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METHOD OF PRODUCING DISPERSOID STRENGTHENED MATERIAL

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This invention relates to a method of producing improved metallic material. The invention relates particularly to a method of producing metallic sheet and strip material having improved strength properties. More particularly, the invention relates to a method of producing improved sheet and strip material by mechanical means.

Various and sundry methods have been employed in the past to manufacture metallic material in sheet and strip form and to improve the strength properties thereof. The prior art is replete with teachings of such methods and, in the main, these methods involve the reduction of blooms, billets, ingots, etc., by means of rolling mills. It is well known that a relationship of direct proportionality exists between strength and hardness values and that an increase in the one is accompanied by an increase in the other. Hence, it is apparent that where improved strength values are obtained in a material, greater energy must be expended to effect the necessary pressure to plasticly deform such material since there is an attendant increase in hardness. Unfortunately, the problem cannot be simply overcome by increasing the size of the mills available for rolling because of the additional problem of brittleness. In this regard it has been found that many improved strength materials break up or crack when relatively high rolling pressures are employed in efforts to produce sheet or strip therefrom.

Another method employed in the prior art to manufacture metallic sheet and strip involves a radically different approach. This method, termed the "sintered aluminum powder" method, and commonly called SAP, attempts to exploit the advantages of the mechanism of dispersoid strengthening. This method is well known as producing remarkable creep properties in aluminum and comprises the dispersion of a stable substance in a matrix of aluminum. The stable substance, usually aluminum oxide, is mixed in particulate form with powdered aluminum and compacted, e.g., by extrusion, with or without additional heating, to form a dispersoid therein. While the method has the distinct advantage of producing considerably increased strength at high temperatures, the very nature of the method (the starting material is in powder form) gives rise to serious rolling problems. A powdered dispersion usually lacks enough ductility to roll well. It also has high hot strength, which further complicates rolling. Attempts to make sheet material by the SAP method have also resulted in such disadvantages as edge cracking and limited continuity.

The present invention obviates the problems attendant the rolling of powder by employing foil in place thereof. The resultant effort has only to bond the stacked foils and effect a reduction of the stack (a very favorable starting shape and condition for making sheet and strip). While the end product in both the SAP method and the instant invention depends primarily on the dispersoid mechanism for its strength characteristics, it is important to note that the methods employed to obtain such product differ markedly. In the SAP method rolling starts with a material comprising a dispersion and, hence, is attended by the aforementioned disadvantages of low ductility, high hot strength, etc. In the present invention rolling starts with material free of any dispersion and is, therefore, substantially unattended by said disadvantages and greatly simplified.

The present invention has for its principal object the production of metallic sheet and strip material which is unattended by the foregoing disadvantages of the prior art.

Another object of the invention is to provide a method for producing metallic sheet and strip material exhibiting improved strength properties at high temperatures.

Still another object of the invention is to provide a method for producing metallic sheet and strip material at substantially reduced compacting pressures.

A further object of the invention is to provide a method for producing metallic sheet and strip material strengthened by stable dispersoids and produced entirely by mechanical working.

Other objects and advantages of the invention will in part be obvious and in part be pointed out hereinafter in the following detailed description of the principles of the invention and several of the embodiments thereof.

In accordance with the general concept of the present invention, metallic material of relatively good workability, i.e., compared with the gross final product, is rolled into sheet or strip form until material of foil thickness is produced. Conditions surrounding such working are controlled in known manner to minimize deleterious effects on ductility such as produced by oxidation, carburization, etc. Resultant metallic foil is then treated in such manner as to produce a thin coating of a non-metallic compound on the surface thereof. The particular manner of forming a non-metallic coating, e.g., oxidation, is well known in the art and does not constitute a part of the instant invention. In carrying out the method of the invention, the easily rollable metallic material is rolled to a foil size of about ½ mil in thickness and the non-metallic coating is applied by proper regulation of atmospheric conditions and surface preparation so that the resultant foil contains a non-metallic coating of about 1 micron in thickness. Thereafter, the non-metallic coated foils are stacked and rolled, cut, restacked, and again rolled, in the manner known in the art as "pack rolling." This procedure effects metal-to-metal contact between the foils, thereby causing bonding. In addition to a cold welding of the foils, there is produced a homogeneous distribution of the non-metallic coating particles, a substantial proportion of which are micron and submicron in size. The achievement of a desired degree of homogeneity is facilitated by intermediate anneals.

While aluminum has been specifically mentioned as an easily rollable metal amenable to the inventive method, the invention obviously is not to be construed as limited thereto but includes other metals relatively easily rollable in the pure state, e.g., nickel, iron, and titanium, as well as other relatively easily rollable alloys. In addition, nonmetals which have been found particularly suitable for the practice of the present invention include oxygen, nitrogen, boron, silicon, carbon, and sulfur. Formation of the non-metallic coating or case on the substantially pure metal or alloy base may be effected by gas diffusion, powder pack heating and other known prior art methods, e.g., application of a slurry containing the non-metallic compound in finely divided, particulate form, followed by heating.

