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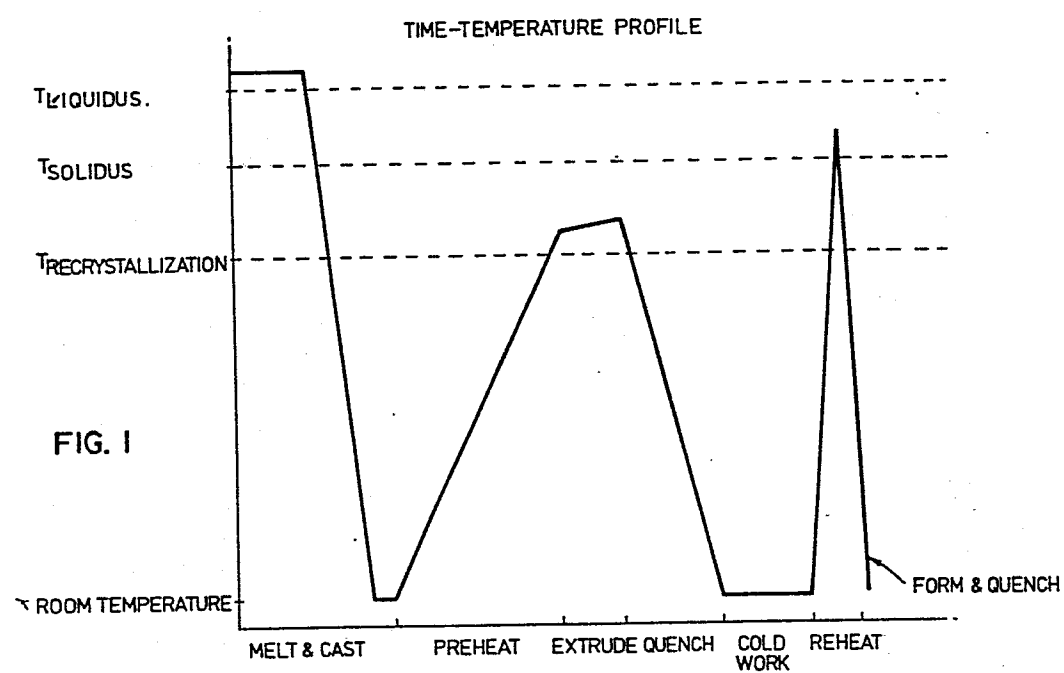
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(54) **Fine grained metal composition.**

(57) A metal alloy composition having a uniform, fine grained microstructure comprising uniform discrete spheroidal particles contained within a lower melting matrix, said alloy being a magnesium, ferrous or copper alloy. The composition is prepared by producing a solid metal alloy composition having an essentially directional grain structure and heating the directional grain composition to a temperature above the solidus and below the liquidus to produce a partially solid, partially liquid mixture containing at least 0.05 volume fraction liquid. The composition, prior to heating, has a strain level introduced such that upon heating, the mixture comprises uniform discrete spheroidal particles contained within a lower melting matrix. The heated alloy is then solidified, the solidified composition having a uniform, fine grained microstructure.

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A METAL ALLOY COMPOSITION

This invention relates to a metal alloy composition.

5 The advantages of shaping metal while in a partially solid, partially liquid condition have now become well known. U.S. patents 3,948,650 and 3,954,455 disclose a process for making possible such shaping processes by the prior vigorous agitation of a metal or metal alloy while
10 it is in a semi-solid condition. This converts the normally dendritic microstructure of the alloy into a non-dendritic form comprising discrete degenerate dendrites in a lower melting matrix. The resulting alloy is capable of being shaped in a semi-solid condition by casting,
15 forging or other known forming processes.

 Considerable cost advantage results from practice of the foregoing semi-solid technology. However, it is subject to certain limitations. The first part of the process normally involves the production of cast bars having the
20 required non-dendritic structure. The technical feasibility of casting diameters of less than about 25.4 mm on a practical scale is very low and, because of the nature of the process even were it feasible, would result in extremely low output. Moreover, the casting process in many instances
25 produces cast bars which exhibit less than desirable skin microstructure which must be trimmed mechanically or otherwise treated for subsequent processing. In addition, the

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generation of diameters of varying size is cumbersome and expensive since each diameter necessitates a complete casting cycle including set-up, mold preparation and running. Flexibility is, therefore, low.

5 It is accordingly a primary object of the present invention to provide a metal composition having a uniform, fine grained microstructure which is unobtainable from any prior metal forming processes.

10 It is an additional object of the invention to provide a metal alloy composition which may be formed in a partially solid, partially liquid condition.

It is an additional object of the invention to provide such a composition which does not require the vigorous agitation of the metal composition during its preparation.

15 The foregoing and other objects of the invention are achieved in a metal alloy composition having a uniform, fine grained microstructure comprising uniform discrete spheroidal particles contained within a lower melting matrix, said alloy being a magnesium, ferrous or copper alloy prepared by producing a solid metal alloy composition having an essentially
20 directional grain structure heating said directional grain composition to a temperature above the solidus and below the liquidus to produce a partially solid, partially liquid mixture, containing at least 0.05 volume fraction liquid, said composition prior to heating having a strain level
25 introduced such that upon heating, the mixture comprises uniform discrete spheroidal particles contained within a matrix composition having a lower melting point than said particles and solidifying said heated alloy compositions.
30 The solidified composition has a uniform, fine grained

microstructure comprising uniform discrete spheroidal particles contained within a lower melting matrix. The metal compositions produced by the foregoing process have a more uniform and finer grain structure than are obtainable by any other known process.

