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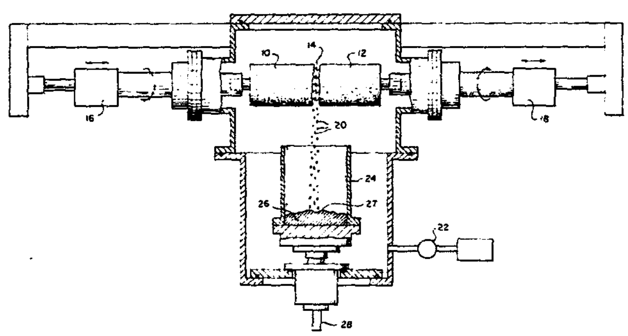
(54) **Alloy remelting process.**

(57) A remelting process for producing an alloyed ingot having a substantially homogeneous distribution of components which tend to form band macrosegregations, comprises the steps of:

providing at least one consumable ingot (10, 12) above a mould (24) the ingot (10, 12) having a composition of the ingot (26) to be produced in the mould (24), the composition having at least two components characterized by their substantially different specific gravities and substantially different melting point;

heating a portion of the ingot (10, 12) to a temperature between the solidus and liquidus temperatures of the alloyed composition for forming partially solidified droplets which fall to a mushy body (27) of droplets on the ingot (26) in the mould (24); and

solidifying the remaining liquid in the mushy body.



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ALLOY REMELTING PROCESS

The invention relates to a remelting process for producing an alloyed ingot having a substantially homogeneous distribution of components (i.e., chemical elements) which would otherwise tend to segregate in macroscopic areas of the ingot during conventional solidification processes.

Complex alloys and alloys having reactive elements are commonly produced by primary melting processes such as vacuum induction melting (VIM) to obtain excellent melt composition control and also a high degree of purity. The molten metal is poured from the VIM crucible into large moulds in a relatively short time. Thus, the majority of the solidification occurs after the mould is full. Many of these alloys have wide solidification ranges (i.e., the difference between the solidus and and liquidus temperatures) and their solutes have different solubilities in the solid vs. liquid solvent. Thus, the first metal to solidify may be vastly different in chemical composition than the last metal to solidify and gross ingot chemical segregation results.

The above macrosegregation (and also structural defects such as shrinkage voids and porosity) that result from primary melting and casting processes require that the ingots be refined by secondary remelting techniques such as vacuum arc remelting (VAR) and electroslag remelting (ESR) in order to produce ingots having the desired chemical and physical homogeneity and structure. The VIM castings are remelted in these processes as consumable electrodes into a cold crucible. (The chemical homogeneity of the resulting ingot is improved by zone refining, whereas the molten metal pool size is controlled and limited during the remelting process). As the metal solidifies in these processes, solute is rejected into the molten pool ahead of the solid-liquid interface, while more liquid is added to the molten pool from the melting electrode. Thus, the composition of the liquid ahead of the solid-liquid interface is nearly equivalent to the exact electrode composition during the entire remelting process and gross ingot segregation is reduced. The progressive directional solidification that occurs in the cold crucible also improves the physical homogeneity of the ingot by reducing the areas of porosity caused by material contraction during solidification.

Other types of macrosegregation often result from secondary remelting complex and reactive alloys by VAR and ESR techniques. Solidification occurs predominantly by columnar dendritic growth. These dendrites are fingerlike solid projections growing from the solidified ingot into the liquid. The volume of material where liquid and solid co-exist at a given time during solidification is termed the "mushy zone". The movement of liquid or solid within the mushy zone can cause areas of localized segregation by entrapment of the liquid or solid between the growing dendrites.[1] Liquid metal movement may result from thermal contractions, solidifications shrinkage and density differences in the liquid. Unstable interdentritic fluid flow during VAR and ESR processing often results in channel type segregates, called "freckles", and solute layering or "banding" in the ingot. Previous attempts to control the liquid flow during secondary remelting, by means such as electromagnetic stirring during vacuum arc remelting, in order to minimize macrosegregation, have failed and have often resulted in increased ingot segregation.[2/3]

