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METHOD OF PRODUCING MOLYBDENUM ALLOY COMPOSITIONS FROM AMMONIACAL SOLUTIONS

Wasyl Kunda, Fort Saskatchewan, Alberta, Canada, assignor to Sherritt Gordon Mines Limited, Toronto, Ontario, Canada, a company of Canada

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This invention relates to molybdenum base compositions and a novel method of producing such compositions. More particularly, it is concerned both with molybdenum base compositions containing a uniform dispersion of discrete insoluble particles and alloys consisting of molybdenum and one or more other metals in partial or complete solid solution. The compositions of this invention may be in the form of solid, finished or intermediate shapes or in the form of molybdenum alloy powders or molybdenum coated composite powders in which discrete, individual particles of core materials are uniformly coated with a film of substantially pure molybdenum metal. The process of the invention involves the production of molybdenum containing powders consisting of a complex molybdenum compound coated upon or intimately mixed with selected core materials by hydrogen reduction of molybdenum from aqueous solutions and the utilization of such powders in the production of molybdenum alloy compositions and molybdenum coated composite powders.

It is known to use composite metal coated powders in the manufacture of special metal parts or special alloys which cannot be manufactured satisfactorily, conveniently or economically by conventional melting and casting methods. The need for a satisfactory method of producing molybdenum composite powders, particularly for use in molydenum alloys, is emphasized by the fact that, in view of molybdenum's high melting point, conventional melting and casting procedures are completely inappropriate or exceedingly expensive. Thus, powder metallurgical techniques are almost universally employed in the production of molybdenum alloys.

Composite metal powders have been produced by spraying or sputtering the metals of interest at a temperature above their melting temperature. But, of course, this method generally is restricted to metals having a low melting temperature. This method is clearly inappropriate for molybdenum in view of its high melting point. Also, these methods cannot be employed to produce composite powders in which individual discrete particles are coated with metal; rather, the composite powder product of these processes consists in an intimate mixture of dispersed particles and a matrix metal. Other methods utilizing, for example, "plasma" coating techniques or "chemical vapour deposition" techniques can be adapted for coating core materials with molybdenum but they are prohibitively expensive for any large scale use.

United States Patent No. 2,949,358, issued August 16, 1960 to Guy B. Alexander et al., discloses a method of producing molybdenum compositions containing a uniform dispersion of a metal oxide. In particular, the patent discloses compositions consisting essentially of molybdenum having uniformly dispersed therein from about 0.5 to 30 weight percent of sub-micron particles of a refractory metal oxide having a melting point above 1000° C. and a free energy of formation at 1000° C. of from 60 to 150 kilogram calories per gram atom of oxygen and a method of producing such compositions. Similar rhenium and tungsten compositions are also described. In essence, the method of the patent involves, insofar as molybdenum is concerned, a procedure in which a hydrous oxygen containing molybdenum compound is deposited as a coating on particles of a refractory oxide or particles of a compound which is convertible to a refractory oxide upon heating. The coated particles are then heated at an elevated temperature in a hydrogen atmosphere to reduce the molybdenum compound to metal and, where applicable, to convert the coated compound to a refractory oxide. The method is carried out by the controlled mixing of three separate solutions: a solution of an ionizable compound of molybdenum; a solution containing dissolved ions which will combine with the dissolved molybdenum to form a hydrous, oxygen containing molybdenum compound that will precipitate; and a liquid dispersion of refractory oxide particles or particles convertible to an oxide on heating.

The method has several disadvantages: The extraction of molybdenum from the solution is not complete; the method is apparently limited to sub-micron size particles of refractory oxide and there is no reference to dispersed particles of other compounds or to particles of other sizes; the precipitate would be somewhat difficult to handle and filter; the cost of reagents is relatively high; and the precipitation step appears somewhat complex from the operational point of view and difficult to properly control; and the process is limited to the production of powder containing a plurality of dispersed refractory oxide particles.

