

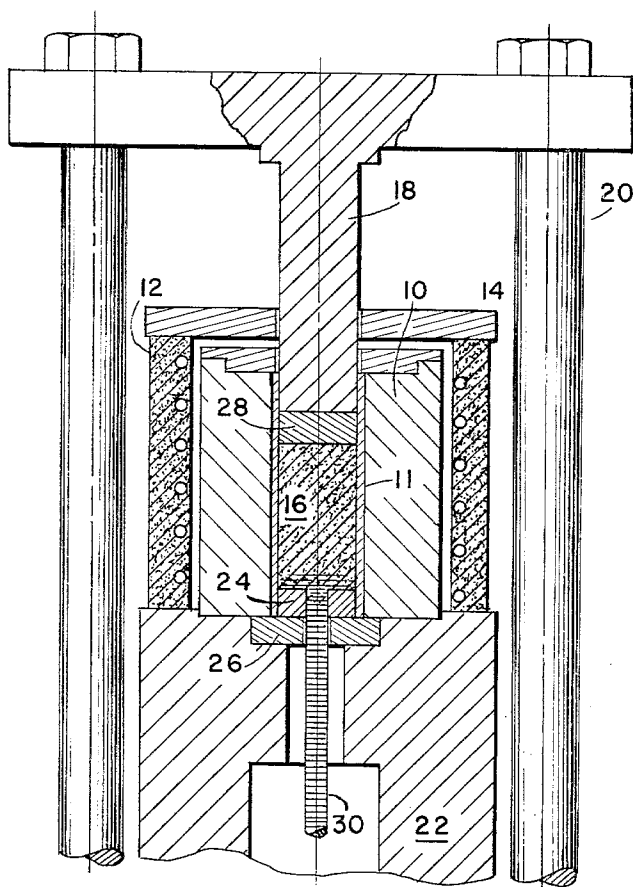
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PROCESS FOR THE EXTRUSION OF ULTRA-FINE WIRES

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PROCESS FOR THE EXTRUSION OF ULTRA-FINE WIRES

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This invention relates to metallurgy and more particularly to a method of producing dispersion-strengthened ultra-fine wires by extrusion. This application is in part a continuation of my copending applications Serial No. 220,734, filed August 31, 1962 and Serial No. 195,623, filed May 17, 1962.

Heretofore production of ultrafine wires has been achieved by drawing processes whereby the metal or alloy is drawn through a hole in a plate or block of harder material known as a die. This operation converts the starting material, a rod, for example, to an elongated product of reduced cross-section. The drawing process is repeated using dies with smaller holes until the desired reduction in area and cross-section is obtained. The wire drawing process, while effective to produce wires, is accompanied by numerous disadvantages. For example, due to the work hardening which takes place during cold drawing the ductility of the metal wire decreases and tensile strength increases with the result that the metal wire produced tends to become hard and brittle and often requires annealing to permit still further reduction to smaller sizes or diameters. Such drawing processes are relatively slow and add greatly to the cost of the drawn wire. For example, the cost of a series of dies is quite large.

In the extrusion of metals wherein a heated billet, suitably confined, is expressed through a die orifice, considerable difficulty has been encountered because of the lack of control of such factors as the speed of extrusion, the temperature of extrusion, the extrusion ratio, and the extrusion pressure and flow stress. While various extrusion processes have been successfully practiced in producing extruded products of relatively large dimensions such as rods, tubes, and the like, attempts to apply such known extrusion practice to the production of ultrafine wires have not been generally successful. Consequently, no technique or method has been devised for obtaining dispersion strengthened ultrafine wires by extrusion.

With respect to pick-up on the inner surface of the billet container, when the billet material is held and extruded from a billet container having an oxidized, nitrided or carbonitrided inner surface adjacent the billet material, pick-up on the billet container surface is eliminated. The oxidized-nitrided or carbonitrided surface provides a surface which is substantially nonreactive with the extrusion material; that is, the extrusion material will not weld, alloy or react with the treated surface. The oxidizing, carbonitriding or nitriding of the billet container may be carried out by methods well known by those skilled in the art. Heating member 12 containing heating coils 14 surrounds the extrusion container 10 and heats the extrusion material 16 by radiation to the desired initial temperature. Extending into the extrusion container is a ram 18 which is supported on suitable columns 20. The extrusion container 10 is supported on die holder 22 and is provided with a shearing-edged flat diamond die 24 and die backup adapter ring 26. Pick-up on the die bearing surface is greatly reduced or eliminated when the metal is expressed through a diamond die.

Positioned between the ram and the extrusion material is a suitable closure disc 28. The ram 18 is advanced against the extrusion material by mechanical or hydraulic means not shown to express the billet material through the die 24, and extrude the ultrafine wire 30. The extruded

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wire is preferably collected on an automatically driven spool not shown. Appropriate means (not shown) may be employed for maintaining a nonoxidizing inert atmosphere within or about the extrusion chamber as those skilled in the art will readily understand.