The invention will be better understood by reference to the accompanying drawing in which:

FIGURE 1 is a time-temperature profile of a typical process in accordance with the practice of the invention;

FIGURES 2 through 14 are photomicrographs showing the microstructure of alloys at various stages in the process of the invention. All micrographs are at a magnification of 100.

It is normally considered extremely harmful to heat an alloy even a small amount above its solidus temperature during heat treating or shaping processes (other than casting) because of grain boundary melting and resulting embrittlement of the metal. Such melting, often referred to as hot shortness or burning, adversely affects workability and decreases the strength and ductility of the alloy. There are isolated disclosures in the literature of exceptions to the avoidance of melting but they are largely variations of solutionizing processes in which heterogeneities are removed by dissolving them in a matrix phase. For example, U.S. patent 2,249,349 heats an aluminum alloy to incipient fusion to improve its hot workability. U.S. patents 3,988,180, 4,106,956, 4,019,927 heat an alloy to just above the solidus temperature and hold the alloy at

that temperature until the dendritic phase becomes globular.

In all of this prior art, however, the heterogeneities caused by melting are deleterious and must be removed prior to subsequent working. The present invention involves

5 a technique for inducing heterogeneities into the structure in such a fashion that the structures can be transformed into a homogeneous mixture of very uniform discrete particles.

The product of the present process is a metal composition having a uniform, fine grained microstructure consisting

10 of spheroidal particles engulfed in a solidified liquid phase. The microstructure is more uniform and the particles are generally rounder and of smaller size than comparable

alloys of the prior art. In the case of aluminum and virtually every other alloy tested, these particles are less than 30μ in

15 diameter.

The process of the invention has a number of very significant advantages. Casting of the starting billet may be carried out in a single convenient diameter, e.g. 152.4 mm,

20 at one location and reduced to any desirable smaller diameter at the same or a second location using conventional extrusion equipment and technology. The process permits removal of any dendritic exterior skin on the starting billet as part of normal practice prior to extrusion so that the extruded billet exhibits no skin effect. Moreover, the
25 process produces a considerable refinement of the microstructure of the final product, including its size, shape and distribution relative to the starting billet microstructure.

In the practice of the present process, a directional

grain structure is produced by hot working a metal composition, as by extrusion, rolling, forging, swaging or other means, at a temperature below the solidus temperature. By hot working is meant any process which deforms a metal or alloy between the recrystallization temperature (typically, $.7T_{\text{solidus Kelvin}}$) and the solidus temperature (T_{solidus}), such that it produces a striated or directional grain structure. According to a preferred embodiment of the invention, the directional grain structure is produced by extrusion. The extrusion ratio should normally be greater than 10/1 to produce the desired directional grain structure and may range as high as economically practical. We have found useful extrusion ratios frequently range from about 19/1 to about 60/1.

A critical level of strain must be introduced into the metal or alloy either concurrently with and as an integral part of the hot working step, or as a separate step subsequent to hot working and prior to heating to above the solidus temperature. Strain is introduced integral with the hot working operation, for example, by an in-line straightening operation, by rapid chilling of the hot worked material to introduce thermal strains or by extruding at lower temperatures such as to leave residual strains in the extruded product. Lower extrusion or other hot working temperatures tend to leave higher residual strains in the extrusion since the extrusion pressures go up as the temperatures go down, i.e. more energy is used up by the extrusion process. As a separate step, strain is introduced by cold working. Cold working operations found to be effective include drawing, swaging, rolling and compression

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or upsetting. Strain level is meant to represent any residual strain remaining within a grain after the deformation process is completed. The actual strain level will vary with the specific metal or alloy and with the type and conditions of hot working. In the case of extruded aluminum alloy, the strain level should be equivalent to at least a 12% cold worked alloy. In general, the level of strain can be determined empirically by determining whether, after heating to above the solidus temperature, the partially solid, partially liquid mixture comprises uniform discrete spheroidal solid particles contained within a lower melting matrix composition. Alloys, in which the directional grain structure is produced by hot working and particularly by extrusion, and which are separately cold worked, have been found to possess a particularly improved uniform, fine grained microstructure unavailable by other processes.

Upon completion of hot working and any required cold working, the alloy is then reheated to a temperature above the solidus and below the liquidus. The specific temperature is generally such as to produce a 0.05 to 0.8 volume fraction liquid, preferably at least 0.10 volume fraction liquid and in most cases a 0.15 to 0.5 volume fraction liquid. The reheated alloy may then be solidified and again reheated for shaping in a partially solid, partially liquid condition or the shaping step may be integral with the original reheat of the alloy to a partially solid, partially liquid state. The second reheat of the alloy may be to a higher fraction solid than the first reheat, but it is preferable not more than 0.20 fraction solid greater.

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In the preferred practice of the invention, the alloy is heated to a semi-solid state and shaped at the same time in a press forging operation. In such a process, the alloy charge is heated to the requisite partially solid, partially liquid temperature, placed in a die cavity and shaped under pressure. Both shaping and solidification times are extremely short and pressures are comparatively low. This press forging process is more completely disclosed in German application DE-OS 29 29 812 and DE-OS 29 29 845, the disclosure of which is hereby incorporated by reference. Other semi-solid forming processes which may be used are die casting, semi-solid extrusion and related shaping techniques.