Materials such as uranium-niobium alloys have a greater propensity to segregate in small diameter (203.2mm) (8") VAR ingots due to their substantially different atomic weights and melting points. The density differences of the liquid and its unstable flow in the mushy zone create banding and gross ingot segregation. (Banding has been somewhat arbitrarily defined by Jessen in this system as a layered chemical inhomogeneity that follows the solidifying melt pool pattern with gradients of ± 1 w/o Nb over 5-10 mm which segregation is seen on radiographs as continuous gradients of niobium (0.1 w/o Nb bottom-to-top and 0.14 w/o Nb edge to centre). Flemings et al.[1] has shown for unidirectional solidification that qualitative relationship exists between fluid flow and the thermal environment between growing dendrites and the thermal environment between growing dendrites in the mushy zone

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1 Flemings, Solid Processing
2 Trans. Int. Vac. Met.Conf. 1967, pg.695 Buehl & McCauly.
3 " " " - " " 1968, pg 599 J.W. Trautman.

Abdrupt changes in the heat transfer rate result in solute-rich or sulute-poor bands. Zanner et al.[4] are currently analyzing the vacuum arc remelting of uranium niobium alloys by numerical and experimental methods in order to reduce macrosegregation by improved process control. The inherent complexity of the physics of the VAR process in having a consumable electrode remelting by an electric arc in intimate contact with a superheated liquid-metal pool limits the degree of process control that can be achieved and also the degree of macrosegregation control.

The present invention provides a remelting process for producing an alloyed ingot having a substantially homogeneous distribution of components which tend to form band macro-segregations comprising the steps of:

providing at least one consumable ingot above a mould, the ingot having a composition of the ingot to be produced in the mould, the composition having at least two components characterized by their substantially different specific gravities and substantially different melting points;

heating a portion of the ingot to a temperature between the solidus and liquidus temperatures of the alloyed composition for forming partially solidified droplets which fall to a mushy body of droplets on the ingot in the mould; and

solidifying the remaining liquid in the mushy body.

The remelting method of the present invention produces an alloyed ingot having a substantially homogeneous distribution of alloying elements (such as uranium-niobium or copper-

[4] Zanner et al., "Computational and Experimental Analysis of a U-6 w/o Nb Vacuum Consumable Arc Remelted Ingot".

aluminum alloys and the like) which tend to form "banding" (and "freckle") macrosegregations when remelted by VAR and ESR techniques. In contrast with prior art methods for solidifying these alloys from a circulating superheated pool over columnar dendrites growing from the ingot surface, the method of the present invention depends upon the solidification of most of the liquid by homogeneous nucleation, creating a mushy zone of independent solid nucleii with liquid metal that has a viscous consistency, like a milk-shake, which allows negligible if any circulation of the liquid. There is substantially no circulation of liquid and thus no sudden changes in convection of the liquid in the mushy zone because the homogeneous mixture of solid particles and liquid effectively retards the movement of liquid during solidification. Solidification occurs before liquid can segregate because the solid particles throughout the body absorb at least a portion of the heat of fusion so that no area of the body is superheated, and there are no abrupt changes in the heat transfer rate. Also, the solidification proceeds more rapidly than VAR and ESR since there is no superheated liquid pool above the mushy zone. The droplets falling off the melting ingot onto the ingot contain solid nucelii and collectively form the mushy zone on the ingots themselves.

For the purposes of definition, the term "mushy" as used in this patent application describes an alloy at a temperature between the liquidus and the solidus temperatures. These temperatures can be accurately determined by differential thermal analysis; see, e.g., William J. Boesch et al.,

<u>Differential Thermal Analysis Detect Superalloy Reactions</u>, METALS PROGRESS, October 1975, for an article describing the technique.

