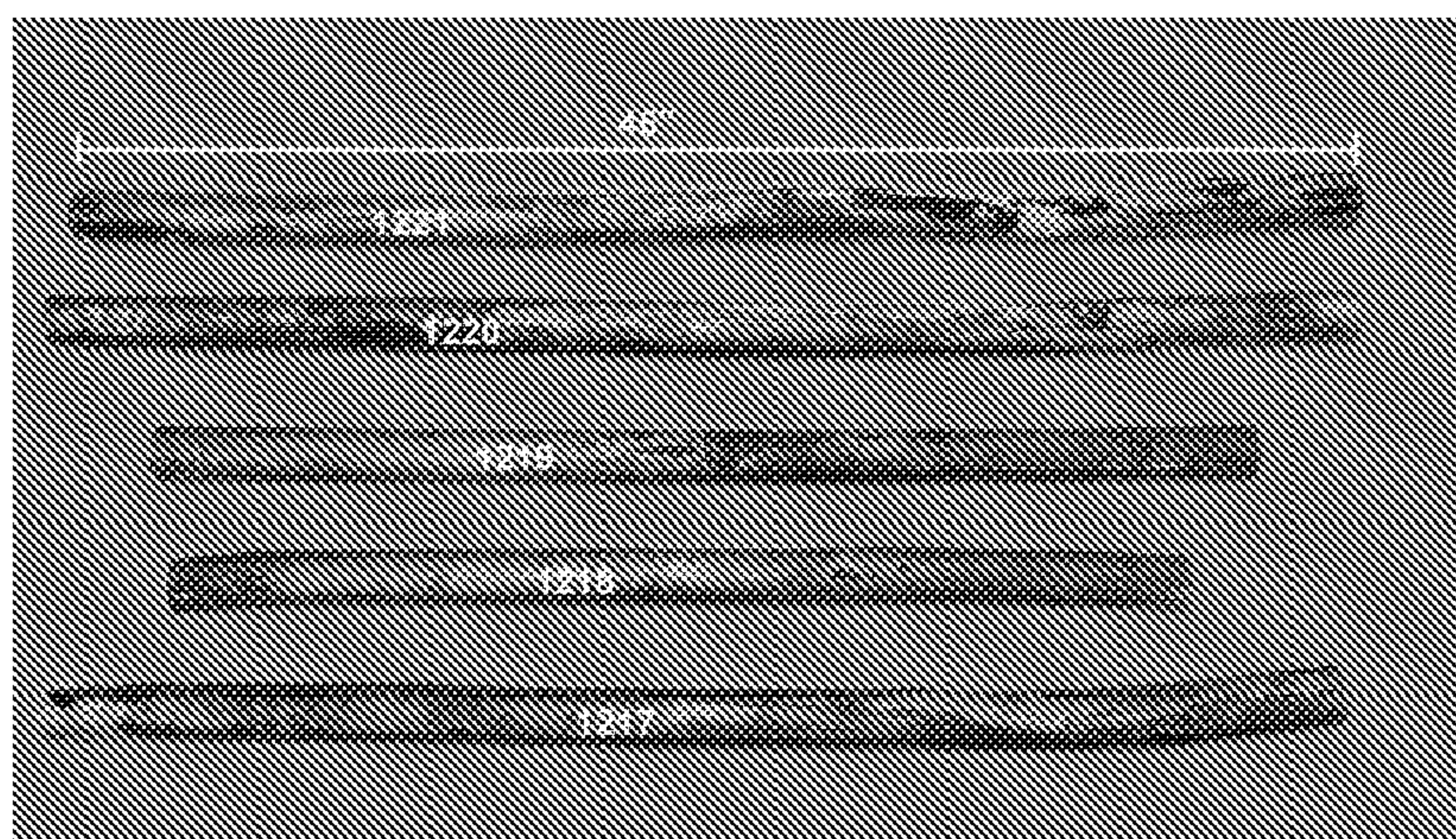


FIG. 16

HIGH STRENGTH L₁₂ ALUMINUM ALLOYS PRODUCED BY CRYOMILLING

CROSS-REFERENCE TO RELATED APPLICATION(S)