A principal object of the present invention is to provide a process of producing dispersion strengthened ultrafine wires by extrusion.

Another object of the present invention is to provide extruded dispersion-strengthened or hardened wires having diameters of less than about 10 mils (.01 inch).

A further object of the present invention is to provide a dispersion-strengthened ultrafine wire extrusion process which is effective in producing a more economic product than heretofore possible.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

The invention accordingly comprises the product possessing the features, properties and the relation of components and the process involving the several steps and the relation and order of one or more of such steps with respect to each of the others which are exemplified in the following detailed disclosure and the scope of the application of which will be indicated in the claims.

For a fuller understanding of the nature and objects of the invention reference should be had to the following detailed description taken in connection with the accompanying drawing which is a diagrammatic, fragmentary sectional view of an extrusion apparatus for practicing the present invention.

In accordance with the present invention an intimate mixture of a matrix metal and a dispersion strengthening material is introduced into an extrusion billet container and then heated to an initial temperature at which the compressive flow stress of the matrix metal is about 10-25 percent of the room temperature value. By compressive flow stress is meant the value of strain or compression required to cause plastic deformation. The heated mix is then expressed through the die opening at a sufficient rate to further heat the mix by internal friction of deformation to a temperature which is in excess of the initial temperature and below the solidus temperature of the matrix metal. The term "solidus temperature," as used in the specification and claims, is intended to include in its scope the melting point of the matrix metals or the temperature of appearance of the first liquid for a matrix alloy when the matrix alloy is slowly heated. In this manner an ultrafine dispersion strengthened wire is produced. Preferably the extrusion is carried out under a non-oxidizing atmosphere. For example, the extrusion may be carried out under an atmosphere of inert gas such as argon.

In one preferred embodiment of the present invention the intimate mixture of the matrix metal and the dispersion material is prepared by blending powders of the metal and dispersion material. The powder mix is then extruded to form a dispersion strengthened ultrafine wire. In this embodiment the matrix metal powder can be prepared for example by such methods as atomization, comminution, and vapor condensation. In one preferred embodiment of the present invention the metal powder is prepared in accordance with U.S. Patent 3,049,421 to L. R. Allen et al. The principal requirements of the matrix metal powder is that the metal powder be free of inclusions such as graphite, carbides, oxides and dirt having a particle size in excess of 20 percent of the diameter of the wire to be extruded. Although the particle size of inclusions must be less than 20 percent, and preferably less than 10 percent, of the wire diameter, it is to be understood that it is preferable that the metal powder be free of inclusions. Further, where the metal to be

extruded is an alloy powder, the alloy powder is prepared or treated to provide an alloy powder having a particle size of the second phase or intermetallic harder constituents of less than 20 percent of the product wire diameter.

In this embodiment of the invention, the dispersion material can be prepared by the same methods as described for the preparation of the matrix metal powder. As dispersion materials, powders of metals, metallic compounds, alloys, oxides, carbides and nitrides, for example, are suitable. The principal requirements of the dispersion powders are that they have a particle size of less than 20 percent, and preferably less than 10 percent, of the wire diameter to permit extrusion of the ultrafine wire, that they have a low or zero solubility with respect to the matrix metal and that they have a higher hot hardness or strength than the matrix metal.

In another preferred embodiment of the present invention, the intimate mixture of matrix metal and the dispersion material is provided by treating the matrix metal powder to form a thin skin or surface layer of the dispersion material on the metal powder. In this embodiment of the invention, the surface of the metal powder is coated, plated, oxidized, carburized, or nitrided, for example, to provide a thin layer of the dispersion compound on the powder particles. The coating, plating, oxidizing, carburizing and nitriding can be carried out by methods well known by those skilled in the art. In accordance with this embodiment, the layer or coating of the dispersion material on the metal powder is broken during the extrusion so that the metal powder becomes the continuous metal matrix and the dispersion coating or skin becomes the dispersed phase.

With respect to the particle size of the dispersion material, fine powder particles are preferred to coarse powder particles. Finer powder particles provide more homogeneous mixtures with uniform spacing of the dispersed material in the matrix metal. Additionally finer powder particles provide smaller interparticle spacing between the dispersed phase particles which, for example, impede deformation on working. In this manner greater strength is achieved in the extruded ultrafine wire. Preferably the dispersed phase particle size is on the order of .1 micron to .001 micron and preferably less than .05 micron. It is to be understood that the invention is not to be limited to the size of the particles for the matrix metal and that a particle size may be selected, depending on the degree of strengthening desired in the extruded wire and the extrusion ratio, since a larger particle requires a larger extrusion ratio to give a wire of equivalent strength.

