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ABSTRACT OF THE DISCLOSURE

A method of manufacturing lamellar composites by 15 means of directionally demixing solid materials having a eutectoid composition, or solid solutions. The starting materials must have a grain size of more than approximately 0.1 mm. The materials are directionally demixed while using a temperature gradient of at least 10° C. per cm. 20 which is passed through the body at a rate which is less than or equal to the maximum demixing rate.

The invention relates to a method of manufacturing 25 bodies of lamellar composites by directionally demixing solid single-phase materials which demix discontinuously in two or more phases upon cooling. These single-phase materials may have a eutectic composition or may consist of solid solutions. Lamellar composites are under- 30 stood to mean materials whose grains consist of at least two phases in which at least one of the phases in at least one direction has a dimension which is relatively much larger than the dimensions in the other directions. These materials thus comprise both composites whose grains are 35 built up from thin sheets of alternatively the one and the other phase and composites one phase of which consists of needles which are embedded in a different phase.

Discontinuous demixing, sometimes also referred to as 40 cellular demixing, is understood to mean that the new phases are produced by nucleation and growth from the grain boundaries at which the composition of the transformed matrix remains uniform except for the incoherent boundary of the growing cell. Methods of directionally 45 coagulating melts of mixtures of materials are known (see, for example, U.S. Pat. 3,124,452). Generally methods are used which are also employed in the manufacture of monocrystalline materials in which the molten material is allowed to cool off in a previously determined direction 50 such as, for example, in the method according to Bridgman. If in this manner mixtures of materials are treated which upon coagulation from the melt constitute at least two phases of lamellar geometry, then it is found afterwards that the lamellae in the coagulated material in all 55 grains have grown substantially perpendicularly onto the coagulation front. However, in their transverse cross-section the lamellae different grains are generally not in parallel. In "A Study of Directionally Transformed Pearlite" by B. L. Bramfitt and A. R. Marder, IMS Pro- 60 ceedings (1968) pages 43–55, the use of directional cooling is described for an iron-carbon alloy having a eutectic composition. However, pearlite obtained from austenite in this method does not have the structure as is obtained when directionally coagulating mixtures of materials. The 65 lamellae in the grains are substantially not parallel to the transformation direction. An object of the present invention is to provide a method of manufacturing bodies of lamellar composites by directionally demixing solid single-phase materials which demix discontinuously in two or 70 more phases upon cooling in such a manner that the lamellae in all grains are substantially parallel to the trans-

formation direction. It was found that this condition can be satisfied by a method which is characterized in that a single-phase material is directionally demixed whose grains have a diameter of not less than approximately 0.1 mm. while using a temperature gradient of at least 10° C. per cm. passed through the body at a rate which is less than or equal to the maximum rate of demixing. It was found that directional demixing by means of directional cooling cannot be compared without any objection with directional coagulation by means of directional cooling of melts of materials having eutectic compositions. For directional coagulation there applies that the square of the lamella distance is inversely proportional to the growth rate, at a constant growth rate in accordance with

$$\lambda^2 v = CD$$

wherein

λ=lamella distance
v=growth rate
C=a constant of the system concerned
D=diffusion constant

The following relationship was determined for eutectoid compositions in case of directional demixing:

$$\lambda^4 v = C$$

This proves that the methods used for directional coagulation cannot be used without difficulty when directionally demixing solid single-phase material having a eutectoid composition or of solid solutions. However, if one starts from grains having a diameter of at least 0.1 mm., the temperature gradient being equal to or larger than 10° C. per cm. and the growth by which the temperature gradient is moved through the body is smaller than or equal to the maximum rate of the transformation front, it is found that structures are obtained which are comparable with the structures obtained when directionally coagulating melts having a eutectic composition. The lamella distance obtained by the method according to the invention is, however, found to be considerably smaller than the lamella distance which is usualy found at corresponding growth rates when directionally coagulating eutectic melts.

The maximum rate of the transformation front is different for each system, but may be experimentally determined in a simple manner. It was found in practice that the maximum rate of the transformation front is greater as the eutectoid temperature or the saturation temperature is higher. Usually the maximum rate lies between

$$10^{-11} \cdot \text{ and } 10^{-12} \cdot T^4 \text{ cm. per hour}$$

wherein T is the eutectoid temperature in ° K. or the saturation temperature in ° K. Failure of the experiments which have become known in literature up till now is probably to be ascribed to the fact that one or more of the three mentioned criteria are not satisfied. No directional effect was found when, for example, the method was used on the systems: 28 at. percent Zn, remainder Fe (solid solution), grain size approximately 50 μm.; 25 at. percent Sn, remainder Fe, grain size between 10 and 30 μm. and 0.8% by weight of C, remainder Fe (after demixing pearlite), grain size approximately 20 μm. The method according to the invention may be performed by means of a technique which is comparable with the Bridgman technique in which the body is passed through a temperature gradient or in which the temperature gradient is moved relative to the body. In practice the temperature gradients to be used may be obtained with comparatively simple standard apparatus. Usually the external temperature gradient to be used is less than 450° C./cm. Generally there applies that the temperature gradient is chosen to be so much greater as the grain diameter is smaller. The method may be used for reinforcing articles, for ex-

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ample, tools. It is then possible to subject the articles to mechanical operations, for example, forging at the temperature above the transformation temperature (eutectoid temperature or the temperature at which saturation occurs) or at room temperature after previous tempering from a temperature above the transformation temperature and by subsequently giving the articles a greater strength by means of directional demixing. The method may alternatively be used to give articles certain magnetical, electrical and/or optical properties.