Figure 1 is a typical time-temperature profile of a process in accordance with the invention. The vertical axis is temperature; the horizontal axis is time. The graph is intended to graphically portray a relative time-temperature relationship rather than set forth precise values. As can be seen from the graph, a metal is melted and solidified to form a cast billet, either dendritic or non-dendritic. The cast billet is preheated, e.g. approximately 30 minutes for a typical aluminum casting alloy, to above the recrystallization temperature, extruded and quenched to produce a solid metal composition having a directional grain structure. The extruded metal composition is then cold worked at room temperature to introduce a proper level of strain. It is then reheated above the solidus temperature, e.g. about 100 seconds for a typical aluminum alloy, to a semi-solid condition and rapidly quenched.

The starting material for practice of the present process may be a dendritic metal or alloy of the type conventionally cast into billets or a non-dendritic metal or alloy of the type in which a billet has been vigorously agitated during freezing in accordance with the teachings of the aforementioned U.S. patent 3,948,650. Such agitation produces a so-called slurry cast structure, that is one having discrete, degenerate dendritic particles within a lower melting matrix. Copending application S.N. 363,621 filed March 30, 1982, is directed to a process in which the starting material is a billet having a slurry cast structure in which the slurry cast structure is rehabilitated by heating to a semi-solid state. The disclosure of said copending application is hereby incorporated by reference. Billets which have been produced under conditions of vigorous agitation may be produced by the continuous direct chill casting process set forth in published British patent application 2,042,306A, the disclosure of which is also hereby incorporated by reference. In that application, molten metal is cooled while it is vigorously agitated in a rotating magnetic field. The process is continuous and produces continuous lengths of billets having a discrete degenerate dendritic structure. Billets are referred to below as billets which have been chill cast under a shearing environment during solidification to distinguish those which have been vigorously agitated from those which have not.

The microstructure of non-dendritic compositions produced in accordance with the aforementioned U.S. patent

3,948,650 and which is also produced in accordance with the process of the present invention may be variously described as comprising discrete spheroidal particles contained within a matrix composition having a lower melting point or, alternatively, as discrete primary phase particles enveloped by a solute-rich matrix. Such a structure will hereinafter be described in accordance with the first-mentioned description, but it should be understood that the various descriptions are essentially alternative ways of describing the same micro-structure.

The following examples are illustrative of the practice of the invention. Unless otherwise indicated, all parts and percentages are by weight except for fraction solids which are by volume.

Example 1

An aluminum casting alloy (Aluminum Association Alloy 357) was direct chill cast without shearing to a 152.4 mm diameter. Figure 2 is a micrograph of a crosssection of the direct chill cast bar in which its dendritic structure is apparent. The alloy had the following percent composition:

Si	7.0	Zn	.02
Cu	.010	Ti	.10
Mn	.004	Al	Remainder
Mg	.50		

A section of the cast bar was preheated to 380°C in less than 1/2 hour and extruded at a 50/1 ratio into a 22.2 mm diameter rod. Extrusion pressure was 67,000 psi. The rod exited at 7.62 m/minute and at 460°C and was fan quenched. The extruded bar was stretched straight (approximately 1% permanent set) to introduce strain into the bar as an integral step of the extrusion process. Figure 3 is a photomicrograph of a longitudinal section of the extruded stretched bar. Its directional grain structure is very evident. The extruded samples were then inductively reheated in a 3,000 Hz field at 6.75 kW in a 50.8 mm / 152.4 mm ID coil by / long for 100±5 seconds to a .7-.9 fraction solid and immediately water quenched to 24°C. These quenched samples were metallographically examined for particle size and shape. Figure 4 is a micrograph of a crosssection of the reheated and quenched sample. Fig. 4 demonstrates the dramatic refinement of the microstructure obtained over that of the starting billet (Fig. 2). It further demonstrates that the severely worked microstructure of the extruded

section can be converted to a slurry microstructure by heating to a 0.1 or higher fraction liquid.

Example 2

5 An aluminum casting alloy (Aluminum Association Alloy 357) was cast as in Example 1, preheated to 380°C within 1/2 hour and extruded into 31.75 mm diameter rod. The extrusion pressure was 9840 bar. The rod exited at 4.27 m/min-
10 ute and 500°C and was fan quenched. The extruded bar was stretched straight approximately 1% permanent set. Portions of the rod were then drawn 36% to 25.4 mm diameter. Samples were taken of the as-extruded and drawn material and inductively reheated and press forged as in Example 1 but this time into a 1.27 mm wall cup. Figure 5 is a representative micrograph
15 of a section through the final product again showing a uniform, fine grained "slurry-type" microstructure.

Example 3

20 An aluminum wrought alloy (Aluminum Association Alloy 2024) was direct chill cast, homogenized (to reduce extrusion pressure and tendency to hot tear during hot working) and extruded to a 25.4 mm diameter. The alloy had the following composition:

25	Cu	4.4
	Mn	.6
	Mg	1.5
	Al	Remainder

Samples of the as-extruded bars were reheated as in Example 1 while other samples of the extruded bars were compressed 29%

and reheated. Figure 6 is a representative micrograph of the final reheated but not cold worked samples. Figure 7 is a representative micrograph of the cold worked samples. It is apparent that the cold worked samples had a considerably more refined microstructure than the sample which had been reheated without cold work.

Example 4

Example 3 was repeated with an aluminum wrought alloy (Aluminum Association Alloy 6061) having the following composition:

Si	.6
Cu	.28
Mg	1.0
Cr	.2
Al	Remainder

Again micrographs were made of samples which were extruded and reheated and samples which were extruded, compressed 29% and reheated. Microstructure differences were as set forth in Example 3 and as illustrated by Figs. 6 and 7.