[0001] This application is related to the following co-pending applications that were filed on Dec. 9, 2008 herewith and are assigned to the same assignee: CONVERSION PROCESS FOR HEAT TREATABLE L₁₂ ALUMINUM ALLOYS, Ser. No. 12/316,020; A METHOD FOR FORMING HIGH STRENGTH ALUMINUM ALLOYS CONTAINING L₁₂ INTERMETALLIC DISPERSOIDS, Ser. No. 12/316,046; and A METHOD FOR PRODUCING HIGH STRENGTH ALUMINUM ALLOY POWDER CONTAINING L₁₂ INTERMETALLIC DISPERSOIDS, Ser. No. 12/316,047.

[0002] This application is also related to the following co-pending applications that were filed on Apr. 18, 2008, and are assigned to the same assignee: L₁₂ ALUMINUM ALLOYS WITH BIMODAL AND TRIMODAL DISTRIBUTION, Ser. No. 12/148,395; DISPERSION STRENGTHENED L₁₂ ALUMINUM ALLOYS, Ser. No. 12/148,432; HEAT TREATABLE L₁₂ ALUMINUM ALLOYS, Ser. No. 12/148,383; HIGH STRENGTH L₁₂ ALUMINUM ALLOYS, Ser. No. 12/148,394; HIGH STRENGTH L₁₂ ALUMINUM ALLOYS, Ser. No. 12/148,382; HEAT TREATABLE L₁₂ ALUMINUM ALLOYS, Ser. No. 12/148,396; HIGH STRENGTH L₁₂ ALUMINUM ALLOYS, Ser. No. 12/148,387; HIGH STRENGTH ALUMINUM ALLOYS WITH L₁₂ PRECIPITATES, Ser. No. 12/148,426; HIGH STRENGTH L₁₂ ALUMINUM ALLOYS, Ser. No. 12/148,459; and L₁₂ STRENGTHENED AMORPHOUS ALUMINUM ALLOYS, Ser. No. 12/148,458.

BACKGROUND

[0003] 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.

[0004] 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.

[0005] 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.

[0006] 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.

[0007] U.S. Pat. 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 A1, also commonly owned, discloses a high strength aluminum alloy that contains scandium and other elements that is strengthened by L₁₂ dispersoids.

[0008] 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 nano range of about 30 to 100 nm. These alloys also have lower ductility.

SUMMARY

[0009] The present invention is a method for consolidating cryomilled aluminum alloy powders into useful components with high temperature strength and fracture toughness. In embodiments, powders include an aluminum alloy having coherent L₁₂ Al₃X 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 powders are classified by sieving and blended to improve homogeneity. Cryomilling is an essential step in the manufacturing process. The cryomilled powders are then vacuum degassed in a container that is then sealed. The sealed container (i.e. can) is compressed by vacuum hot pressing, hot isostatic pressing or blind die compaction to densify the powder charge. 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

[0011] FIG. 1 is an aluminum scandium phase diagram.

[0012] FIG. 2 is an aluminum erbium phase diagram.

[0013] FIG. 3 is an aluminum thulium phase diagram.

[0014] FIG. 4 is an aluminum ytterbium phase diagram.

[0015] FIG. 5 is an aluminum lutetium phase diagram.

[0016] FIG. 6A and 6B are SEM photos of the gas atomized inventive L₁₂ aluminum alloy powder.

[0017] FIG. 7A and 7B are photomicrographs of cross-sections showing the cellular microstructure of the gas atomized inventive L₁₂ aluminum alloy powder.

[0018] FIG. 8A and 8B are photomicrographs of cryomilled powder of the inventive L₁₂ aluminum alloy powder.