Reference will now be made to the drawing which illustrates a diagrammatic, fragmentary, sectional view of a direct vertical extrusion apparatus of the type used in practicing the present invention. In the drawing 10 represents the billet or extrusion container having a 1/2 inch inner diameter bore. The inner surface of the extrusion container 10 is oxidized, nitrided or carbonitrided to provide a layer 11 which is nonreactive to extrusion material 16 which is shown as a powder mix.

As discussed hereinbefore, in order to obtain uniform continuous extrusion of ultrafine wire, the matrix metal powder to be extruded must be free of hard particles in excess of 20 percent; and preferably less than 10 percent of the diameter of ultrafine wire to be extruded. By the term "hard particle" as used in the specification and claims is meant inclusions such as graphite, carbides, oxides, dirt and other foreign impurities and/or intermetallic particles such as intermetallic compounds or hard second phases which are present in certain alloys.

In one preferred embodiment of the present invention, the non-intentional hard particle inclusions are removed by zone melting techniques which are familiar to those skilled in the art. In this embodiment of the invention the metal to be extruded or the metals which form the alloy to be extruded are zone melted to remove inclusions having a particle size in excess of 20 percent of the diam-

eter of the ultrafine wire. Preferably the zone melting is carried out in vacuum or in a clean atmosphere of inert gas such as argon or under a suitable flux.

In another preferred embodiment of this aspect of the invention, the inclusions in the matrix metal are removed by filtration techniques. In this embodiment of the invention the heated molten metal is passed through a ceramic or stainless steel sieve of suitable mesh orifice size to filter out the inclusions. Preferably the filtration is carried out in vacuum or a clean atmosphere of inert gas.

The matrix metal free of hard particles is then comminuted to powder under non-contaminating conditions.

With respect to alloys the particle size of intermetallic particles may be reduced by rapidly cooling the alloy from a molten condition. In one preferred embodiment of the present invention the molten alloy is poured in a stream through a stainless steel or ceramic screen into a container of distilled water. In this manner the molten alloy is rapidly cooled and formed into shot in which the intermetallic particle size is reduced to less than about 5 microns. The screen serves to form equal streams of the molten alloy which upon introduction into the water are rapidly cooled and formed into shot. Preferably the heating and pouring is carried out under clean atmosphere of a non-reactive inert gas such as argon or nitrogen.

In another preferred embodiment of this aspect of the invention the alloy is heated and the molten alloy poured into a heavy walled chill mold such as copper or graphite to rapidly cool the molten alloy. Preferably the mold is cooled by a coolant such as water although other cooling media may be employed. The heating, pouring and casting is preferably carried out under vacuum. In this manner, the intermetallic particle size of less than about 2-5 microns can be achieved. The casting is then comminuted to the desired particle size under non-contaminating conditions.

In accordance with the present invention one of the important features is that of maintaining the temperature of the matrix metal being extruded within the above-mentioned temperature interval: that is, between the initial temperature and the solidus temperature in order to obtain continuous uniform extrusion of ultrafine wire. It has been determined that once the particular matrix metal to be extruded has been heated to the initial temperature the temperature to which the metal will be raised during the extrusion due to the heat of deformation will be controlled by the extrusion rate and hence the ram speed.

One important aspect of the present invention is that the billet material be extruded through a sharp or shearing-edged orifice in a die having a flat surface adjacent the billet material; that is to say the surface of the die against which the billet material is placed should not have any coning or tapering lead-in surface or rounded edge to the die orifice. By extruding the billet material through this type of die the extrusion can be carried out at lower pressures which permit higher extrusion rates of the wire. Thus the internal frictional heating of the billet material at the point of maximum deformation can be sufficiently controlled to maintain the proper temperature interval for extrusion. Additionally the extrusion can be carried out without exceeding the limiting stresses for the extrusion tools. With respect to the ram speed it is to be understood that for a given extrusion rate the ram speed will vary with the extrusion ratio.

The invention will now be described by way of the following non-limiting examples:

Example 1

In this example the extrusion material consisted of commercial atomized aluminum metal powder having a particle size of about 20 microns. The aluminum metal powder was free of hard particles and had an aluminum oxide coating of 5-10 percent by weight. 45 grams of

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the oxide coated aluminum powder was then placed in the 1/2 inch diameter extrusion container and heated to a temperature of 455° C. At this temperature the compressive flow stress of the matrix aluminum metal particles is about 20 percent of the room temperature value. The extrusion mixture was then expressed through a diamond die having an orifice of 0.003 inch diameter at extrusion rates of 100 feet per minute of 0.003 inch wire. The bulk temperature of the extrusion was maintained below about 550° C. Above 550° C. the extrusion interrupts itself by splitting molten aluminum at ram speeds as low as 0.1 inch per minute. The 0.003 inch diameter wire produced was uniform and was tested for tensile strength. The tensile strength of the wire was 33,500 lbs./sq. inch. This tensile strength compares favorably with aluminum wire having a larger diameter and prepared by normal methods.