Example 5

Example 3 was again repeated with an aluminum wrought alloy (Aluminum Association Alloy 6262) having the following composition:

Si	.6	Zn	2.0
Cu	.28	Pb	.6
Mg	1.0	Bi	.6
Cr	.09	Al	Remainder

Comparative results were as set forth in Examples 3 and 4.

Example 6

Example 5 was again repeated with an aluminum wrought alloy (Aluminum Association Alloy 7075) having the following composition:

5	Cu	1.6	Zn	5.6
	Mg	2.5	Al	Remainder
	Cr	.23		

Results were as set forth in Examples 3-5.

Example 7

10 An aluminum alloy (Aluminum Association Alloy 357) was direct chill cast under a shearing environment to a 152.4 mm diameter. The alloy had the following percent composition:

	Si	7.0	Zn	.02
	Cu	.010	Ti	.10
15	Mn	.004	Al	Remainder
	Mg	.30		

A $\frac{559}{1}$ mm length was preheated to 520°C in less than 1/2 hour and extruded into a $\frac{22.23}{1}$ mm diameter rod. Extrusion pressure was 7031 bar. The rod exited at 7.3m/minute and at 520°C and was fan quenched. $\frac{25.4}{1}$ mm sections were then axially compressed at room temperature between two parallel plates so that the length was reduced 5, 10, and 16%. Samples then were taken of the as-extruded and the compressed sections and inductively reheated in a 3,000 Hz field at 6.75 kW in a $\frac{50.8}{1}$ mm ID coil by 152.4 mm long for 100±5 seconds to a .7-.9 fraction solid and immediately water quenched to 24°C. These quenched samples were metallographically examined for particle size and shape.

A 35 gram ^{25.4 mm} / section of the extruded billet was then axially compressed 25% and press forged into a threaded plug in accordance with the process of the aforementioned copending application S.N. 290,217 in a partially solid, partially liquid condition. Reheat time was 50 seconds, fraction solid was 0.85, dwell time was 0.5 seconds and pressure was 1054 bar with respect to atmosphere.

Photomicrographs at various stages of the process were taken. The starting ^{152.4 mm} / diameter billet exhibited particles of approximately 100 microns diameter. The extruded billet showed a directional grain microstructure in which the grains were very elongated. Micrographs of the center section of reheated billets, which were as-extruded and compressed 5, 10 and 16% respectively, showed that particle size and shape continued to improve as the strain was increased, particularly as strain was increased over 10%. The microstructure of a sample which was compressed 25% and press. forged into a threaded plug showed much finer scale microstructure and more uniform shape and distribution of the grains in the final product as compared with the starting billet. It also showed the remarkable influence of the residual strain upon the reheated grain structure of the extruded product.

Example 8

The aluminum casting alloy of Example 7 was direct chill cast as in that example to a ^{152.4 mm} / diameter billet. A 559 mm

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section was preheated within 1/2 hour to 330°C (much lower than Example 1) and extruded into a 28.58 mm diameter rod. Extrusion pressures for this rod were 31639 bar (much greater than Example 1). The rod exited at 7.01 m per minute 490 °C and was fan quenched. Samples were inductively reheated to a .7-.9 fraction solid as in Example 7 and water quenched. These quenches were metallographically examined for particle size and shape and found to be similar to the reheated, compressed 25% and press forged sample of Example 7. In this extrusion, the combination of low preheat T° (330°C) and fan cooling produced suitable residual strain in the extrusion.

Example 9

A copper wrought alloy C544 of 4%Zn, 4%Sn, 4%Pb, balance copper, was extruded to produce a directional grain structure and cold reduced 35% to a ^{25.4 mm} / diameter. Samples of the as-extruded bars were reheated using the procedure of Example 1 but for longer times, typically 200 seconds, in order to produce the partially solid, partially liquid structure and press forged into cams for use in water pumps. Fig. 8 is a micrograph of a crosssection of the press forged final product.

Example 10

Copper wrought alloy C360 containing 3.0% lead, 35.5 zinc, balance copper, was extruded and then cold reduced approximately 18% to a ^{25.4 mm} / diameter. Samples of the cold worked extrusion were reheated as in Example 1. Micrographs of

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crosssections of the final reheated alloy showed a microstructure very similar to that of Fig. 8.

Examples 11-13

Three additional copper alloys were hot worked to a small diameter substantially as described in the previous examples. Example 11 was hot worked by extrusion and cold worked an additional 50%. Example 12 was also hot worked by extrusion and cold worked an additional 29%. Example 13 was also hot worked by extrusion and cold worked an additional 20%, and then cold worked approximately 20-25%. Example 11

was alloy C110 which is 99.9% copper, balance oxygen and thus represents an almost pure metal. Example 12 was alloy C187 which is 99% copper and 1% lead. Example 13 was alloy C360 which is 60% copper, 3% lead, balance zinc.

Micrographs of crosssections of the final reheated and press forged or reheated and quenched alloys are shown in Fig. 9 (Example 11), Fig. 10 (Example 12) and Fig. 11 (Example 13).

The alloys of these three examples were reheated to a temperature of about 1070°C, 980°C and 890°C respectively.

The microstructures shown in these micrographs show substantial grain refinement relative to comparable microstructures of alloys produced by vigorous agitation of the alloy while in a semi-solid condition.