[0019] FIG. 9A and 9B are photomicrographs of cross-sections of cryomilled powder of the inventive L₁₂ aluminum alloy powder.

[0020] FIG. 10 is a diagram showing the processing steps to consolidate L₁₂ aluminum alloy powder.

[0021] FIG. 11 is a photo of a 3-inch diameter copper jacketed L₁₂ aluminum alloy billet.

[0022] FIG. 12 is a photo of extrusion dies for 3-inch diameter billet.

[0023] FIG. 13 is a photo of extruded $L1_2$ aluminum alloy rods from 3-inch diameter billets.

[0024] FIG. 14 is a photo of machined $L1_2$ aluminum alloy billets.

[0025] FIG. 15 is a photo of a machined three-piece $L1_2$ aluminum alloy billet assembly for 6-inch copper jacketed extrusion billet.

[0026] FIG. 16 is a photo of extruded $L1_2$ aluminum alloy rods from 6-inch diameter billets.

DETAILED DESCRIPTION

1. $L1_2$ Aluminum Alloys

[0027] Alloy powders refined by 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.

[0028] 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

[0029] 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

[0030] 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.

[0031] 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.

[0032] 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.

[0033] 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 Al_3Ni .

[0034] 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 Al_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.

[0035] 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. 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.

[0036] Erbium forms Al_3Er dispersoids in the aluminum matrix that are fine and coherent with the aluminum matrix. The lattice parameters of aluminum and Al_3Er are close (0.405 nm and 0.417 nm respectively), indicating there is minimal driving force for causing growth of the Al_3Er dispersoids. This low interfacial energy makes the Al_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 Al_3Er to coarsening. Additions of zinc, copper, lithium, silicon, and nickel provide solid solution and precipitation strengthening in the aluminum alloys. 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.

[0037] 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. 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.

[0038] Ytterbium forms Al_3Yb dispersoids in the aluminum matrix that are fine and coherent with the aluminum

matrix. The lattice parameters of Al and Al_3Yb are close (0.405 nm and 0.420 nm respectively), indicating there is minimal driving force for causing growth of the Al_3Yb dispersoids. This low interfacial energy makes the Al_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. 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.

[0039] 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. 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.

[0040] 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 DO_{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.

[0041] Yttrium forms metastable Al_3Y dispersoids in the aluminum matrix that have an L1_2 structure in the metastable condition and a DO_{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.

[0042] Zirconium forms Al_3Zr 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_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.

[0043] 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 dispersoids 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.

[0044] Hafnium forms metastable Al_3Hf 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_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.

[0045] Niobium forms metastable Al_3Nb dispersoids in the aluminum matrix that have an L1_2 structure in the metastable condition and a DO_{22} structure in the equilibrium condition. Niobium has a lower solubility in the Al_3X dispersoids than hafnium or yttrium, allowing relatively lower amounts of niobium than hafnium or yttrium to substitute for X in the Al_3X dispersoids. Nonetheless, niobium can be very effective in slowing down the coarsening kinetics of the Al_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.

[0046] 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.

[0047] 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.

[0048] Exemplary aluminum alloys for the bimodal system alloys of this invention include, but are not limited to (in weight percent unless otherwise specified):