The process of the present invention may be particularly useful in connection with systems which are characterized by inverse segregation, i.e., where the one component having a relatively high melting point also has a relatively low specific gravity or where the one component having a relatively low melting point also has a relatively high specific gravity.

The present invention will be more completely understood with reference to the preferred embodiment which is illustrated by the accompanying schematic drawings.

As is shown by the drawing at least one consumable electrode 10 is provided in a controlled atmosphere furnace above a mould (or crucible) 24. The preferred practice and figure are described in more complete detail in U.S. Patent No. 4,261,412, which is assigned to be Applicants of the present application and is hereby incorporated by reference. The consumable ingot 10 may be prealloyed and have a nominal composition of the ingot to be cast or, less commonly, the ingot may comprise alternate strips of the metals which make up the alloy system. Also, as is shown in the figure there may be two ingots both of which may be consumable. Also there may be multiple pairs of ingots consumed simultaneously. In addition to arc processes, the ingot or ingots may also be melted (as described below) by electron beams, plasma arc and like processes.

A 203.2mm (8 inch) diameter VIM prealloyed 4.3 w/o copper-aluminium ingot was melted in accordance with the present invention into a 152.4mm (6 inch) diameter by 355.6mm (14 inches) long remelted ingot in a furnace depicted in the figure. The ingot was melted at a rate of 3.375.kg (7.5 pounds) per minute at a nominal current of 4000 amps and voltage of 26 to 28 volts. Seven Type K thermocouples at 50.8mm (2 inch) intervals along the sidewall of the mould 24 measured the temperature of the ingot being formed. In accordance with the practice of the present invention, a portion of the consumable ingot 10 was heated to a temperature between the solidus and liquidus temperatures of the nominal composition of the ingot to be formed in the mould below. A second ingot (in this case, a consumable ingot) 12 having the same composition was positioned near to the first ingot 10 to form a gap 14 between their adjacent ends. An electric arc was struck in the gap 14 between the adjacent ends of ingots 10 and 12. When the temperature of the ingots 10 and 12 exceeded the solidus temperature, partially solidified droplets 20 began to fall onto the ingot 26 in the mould 24 and form a mushy body 27. The thermocouples in the wall of the mould 24 indicated that the maximum temperature of the mushy body 27 on the 4.3 w/o copper-aluminium ingot 26 varied between 1189°F (642.8°C) to 1205°F (651.7°C). The solidus and liquidous temperatures of this composition was shown by differential thermal analysis to be 1117°F (602.8°C) and 1234°F (667.8°C) respectively. The phase diagram indicates that systems such as these copper-aluminium compositions (i.e., containing less than about 26 w/o copper) will tend to inversely segregate because the solidifying alloy tends to be lighter than the remaining liquid. The relative specific gravities of these components (copper 8.92 and aluminium 2.70) indicates that the segregations would tend to segregate as bands.

The mushy body 27 on the ingot 26 solidified without a liquid pool having formed. The 152.4mm (6 inch) diameter ingot was sectioned longitudinally and chemically etched for macrostructure inspection. The ingot had a uniform equidexed grain structure with the grain size being less than 1.5875 mm (1/16 inch) in diameter. There was no evidence of banding.

In the arc furnace depicted in the figure, the consumable ingots 10 and 12 are oscillated by any suitable means 16 and 18 to promote even burn-off and to prevent the partially solidified droplets from being significantly superheated. Also, the mould 24 itself may be of various forms and shapes depending upon the desired products, including forms and shapes of the final products as well as traditional intermediate forms such as billets. The figure depicts a rotatable mould driven by rotated shaft means 28 for achieving even distribution of droplets 20 across the ingot surface. The mould 24 may also have a means for lowering the ingot 26 as it forms. The furnance should have a controlled atmosphere, e.g., a vacuum created by any suitable means such as pump 22 as depicted.