Example 14

Magnesium alloy A231B (4%Al, 1% Zn, 0.2% Mn, balance Mg) was prepared and hot worked as set forth in preceding Examples 11-13 and the bars were then subjected to an additional 12% compressive cold work. Fig. 12 shows the

microstructure of a crosssection of the final alloy reheated to about 610°C and quenched. Again, the structure is superior in both particle size and shape to the most closely analogous alloys of the prior art - comparable magnesium alloys prepared by vigorous agitation during freezing of the alloy while in semi-solid condition.

Example 15

Stainless steel alloy 316 (17% Ni, 12% Cr, 2.5% Mo, balance Fe) was hot worked by hot rolling and cold worked an additional 40%. Fig. 13 is a micrograph of a crosssection of the alloy reheated to about 1380°C and quenched. Particle sizes are considerably finer and more uniform than comparable particles of vigorously agitated stainless steel alloys.

Example 16

A stainless steel alloy 440C (.95-1.2%C, 16-18% Cr, Max 1% Mn, Max. 1%Si, Max. .75% Mo, balance Fe) was cast, hot rolled and cold worked an additional 20% by cold compression. The microstructure of the alloy reheated to about 1390°C and quenched is shown in Fig. 14.

While the foregoing examples have demonstrated practice of the process with a variety of aluminum, copper, magnesium and ferrous alloys, the process is applicable to other metals and metal alloys as long as the metal is capable of forming a two-phase system having solid particles in a lower melting matrix phase. Representative additional alloys which may be used are those of nickel, cobalt, lead, zinc and titanium. The alloys may be so-called casting alloys such as aluminum alloys 356 and 357 or wrought alloys such as aluminum alloys 6061, 2024 and 7075 and copper alloys C544 and C360.

WE CLAIM:

1. A metal alloy composition characterized by having a uniform, fine grained microstructure comprising uniform discrete spheroidal particles contained within a lower melting matrix, said alloy being a magnesium, ferrous or copper alloy prepared by

producing a solid metal alloy composition having an essentially directional grain structure,

heating said directional grain composition to a temperature above the solidus and below the liquidus to produce a partially solid, partially liquid mixture containing at least 0.05 volume fraction liquid, said composition prior to heating having a strain level introduced such that upon heating, the mixture comprises uniform discrete spheroidal particles contained within a matrix composition having a lower melting point than said particles, and

solidifying said heated alloy composition.

2. The metal composition of claim 1 in which the ferrous alloy is stainless steel.

3. The metal composition of claim 1 in which the directional grain structure is produced by hot working said metal alloy.

4. The metal composition of claim 3 in which the hot working is performed by extruding said metal alloy.

5. The metal composition of claim 3 in which the composition is cold worked subsequent to production of the directional grain structure by hot working.