- [0049] about Al-M-(0.1-4)Sc-(0.1-20)Gd;
- [0050] about Al-M-(0.1-20)Er-(0.1-20)Gd;
- [0051] about Al-M-(0.1-15)Tm-(0.1-20)Gd;
- [0052] about Al-M-(0.1-25)Yb-(0.1-20)Gd;
- [0053] about Al-M-(0.1-25)Lu-(0.1-20)Gd;
- [0054] about Al-M-(0.1-4)Sc-(0.1-20)Y;
- [0055] about Al-M-(0.1-20)Er-(0.1-20)Y;
- [0056] about Al-M-(0.1-15)Tm-(0.1-20)Y;
- [0057] about Al-M-(0.1-25)Yb-(0.1-20)Y;
- [0058] about Al-M-(0.1-25)Lu-(0.1-20)Y;
- [0059] about Al-M-(0.1-4)Sc-(0.05-4)Zr;
- [0060] about Al-M-(0.1-20)Er-(0.05-4)Zr;
- [0061] about Al-M-(0.1-15)Tm-(0.05-4)Zr;
- [0062] about Al-M-(0.1-25)Yb-(0.05-4)Zr;
- [0063] about Al-M-(0.1-25)Lu-(0.05-4)Zr;
- [0064] about Al-M-(0.1-4)Sc-(0.05-10)Ti;
- [0065] about Al-M-(0.1-20)Er-(0.05-10)Ti;
- [0066] about Al-M-(0.1-15)Tm-(0.05-10)Ti;
- [0067] about Al-M-(0.1-25)Yb-(0.05-10)Ti;
- [0068] about Al-M-(0.1-25)Lu-(0.05-10)Ti;
- [0069] about Al-M-(0.1-4)Sc-(0.05-10)Hf;
- [0070] about Al-M-(0.1-20)Er-(0.05-10)Hf;
- [0071] about Al-M-(0.1-15)Tm-(0.05-10)Hf;
- [0072] about Al-M-(0.1-25)Yb-(0.05-10)Hf;
- [0073] about Al-M-(0.1-25)Lu-(0.05-10)Hf;
- [0074] about Al-M-(0.1-4)Sc-(0.05-5)Nb;
- [0075] about Al-M-(0.1-20)Er-(0.05-5)Nb;
- [0076] about Al-M-(0.1-15)Tm-(0.05-5)Nb;
- [0077] about Al-M-(0.1-25)Yb-(0.05-5)Nb; and
- [0078] about Al-M-(0.1-25)Lu-(0.05-5)Nb.

[0079] M is at least one of about (4-25) weight percent silicon, (1-8) weight percent magnesium, (0.5-3) weight percent lithium, (0.2-3) weight percent copper, (3-12) weight percent zinc, and (1-12) weight percent nickel.

[0080] 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.

[0081] 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.

[0082] 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.

[0083] The amount of copper present in the fine grain matrix, if any, may vary from about 0.2 to about 3 weight percent, more preferably from about 0.5 to about 2.5 weight percent, and even more preferably from about 1 to about 2.5 weight percent.

[0084] 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.

[0085] 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.

[0086] 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 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^{30} C./second.

[0087] 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 $L1_2$ 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^{30} C./second.

[0088] 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 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^{30} C./second.

[0089] 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 $L1_2$ intermetallic Al_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 $10^{3^{\circ}}$ C./second.

[0090] 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 $L1_2$ 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 $10^{3^{\circ}}$ C./second.

[0091] 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.

[0092] 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.

[0093] 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.

[0094] 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.

[0095] 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.

[0096] 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.

[0097] 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 $L1_2$ Alloy Powder

[0098] Gas atomized high temperature $L1_2$ aluminum alloy powder needs to be consolidated into solid-state forms suitable for engineering applications. Scanning electron micrographs of gas atomized $L1_2$ 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 $10^{3^{\circ}}$ C./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 $L1_2$ 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 $L1_2$ strengthening dispersoids in the final consolidated alloy structure. FIGS. 8A and 8B show photomicrographs of cryomilled powders at different magnifications indicating that the shape of the spherical powder has changed from spherical to irregular due to the milling operation. The microstructure of cryomilled powder shown in FIGS. 9A and 9B indicates that the cellular structure is refined and there is a more uniform distribution of fine particles present in the powder. The process of consolidating the alloy powders into useful forms is schematically illustrated in FIG. 10.

[0099] $L1_2$ 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. The starting stock should be at least -325 mesh powder (step 30). Other benefits of blending will be discussed later. Powders are then cryomilled to decrease grain size and improve strength (step 40). Cryomilling is carried out in a high-energy ball mill under liquid nitrogen, and offers several benefits that will be discussed later.