Other banding macrosegregation sensitive systems such as uranium-columbium (such as the 6 w/o columbium-uranium alloy) and lithium-aluminium systems may be remelted into substantially homogeneous ingots in accordance with the present invention. In addition strengthening particles such as yttria or thora or fibres such as silicon carbide may be injected to the stream of droplets 20 as they fall to the mushy body.

CLAIMS

1. A remelting process for producing an alloyed ingot having a substantially homogeneous distribution of components which tend to form band macrosegregations characterised in that it comprises the steps of:-

providing at least one consumable ingot above a mould, the ingot having a composition of the ingot to be produced in the mould, the compositon having at least two components characterized by their substantially different specific gravities and substantially different melting points;

heating a portion of the ingot to a temperature between the solidus and liquidus temperatures of the alloyed composition for forming partially solidified droplets which fall to a mushy body of droplets on the ingot in the mould; and

solidifying the remaining liquid in the mushy body.

2. The process of claim 1, characterised in that a plurality of consumable ingots are heated above the mould.

3. The process of claim 1 or 2, characterised in that the ingot or ingots to be heated above the mould is or are first prealloyed.

4. The process of claim 1, 2 or 3, characterised in that the ingot or ingots is or are heated by an electric arc.

5. The process of claim 1, 2 or 3, characterised in that the ingot or ingots is or are heated by an electron beam.

6. The process of claim 1, 2 or 3 characterised in that the ingot or ingots is or are heated by a plasma arc.

7. The process of any one of the preceding claims, characterised in that the composition comprises at least two components, the first component having a melting point substantially greater than the melting point of the second component and the second component having a specific gravity substantially higher than the specific gravity of the first component.

8. The process of claim 7, characterised in that the melting point of the first component is at least 1000°C greater than the melting point of the second component and the specific gravity of the second component is at least twice the specific gravity of the first component.

9. The process of claim 8, characterised in that the first component is columbium.

10. The process of claim 8 or 9, characterised in that the second component is uranium.

11. The process of claim 10, characterised in that the composition comprises by weight substantially 6.0% of columbium in uranium.

12. The process of any one of claims 1 to 6 characterised in that the composition comprises at least two components, the first component having a melting point and a specific gravity which are substantially greater than the melting point and specific gravity of the second component.

13. The process of claim 12, characterised in that the melting point of the first component is at least 300°C greater than the melting point of the second component and the specific gravity of the first component is at least twice the