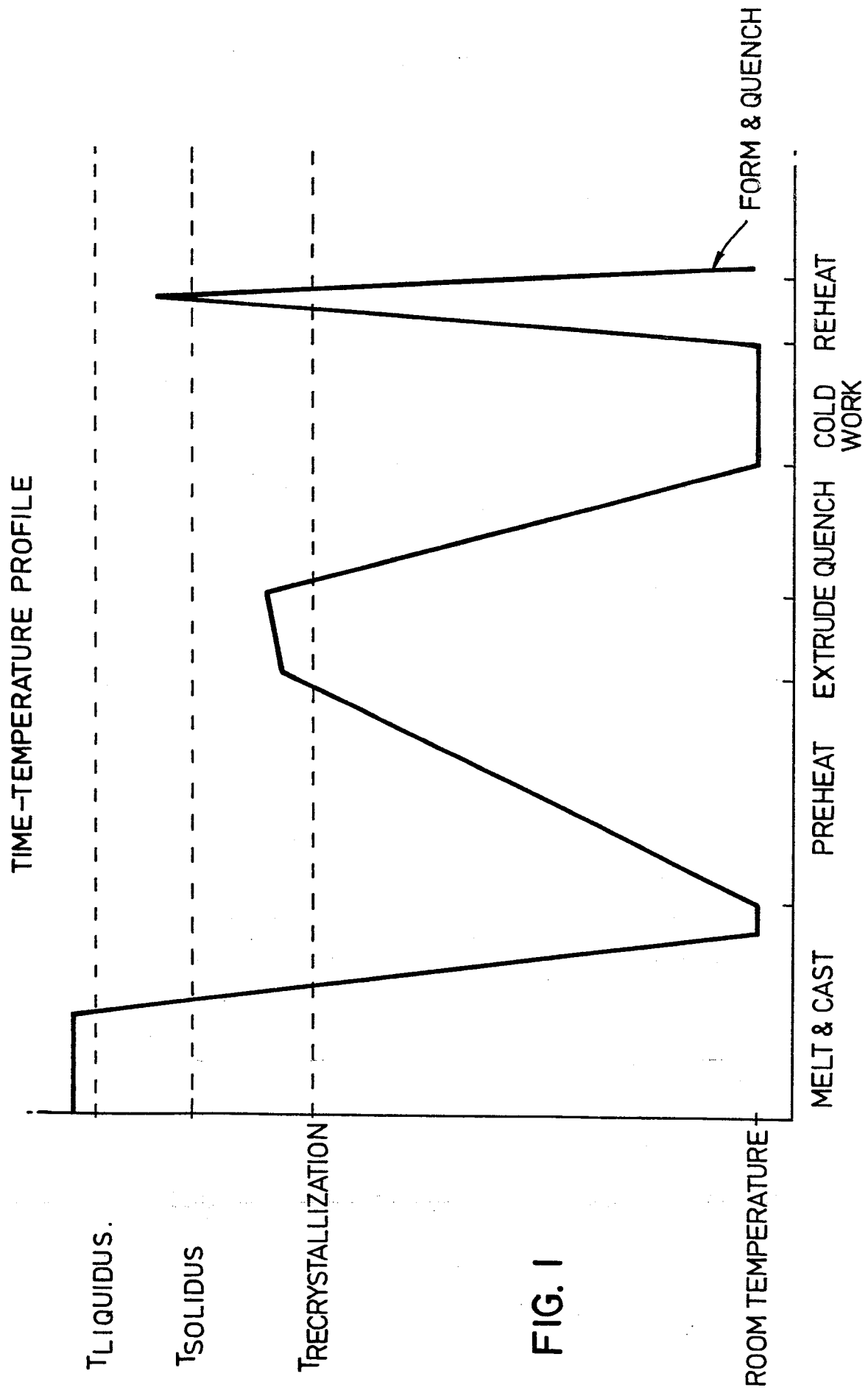


FIG. 1

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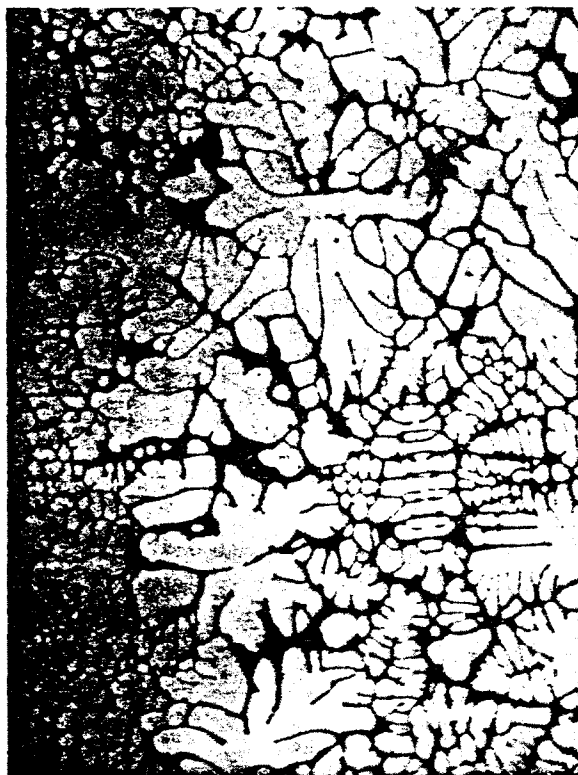


FIG. 2



FIG. 3

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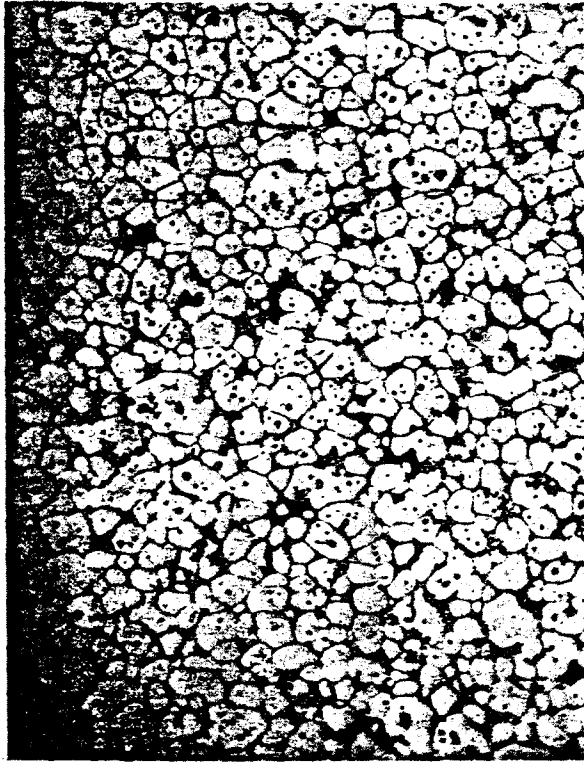