[0100] The sieved, blended and cryomilled powders are then put in a can (step 50) and vacuum degassed (step 60). Following vacuum degassing the can is sealed (step 70) under vacuum and hot pressed (step 80) to densify the powder compact. The compact is then hot worked (step 90) to refine the microstructure. Finally, the densified compact is machined into an extrusion billet and extruded (step 100) to produce a product with improved mechanical properties useful for subsequent service as a high temperature $L1_2$ strengthened aluminum alloy. Extrusion results in optimum mechanical properties in the extrusion direction. If more uniform directional properties are required, forging and/or rolling (step 110) is necessary.

[0101] Sieving (step 20) is a preferred step in consolidation because the final mechanical properties relate directly to the particle size. Finer particle size results in finer $L1_2$ particle dispersion. Optimum 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 improve the efficiency of sieving. If the as-atomized powder is oxygen deficient, the powder can have a tendency to stick together which will lower the efficiency for sieving. Ultrasonic sieving is preferred for its efficiency.

[0102] 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 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.

[0103] Cryomilling (step 40) is a preferred step and is 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. Pat. No. 6,902,699, Fritze-meier et al. and in U.S. Pat. No. 7,344,675, Van Daam et al., both owned by the assignee of the present invention, and are incorporated herein in their entirety by reference. Cryomilling involves high-energy ball milling under liquid nitrogen. The liquid nitrogen facilitates efficient breaking up of powder particles. 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 structure 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 (0.04 microinch) and the structure is considered a nanostructure.

[0104] In addition, the nitrogen environment results in the formation of nitride particles that reside at the grain boundaries and inside grains themselves 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 L1₂ aluminum alloy particles during milling.

[0105] Following sieving (step 20), blending (step 30) and cryomilling (step 40), the powders are transferred to a 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 configu-

material. Forging and/or rolling (step 110) can improve the uniformity of the short transverse mechanical properties.

[0108] FIG. 11 shows a 3-inch (7.62 cm) diameter copper jacketed L1₂ aluminum alloy billet ready for extrusion. FIG. 12 is a photo of three 3-inch (7.62 cm) diameter extrusion dies. Representative extrusions using the 3-inch (7.62 cm) diameter dies are shown in FIG. 13. A 12-inch (30.48 cm) ruler is included in the photo for size comparison. Larger 6-inch (15.24 cm) diameter billets were also extruded. Machined 6-inch (15.24 cm) diameter L1₂ aluminum alloy extrusion billets are shown in FIG. 14. FIG. 15 is a photo of a machined three-piece copper jacketed 6-inch (15.24 cm) diameter billet assembly. A 12-inch (30.48 cm) 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.

[0109] Extruded L1₂ aluminum alloy rods from 6-inch diameter billets are shown in FIG. 16. The top rod is 46 inches (116.8 cm) long.

[0110] Representative processing parameters for 3-inch diameter L1₂ aluminum alloy billets are listed in Table 1.

TABLE 1

Processing Details of L12 Strengthened Alloys					
Billet Number	Degassing/VHP Temperature	Extrusion Temperature	Billet Temperature/Time	Liner Temperature	Extrusion Speed
1	600° F. (316° C.)	450° F. (232° C.)	450° F. (232° C.)	450° F. (232° C.)	0.5"/min (1.2 cm/min)
2	700° F. (371° C.)	400° F. (204° C.)	400° F. (204° C.)	400° F. (204° C.)	0.5"/min (1.2 cm/min)
3	600° F. (316° C.)	650° F. (343° C.)	650° F. (343° C.)	650° F. (343° C.)	0.5"/min (1.2 cm/min)
4	550° F. (288° C.)	600° F. (316° C.)	600° F. (316° C.)	600° F. (316° C.)	0.5"/min (1.2 cm/min)
5	700° F. (371° C.)	450° F. (232° C.)	450° F. (232° C.)	450° F. (232° C.)	0.5"/min (1.2 cm/min)
6	700° F. (371° C.)	350° F. (177° C.)	350° F. (177° C.)	350° F. (177° C.)	0.5"/min (1.2 cm/min)

ration with a central axis. Vacuum degassing times can range from 12 hours to over 8 days. 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 of large amounts of powder are 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 the stearic acid lubricant as well as oxygen and hydrogen from the powder.