Example 2

In this example the aluminum oxide coated aluminum metal powder of Example 1 was further oxidized by steam treatment at 200° C. to produce a layer of aluminum oxide of about 10–15 percent aluminum oxide by weight. 45 grams of the oxide coated aluminum particles were introduced into the 1/2 inch diameter extrusion container and heated to a temperature of 500° C. At this temperature the compressive flow stress of the matrix aluminum metal particles is about 12 percent of the room temperature value. The extrusion mixture was then expressed through a diamond die having an orifice of .003 inch diameter at extrusion rates of 100 feet per minute of 0.003 inch wire. The temperature of the extrusion was maintained below about 550° C. The 0.003 inch diameter wire produced was uniform and was tested for tensile strength. The tensile strength of the wire was 46,250 lbs./sq. inch. This tensile strength compares favorably with aluminum wire of larger diameter prepared by normal methods.

While preferred embodiments of the invention as practiced with respect to aluminum as the matrix metal have been described in the foregoing examples, the table set forth below is exemplary of the invention as applied to other matrix metals. Included in the table is Example 1.

Matrix Metal	Extrusion Temperature Interval (° C.)	Initial Temperature (° C.)
Al.....	400–550	445
Bi.....	225–265	246
Cd.....	200–300	255
In.....	25–140	100
Mg.....	300–500	320
Sn.....	140–210	145
Zn.....	250–400	370
Pb.....	200–310	210

Since certain changes may be made in the above products and process without departing from the scope of the invention herein involved, it is intended that all matter contained in the above description, or shown in the accompanying drawings, shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. The process of producing dispersion hardened ultra-fine metal wire having a diameter of less than about 0.01 inch comprising forming a powder of matrix metal having hard particle sizes of less than 20 percent of said wire diameter, intimately mixing a dispersion hardening material having a particle size of less than 20 percent of said wire diameter with said matrix metal, introducing said mixture into a billet container, heating said mixture to an initial temperature at which the compressive flow stress of said matrix metal is about 10–25 percent of the

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room temperature value, expressing said mixture through a shearing-edged opening in a substantially flat die surface at a sufficient extrusion rate to further heat said mixture to a temperature which is in excess of said initial temperature and below the solidus temperature of said matrix metal to thereby form a dispersion hardened extruded wire.

2. The process of claim 1 wherein an aluminum matrix is extruded at a temperature between about 400° C. and 550° C.

3. The process of claim 1 wherein a bismuth matrix metal is extruded at a temperature between 225° C. and 255° C.

4. The process of claim 1 wherein a cadmium matrix metal is extruded at a temperature between about 200° C. and 300° C.

5. The process of claim 1 wherein an indium matrix metal is extruded at a temperature between about 25° C. and 140° C.

6. The process of claim 1 wherein a magnesium matrix metal is extruded at a temperature of between about 300° C. and 500° C.

7. The process of claim 1 wherein a zinc matrix metal is extruded at a temperature between about 250° C. and 400° C.

8. The process of claim 1 wherein a lead matrix metal is extruded at a temperature between about 200° C. and 310° C.

9. The process of producing dispersion hardened ultra-fine metal wire having a diameter of less than about .01 inch comprising forming a powder of a matrix metal having hard particle sizes of less than 20 percent of said wire diameter, treating said matrix metal powder to form thereon a thin layer of a dispersion hardening material selected from the group consisting of metals, metallic compounds, alloys, oxides, carbides, and nitrides, insoluble in said matrix metal, introducing said treated matrix metal into an extrusion container, heating said treated matrix metal to an initial temperature at which the compressor flow stress of said matrix metal is about 10–25 percent of the room temperature value, expressing said mixture through a shearing-edged opening in a substantially flat die surface at a sufficient extrusion rate to further heat said matrix metal to a temperature which is in excess of said initial temperature and below the solidus temperature of said matrix metal to thereby form a dispersion hardened extruded wire.

References Cited by the Examiner

UNITED STATES PATENTS

791,096	5/05	Hoopes	29–193
988,921	4/11	Abbott	29–193
2,630,623	3/53	Chisholm et al.	207–10
2,686,864	8/54	Wroughton et al.	207–10
2,720,310	10/55	Yack	207–10
2,738,064	3/56	Kreidler	207–10
2,974,790	3/61	Murphy et al.	207–1.2
3,088,589	5/63	Botes	207–10

FOREIGN PATENTS

564,081 9/44 Great Britain.

OTHER REFERENCES

Wire and Wire Products, October 1960, vol. 35, No. 10, pp. 1341, 1344, and 1345.

Metals Handbook 1948 ed., published by the American Society for Metals: Cleveland, p. 975 relied on.

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