FIG. 4

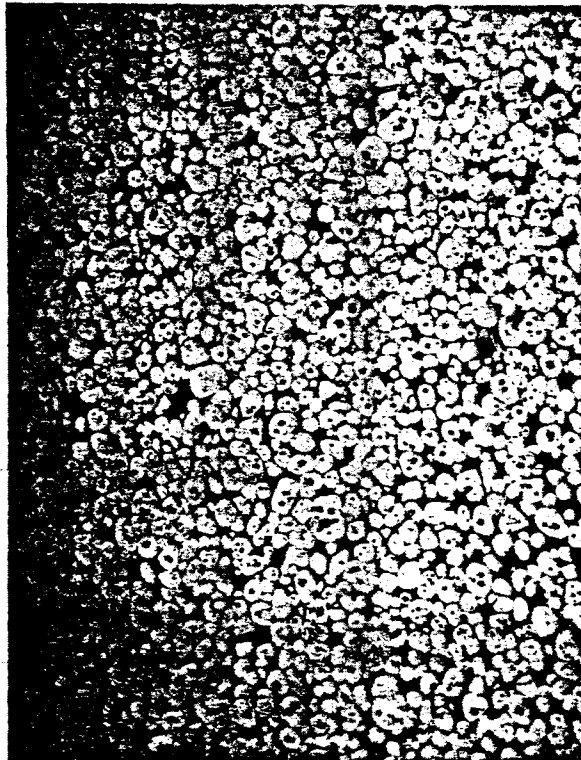


FIG. 5

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FIG. 6



FIG. 7

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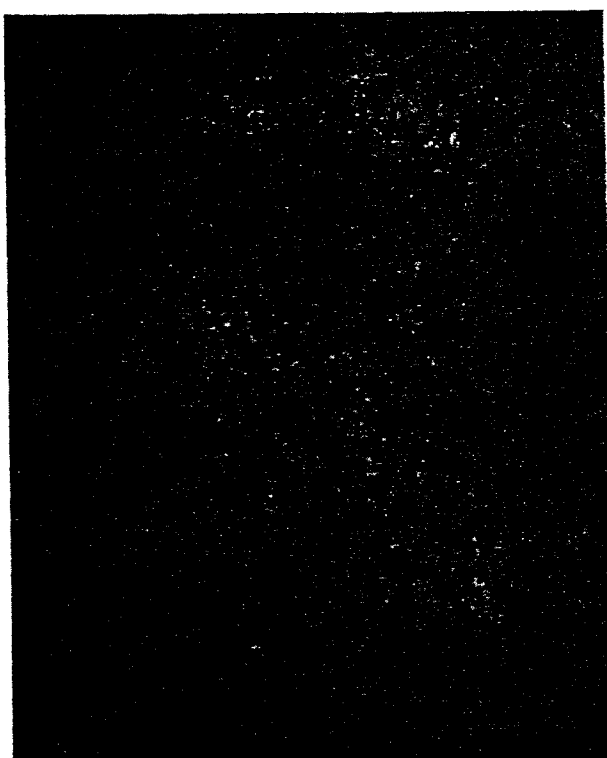
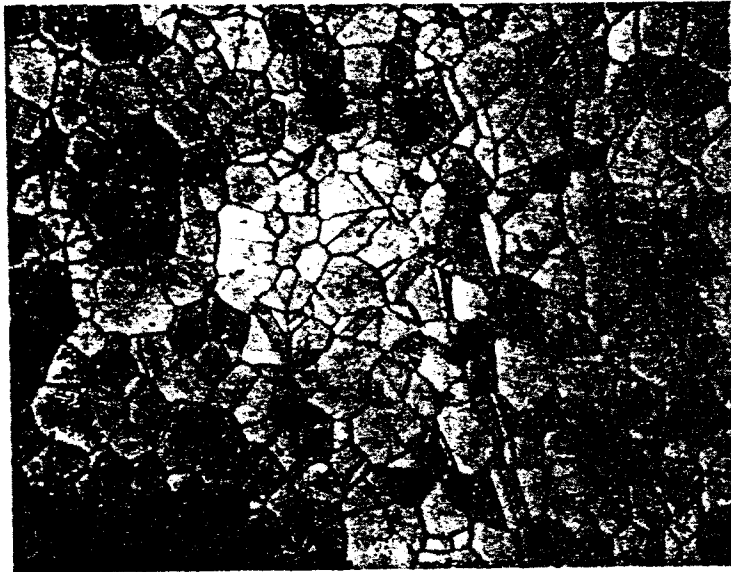


FIG. 8

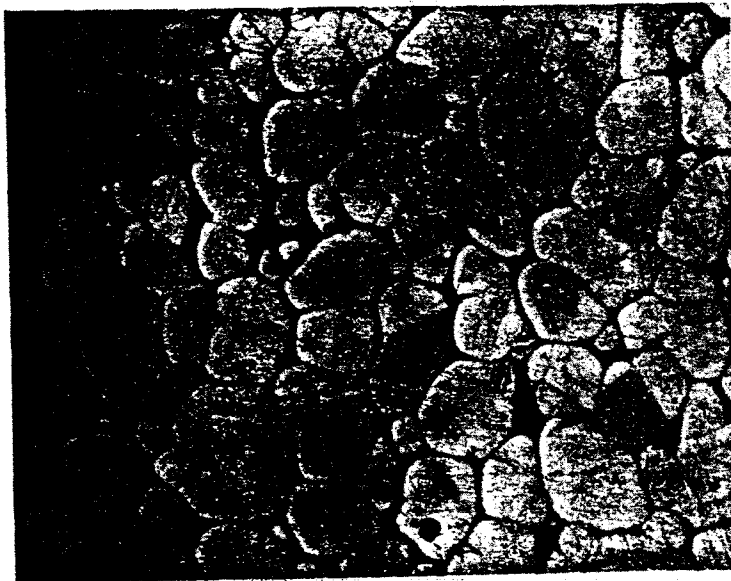
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ALLOY C187