Accordingly, in the practice of the present invention, a specific embodiment comprises the controlled oxidation of substantially pure nickel foil, which foil is obtained by rolling nickel sheet or strip until a foil of about ½ mil in thickness is obtained. The conditions of oxidation should be such as to produce an oxide coat of no more than about 1 micron in thickness. The resultant foil is then alternately stacked with foils, also about ½ mil in thickness, of nickel-aluminum alloy, rolled, cut, repacked,

and rerolled. This mechanical working not only effects a cold-welding and permanent bonding of the interleaved foils but also produces a substantially homogeneous dispersion of oxide particles throughout the resulting sheet or strip of nickel-aluminum alloy. Such sheet or strip material, in contrast with that conventionally produced, exhibits substantially enhanced strength properties, particularly that of creep-strength.

Another embodiment of the invention includes the preparation of a high strength sheet or strip of titanium. Substantially pure and ductile titanium may be reduced to foil and given a coating of an alkaline earth oxide, e.g., by means of an aqueous slurry containing finely divided particles of CaO or MgO. Ideally the gross final product contains about 1 to 10% of the inert oxide. Hot rolling of the coated foils in welded packs to about a 90% reduction results in the high strength product of the invention. Obviously, other dispersoids, e.g., oxides of beryllium and thorium as well as certain rare earth oxides including gadolinium and other materials suggested by my co-pending application Serial No. 634,156 filed January 15, 1957, now Patent No. 3,066,391, are contemplated within the broad inventive concept, the essential quality being that the dispersoid comprise material which is stable and insoluble in the base metal at elevated temperatures.

Illustrative of a further aspect of the invention, the stock for rolling may comprise a unitary article, as an elongated strip or sheet consisting of a plurality of laminations of metal or alloy, as above described, and alternate layers of non-metallic, dispersoid-forming material as above described. Such a stock is prepared by application, to the surface of the desired metal, in foil form, of a thin layer of suitable non-metal, for example, oxide, by any suitable means, as above mentioned. Thereupon, a further, thin lamination of the same or another metal, depending upon the desired composition of the final product, is applied over the non-metallic layer, for example, by vapor deposition or dipping. For example, thin aluminum foil, about ½ mil thickness, is subjected to alternate oxidation, to form an oxide coat of micron or sub-micron thickness, and subsequent metallization, as by deposition of aluminum, from the vapor phase, until a stock of substantial thickness, e.g., about 20 mils, is built up. The laminations may be conveniently formed, for example, by mounting the metallic foil base upon a rotatable drum, or between spaced-apart rolls, and the thus-mounted foil passed alternately through oxidizing and metal deposition chambers. The laminated stock thus formed is then mechanically reduced in thickness, say to 10 mils or less, as by rolling, whereupon the discrete metal and oxide laminar structure is destroyed and homogeneous metal-to-metal bonding and oxide dispersion is achieved, resulting in a composite, dispersoid strengthened article as aforesaid.

The metal rolling stock in accordance with this aspect of the invention, in certain instances, may be conveniently deposited by decomposition of a vapor phase compound of the desired metal, e.g., nickel carbonyl may be so utilized where one or more of the stock laminations are metallic nickel.

It will be further understood that various other changes and modifications may be made in the inventive method and that materials other than those specifically described herein may be employed without departing from the spirit and scope of this invention.

Having thus described my invention so that others skilled in the art may be able to understand and practice

the same, I state that what I desire to secure by Letters Patent is defined in what is claimed.

I claim:

1. A method of producing stable dispersoid strengthened base metal articles comprising:
   (a) rolling the metal to foil;
   (b) applying to the foil a coating of a stable dispersoid forming material selected from the group consisting of metal oxides, nitrides, borides, silicides, carbides, and sulfides which are stable and insoluble in the base metal at temperatures up to the maximum end use application temperature of the article;
   (c) forming laminae by cutting the coated foil into desired lengths;
   (d) stacking a plurality of laminae into a pack for rolling;
   (e) rolling the pack to a reduced thickness;
   (f) repeating the cutting, stacking and rolling steps until a unitary article is obtained wherein the laminae are welded together to form a continuous metal matrix and wherein submicron-sized particles of the stable dispersoid material are substantially uniformly distributed throughout the matrix volume.

2. A method in accordance with claim 1 wherein the metal foil has a maximum thickness of about 0.5 mil and the applied coating has a maximum thickness of about one micron.

3. A method in accordance with claim 2 wherein the cutting, stacking and rolling steps are repeated until the metal laminae are reduced to a maximum thickness of about one micron.

4. A method in accordance with claim 1 wherein the stable dispersoid strengthening material is an oxide.

5. A method in accordance with claim 4 wherein the stable dispersoid strengthening material comprises an oxide of at least one metallic element of the base metal.

6. A method in accordance with claim 1 wherein the coating is of a substantially lesser thickness than the foil.

7. A method of producing a dispersoid strengthened flat-rolled article comprising:
   (a) forming a supporting surface of at least one component of a matrix metal;
   (b) depositing upon said surface a first layer of a dispersoid forming material selected from the group consisting of metal oxides, nitrides, borides, silicides, carbides, and sulfides which are stable and insoluble in the base metal at temperatures up to the maximum end use application temperature of the article;
   (c) depositing upon said first layer a second layer of at least one component of said matrix metal;
   (d) repeating steps (b) and (c) until a rolling stock of desired composition and thickness is produced; and
   (e) rolling said stock to a reduction of at least 10%.

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