[0106] Following vacuum degassing (step 60), the vacuum line is crimped and welded shut. The powder is then consolidated further by uniaxially 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. At this point the powder charge is nearly 100 percent dense. The billet can be compressed by blind die compaction (step 90) to further densify the structure. Blind die compaction is preferred to further densify the billet as prior consolidation processes may not provide 100% density due to insufficient load available with the press. If uniaxial hot pressing or hot isostatic pressing are used 100% density can be achieved and blind die compaction may not be required. Following densification, the can may be removed by machining.

[0107] Following blind die compaction, the billet is machined into an extrusion billet, copper jacketed and extruded (step 100). The extrusion process preferably increases the hardness and improves the tensile ductility. Extrusion imparts directional mechanical properties to the

TABLE 2

Tensile Properties of L12 Strengthened Aluminum Alloys				
Billet Number	Tensile Strength, ksi (MPa)	Yield Strength, Ksi (MPa)	Elongation, %	Reduction in Area, %
1	113 (779)	103 (710)	3.8	6.7
2	119 (820)	112 (772)	3.2	7.0
3	101 (696)	97 (669)	3.9	9.7
4	111 (765)	105 (724)	2.7	8.3
5	118 (813)	102 (703)	2.1	3.5
6	120 (827)	108 (745)	0.5	2.0

Representative mechanical properties of extruded aluminum alloy billets are listed in Table 2. Table 1 shows processing details of degassing, vacuum hot pressing (VHP), and extrusion parameters used for fabrication of this material. Degassing and consolidation of billets were performed in the range of 550° F. (288° C.) to 700° F. (371° C.). Extrusion was performed in the 350° F. (177° C.) to 650° F. (343° C.) temperature range where billet, die and liner temperatures were maintained equal. Extrusion speed was maintained at 0.5 inches per minute (1.27 cm per minute). Lower speed is desired for higher strength due to less adiabatic heat generation during extrusion. Table 2 includes tensile properties of extrusions that resulted from the above processing parameters. The measured tensile strength ranges from 101 ksi (696 MPa) to 120 ksi (827 MPa) and yield strength ranges from 97 ksi

(667 MPa) to 108 ksi (745 MPa). These strength values are significantly higher than commercially available existing aluminum alloys. It should be noted that higher strength was obtained for lower extrusion temperature conditions. For example, billet number 3 showed a yield strength of 97 ksi (669 MPa) and tensile strength of 101 ksi (696 MPa) for an extrusion temperature of 650° F. (343° C.), whereas billet number 6 showed a yield strength of 108 ksi (745 MPa) and a tensile strength of 120 ksi (827 MPa) for an extrusion temperature of 350° F. (177° C.). Billet number 2 showed a yield strength of 112 ksi (772 MPa) and a tensile strength of 118 (813 MPa) for an extrusion temperature of 400° F. (204° C.) which is very close to the strength obtained for an extrusion temperature of 350° F. (177° C.). Ductility which is measured by elongation and reduction in area also showed variations with extrusion temperature. Higher extrusion temperatures resulted in higher ductility. Higher strength values obtained for these extrusions made from cryomilled billets suggest that cryomilling has worked very effectively for L1₂ strengthened aluminum alloys.

[0111] The above mentioned processing steps (and others) for consolidating L1₂ aluminum alloy powder are well known to those versed in the art and need not be described in more detail.

[0112] 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.