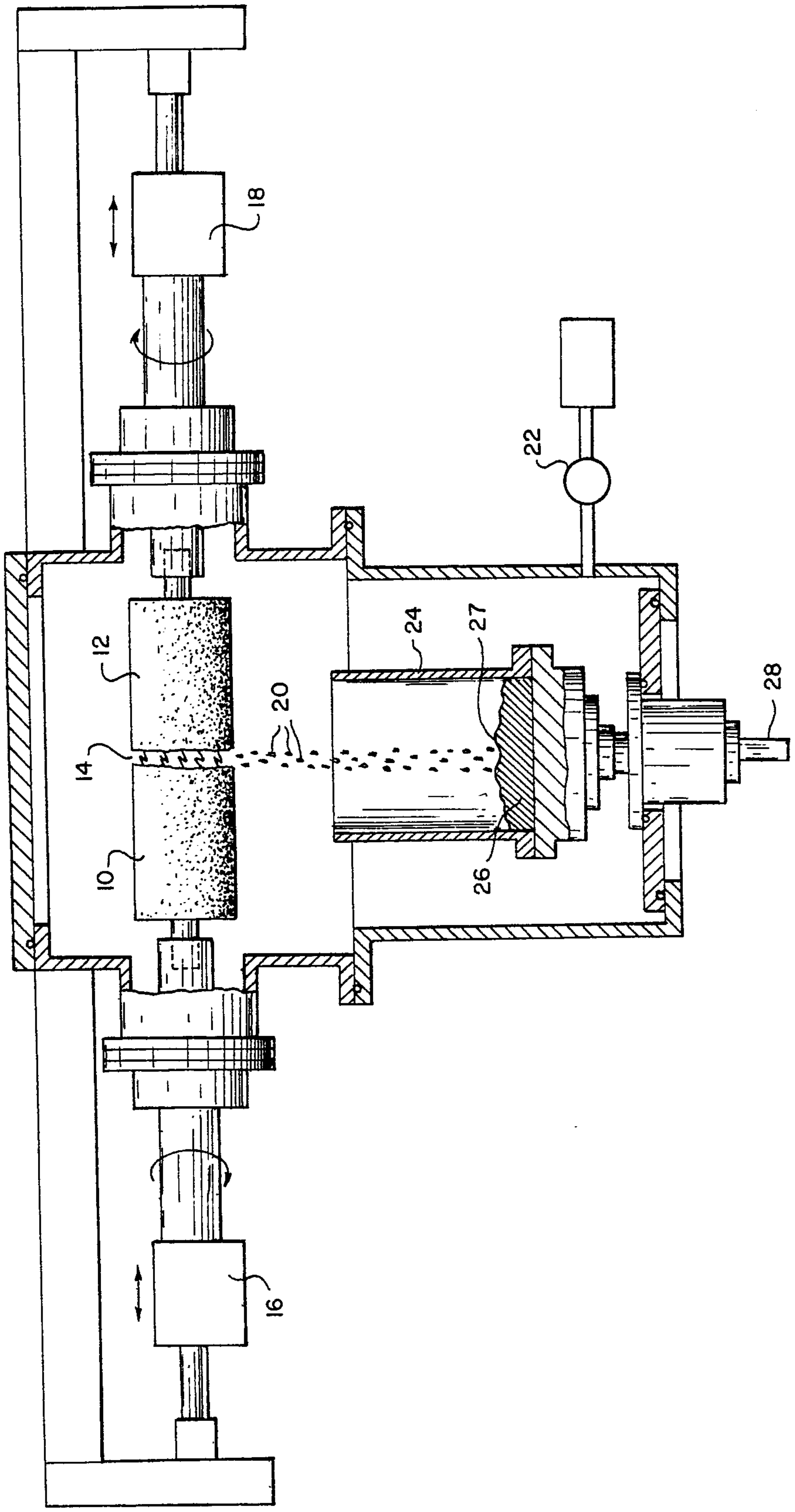
specific gravity of the second component.

14. The process of claim 12 or 13, characterised in that the first component is copper.

15. The process of claim 13 or 14, characterised in that the second component is aluminium.

16. The process of claim 15, characterised in that the composition comprises by weight substantially 4.3% copper in aluminium.

17. An alloyed ingot having a substantially homogeneous distribution of components which tend to form band macrosegregations when made by the process of any one of the preceding claims.



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EUROPEAN SEARCH REPORT

Application number

EP 82 30 4233

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. 3)
Y,D	GB-A-2 049 513 (SPEICAL METALS CORPORATION) *Claims 1-3,6,10,11,17; page 1, lines 6,7,39-45; page 2, lines 85-92; figures 1-3* & US - A - 4 261 412	1-3	C 23 B 9/20 C 22 C 1/02
Y	US-A-2 899 294 (W.J.SIEMONS) *Claims 1,2,4; figures 1,2*	1,6,9	
A	FR-A-1 187 024 (WESTINGHOUSE) *Claims; abstract a-b; page 3, right-hand column, paragraphes 4,5; figure 1*	1,11	
A	DE-C-1 213 874 (OSAMU MADONO) *Claim 1*	1-3,7, 12,13, 15,17	TECHNICAL FIELDS SEARCHED (Int. Cl. 3) C 22 C C 22 B B 22 D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	02-12-1982	RIES R

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
O : non-written disclosure
P : intermediate document

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