In order that the invention may be readily carried into effect, it will now be described in detail with reference to the few examples.

EXAMPLE I

A quartz tube having a length of 10 cms. and an internal diameter of 0.4 cm. and an external diameter of 0.54 cm. was filled with a molten alloy of nickel and indium (55.5 at. percent In, remainder Ni), melting point approximately 950° C., eutectoid temperature 770° C. The alloy was heated for two hours at 1050° C. and it was subsequently coagulated by decreasing the temperature to approximately 850° C. After this treatment the grains had a diameter of between 0.5 and 3 mms. The alloy-filled tube was subsequently passed through a temperature gradient of 65° C. per cm. at a constant rate of 0.1 cm. per hour (Bridgman technique). The lamella distance in the directionally demixed body was 0.7 μm. after finishing the treatment, at a rate of 1 cm. per hour: 0.4 μm. and at a rate of 7 cms. per hour: 0.2 μm. The lamellae in the grains were parallel to the temperature gradient. The lamellae consisted alternately of NiIn and Ni_2In_3.

EXAMPLE II

The method described in Example I was used on an alloy of copper and indium (20.2 at. percent In, remainder copper, melting point approximately 700° C., eutectoid temperature 574° C.). The alloy was heated for two hours at 850° C. and subsequently coagulated by decreasing the temperature to 650° C. The grains of the alloy had a diameter of between 1 and 10 mms. after 2 hours of heating at 650° C. in the quartz tube. The external temperature gradient was 65° C. per cm. A lamella distance of .038 μm. was obtained at a passing rate of 0.16 cm. per hour and a lamella distance of 0.21 μm. was obtained at a passing rate of 1.15 cm. per hour. The lamellae in the grains were parallel to the temperature gradient. The lamellae consisted alternatively of 29 at. percent In, remainder Cu and 11 at. percent In, remainder Cu. In one specimen which in addition to grains of more than 0.1 mm. also contained smaller grains (approximately 50 μm.) it was found that the lamellae in the smaller grains were not directed parallel to the temperature gradient in contrast with the grains of more than 0.1 mm. in which this was the case (temperature gradient approximately 30° C. per cm., passing rate 0.5 cm. per hour).

EXAMPLE III

The method described in Example I was used on an alloy of copper and aluminium (24 at. percent Al, remainder Cu, melting point 1100° C., eutectoid temperature 565° C.) which alloy was present in a tube of Al_2O_3. The alloy was passed through a temperature gradient immediately after coagulation. The grain diameter was between 0.5 and 1 mm. The temperature gradient was 100–150° C. per cm. At a passing rate of 0.2 cm. per hour a lamella distance of 0.25 μm. was obtained, and

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at a passing rate of 0.5 cm. per hour a lamella distance of 0.20 μm. was obtained. The lamellae consisted alternately of 19.6 at. percent Al, remainder Cu, and 30.3 at. percent Al, remainder Cu. At a passing rate of 0.2 cm. per hour and a temperature gradient of 50° C. per cm. a rod was obtained which in the direction parallel to the temperature gradient had a tensile strength of 113 kg. per sq. mm. The maximum tensile strength of non-directionally cooled copper-aluminium (24 at. percent Al, remainder Cu) is approximately 68 kg. per sq. mm., in accordance with recent literature.

EXAMPLE IV

The method described in Example I was used on an alloy of cobalt and silicon (25 at. percent Si, remainder Co, peritectic decomposition temperature 1219° C., eutectoid temperature 1170° C.). The temperature gradient was 100–300° C. per cm. At a rate of 0.05 cm. per hour the lamella distance was 1.4 μm., at a rate of 6 cm. per hour the lamella distance was 0.4 μm. The lamellae were parallel to the temperature gradient. The grain diameter was between 0.5 and 3 mms.

EXAMPLE V

The method according to Example I was used on a solid solution of tin in lead (16.6 at. percent Sn, remainder Pb). This solid solution is oversaturated at temperatures below 148° C. The grain diameter was between 0.5 and 1 mm., the temperature gradient was 100° C. per cm. At a rate of 0.05 cm. per hour a lamellar structure was obtained having a lamella distance of 0.9 μm., the lamellae were parallel to the temperature gradient.

What is claimed is:

1. A method of producing a composite of parallel oriented lamella shaped grains of two different metal phases, said method comprising, heating an alloy capable of separating discontinuously into at least two phases upon cooling, to a temperature above the melting point of the alloy, cooling said melted alloy to a temperature between the melting point of the alloy and the eutectoid temperature of the alloy to thereby coagulate the molten alloy and form grains having minimum diameters of 0.1 mm. and then further cooling the resultant metal grains to room temperature by unidirectionally decreasing the temperature of said grains at a rate of at least 10° C. per cm. and not greater than the maximum rate at which the separation of said alloy into two or more phases occurs.

2. The method of claim 1 wherein the rate at which the metal grains are unidirectionally cooled to room temperature is not greater than $10^{-11} \cdot T^4$ cm. per hour wherein T is the eutectoid or saturation temperature in ° K.

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