1. A method for producing a high strength aluminum alloy billet containing L1₂ dispersoids, comprising the steps of:

- cryomilling a quantity of an aluminum alloy powder containing an L1₂ dispersoid therein to produce a mesh size of less than -325 mesh;
- placing the cryomilled powder in a container;
- vacuum degassing the powder at a temperature of about 500° F. (260° C.) to about 900° F. (482° C.) for about 12 hours to about 8 days;
- sealing the degassed powder in the container under vacuum;
- heating the sealed container at about 700° F. (371° C.) to about 900° F. (482° C.) for about one 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 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 hour to about 8 hours at a temperature of 700° F. (371° C.) to about 900° F. (482° C.) under uni-axial pressure of about 20 ksi (138 Mpa) to about 100 ksi (690 MPa).

4. The method of claim 1, wherein the vacuum hot pressing produces a billet of the aluminum alloy powder having a theoretical 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 pressure of about 20 ksi (138 Mpa) to about 100 ksi (690 MPa).

7. The method of claim 6, wherein the extrusion temperature is about 300° F. (149° C.) to about 850° F. (454° C.), the billet soak time is about 0.5 hours to about 8 hours at a rate of about 0.2 inch per minute (0.51 cm per minute) to about 10 inch per minute (25.4 cm per minute), and an extrusion ratio of about 2:1 to about 40:1.

8. The method of claim 1, wherein the L1₂ dispersoids comprise Al₃X 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.

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 3 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 L1₂ dispersoids comprise Al₃X 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.

12. A method for producing a high strength aluminum alloy billet containing L1₂ dispersoids, comprising the steps of:

- sieving a quantity of an aluminum alloy powder containing an L1₂ dispersoid therein to produce a particle size of less than 100 mesh;
- blending the sieved aluminum alloy powder to homogenize the particle size distribution;
- cryomilling the blended aluminum alloy powder containing an L1₂ dispersoid therein to produce a mesh size of less than -325 mesh; and
- compacting the cryomilled powder to form a billet.

13. The method of claim 12, wherein the cryomilled powder is placed in a container, vacuum degassed at a temperature of about 500° F. (260° C.) to about 900° F. (482° C.) for about 12 hours to about 8 days; followed by sealing the degassed

powder in the container under vacuum and heating the sealed container at about 700° F. (371° C.) to about 900° F. (482° C.) for about one to eight hours.

14. The method of claim **13**, which further includes the step of vacuum hot pressing the heated container to form a billet; and removing the container from the formed billet.

15. The method of claim **14**, wherein the vacuum hot pressing produces a billet of the aluminum alloy powder having a density of about 100 percent.

16. The method of claim **13**, wherein the degassing includes rotating the aluminum alloy powder to heat and exposing to vacuum all the powder.

17. The method of claim **12**, wherein the formed billet is extruded at a pressure of about 20 ksi (138 MPa) to about 100 ksi (690 MPa).

18. The method of claim **17**, wherein the extrusion temperature is about 300° F. (149° C.) to about 850° F. (454° C.), the billet soak time is about 0.5 hours to about 8 hours at a rate of about 0.2 inch per minute (0.51 cm per minute) to about 10 inch per minute (25.4 cm per minute), and an extrusion ratio of about 2:1 to about 40:1.

19. The method of claim **12**, wherein the $L1_2$ dispersoids comprise 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.

20. The method of claim **19**, 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 3 weight percent copper, about 3 to about 12 weight percent zinc, about 1 to about 12 weight percent nickel.

21. A high strength aluminum alloy billet comprising:

aluminum alloy matrix; and

dispersoids within the aluminum matrix wherein X is at least one first element selected from the group consisting of:

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 consisting of 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

at least one selected from the group consisting 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 3 weight percent copper, about 3 to about 12 weight percent zinc, about 1 to about 12 weight percent nickel;

wherein the billet tensile strength of at least 100 ksi (690 MPa) and a yield strength of at least 95 ksi (655 MPa).

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