FIG. 10

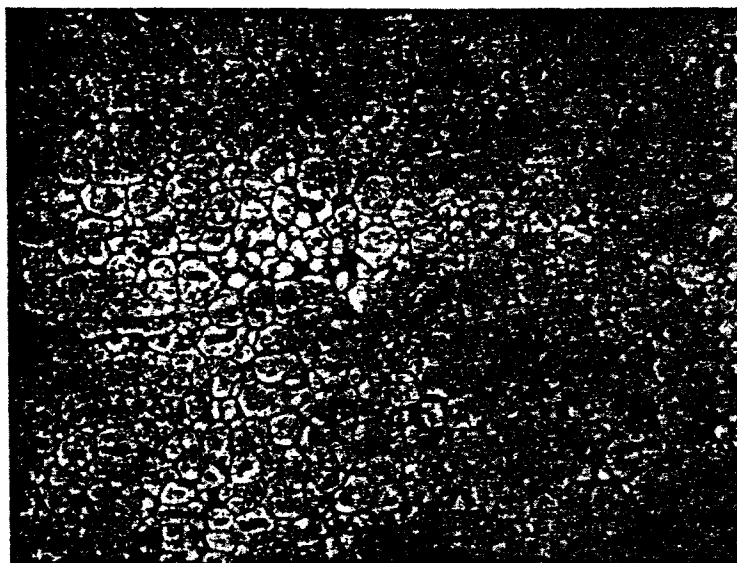


ALLOY C110

FIG. 9

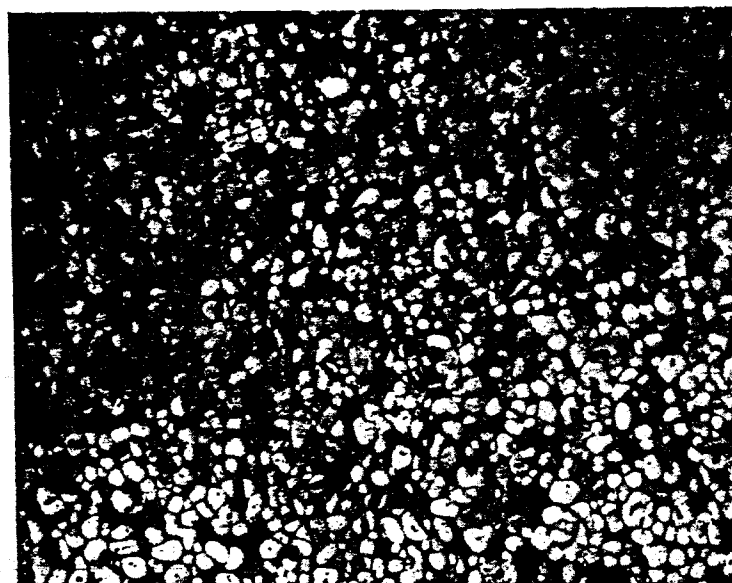
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ALLOY AZ 31-B

FIG. 12

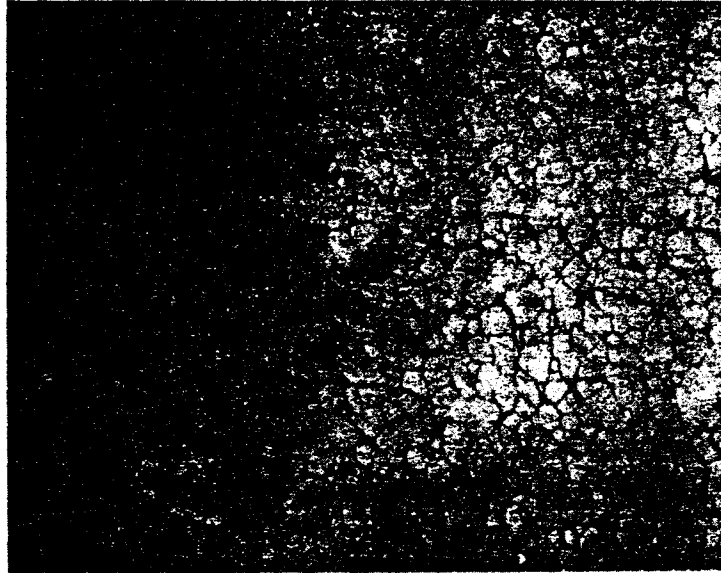


ALLOY C360

FIG. 11

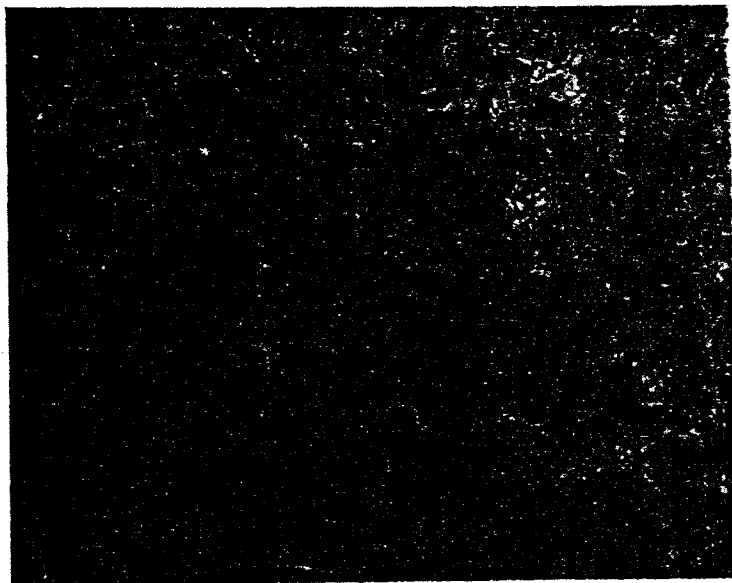
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ALLOY 440C

FIG. 14



ALLOY 316

FIG. 13

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
Y	FR-A-2 266 749 (SOCIETE DE VENTE DE L'ALUMINIUM PECHINEY) *Claims 1,3,5,6*	1	C 22 C 1/00
Y	FR-A-2 266 748 (SOCIETE DE VENTE DE L'ALUMINIUM PECHINEY) *Claims 1,3,5-7*	1	
X,P	EP-A-0 090 253 (ITT INDUSTRIES) *Claim 1; page 16, lines 3-11*	1	
X,P	*Claims 2,3*	3,4	
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)
			C 22 C 1/00 C 22 F C 21 D 8/00
The present search report has been drawn up for all claims			
Place of search THE HAGUE	Date of completion of the search 19-12-1984	Examiner LIPPENS M.H.	
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			