It is accordingly an object of the present invention to provide an improved method for producing molybdenum base compositions containing molybdenum metal together with other metallic and/or non-metallic constituents. It is also an object of this invention to provide a method by which a much wider range of molybdenum base compositions can be produced than was previously possible. Another object of this invention is to provide a method for producing a wide variety of molybdenum coated composite powders consisting of individual core particles uniformly coated with molybdenum. A further object of the invention is to provide an efficient and economic method by which dispersion strengthened molybdenum compositions can be obtained. A still further object of this invention is to provide a number of novel molybdenum base compositions which were heretofore unobtainable. Additionally, it is an object of this invention to provide a novel procedure for preparing alloys of molybdenum and other metals in particulate and in solid form. Other objects will become evident as the description proceeds.

Broadly, this invention involves providing solid particles of a suitable core material as nuclei in an ammoniacal ammonium sulphate solution in which molybdenum is present as a soluble salt, precipitating the molybdenum from the solution by hydrogen reduction as a complex molybdenum compound to form a particulate product consisting of core material coated by or intimately mixed with the precipitated complex molybdenum compound, separating the so-formed product from the solution and heating the same, either before or after compaction into intermediate or final shapes, in the presence of hydrogen to reduce the complex molybdenum compound to substantially pure molybdenum metal.

In United States Patents Nos. 2,853,398, 2,853,401 and 2,853,403, processes are disclosed by which metals selected from the group consisting of osmium, rhodium, ruthenium, iridium, gold, platinum, silver, copper, arsenic, tin, nickel and cobalt can be deposited as a coating on the surfaces of core materials. These processes involve the precipitation of the coating metal by gas reduction from a solution in which it is present as a soluble salt and which contains a physical dispersion of particles of the substance which is to be coated by the precipitated metal.

It was previously believed that it was not possible to

quantitatively recover molybdenum from a solution of its salt by precipitation by gas reduction; however, a process has now been discovered by which molybdenum can be quantitatively precipitated from an aqueous solution of its salts by gas reduction at elevated temperature and pressure. This process is described in detail in copending United States application Serial No. 269,642 filed April 1, 1963. According to this process, dissolved molybdenum is precipitated from an ammoniacal ammonium sulphate solution as a complex salt by reacting the solution with hydrogen at elevated temperature and pressure. The reaction proceeds only in the presence of a catalyst and when the solution composition and reduction conditions are controlled within specific limits.

The present invention utilizes the basic process of copending application Serial No. 269,642 to produce molybdenum alloy compositions and molybdenum coated composite powders. Powders containing molybdenum and at least one other selected constituent are produced by providing particles of the selected constituent as nuclei or core material in an ammoniacal ammonium sulphate solution which contains, in solution, a compound of molybdenum, and reacting the solution, in the presence of a catalyst, with hydrogen at a temperature above about 250° F. and under a partial pressure of hydrogen above about 100 pounds per square inch. The reducing reaction is continued to precipitate the molybdenum from solution in the form of a complex compound as a coating on the surfaces of the core particles. The resulting coated particles are subsequently heated in a strongly reducing atmosphere to yield molybdenum coated composite particles as such or molybdenum alloy particles, or the particles are formed into compacts, either before or after preliminary treatment to reduce the complex molybdenum compound, and subjected to further sintering and hot or cold working to produce useful wrought shapes.

A large variety of materials can be utilized as the alloying agents or core materials, for example, iron, aluminum, chromium, tungsten, silicon, carbon, alumina, zirconia, titania, tungsten carbide, titanium carbide, chromium carbide, silicon carbide, silicon nitride, glass and asbestos and any other solid material which is insoluble in ammoniacal ammonium sulphate solution under the reaction conditions. In addition, any one or more of the metals of the group silver to cadmium inclusive in the E.M.F. series can be utilized. When one or more of these metals is selected as the alloying agent, they can be provided in the reduction solution directly as a physical dispersion of discrete particles, or they can be provided in the solution as a soluble salt in which case they will subsequently precipitate from the solution, upon initiation of the reduction reaction, to provide nuclei on which the molbydenum precipitates during the continuance of the reducing reaction.

Dispersion strengthened molybdenum compositions having the improved physical properties characteristic of such materials can be conveniently produced by compacting, sintering and working, such as by extrusion, those powders containing inert refractory compounds as the core material. The complex molybdenum compound can be reduced to metallic form either before or after compaction and/or other conventional forming procedures.

Powders in which the core materials consist of metals which form useful alloys with molybdenum have been found particularly useful in producing alloys of molybdenum and such other metals. It has been found that the molybdenum compound is highly reactived during its conversion to metallic form, and this, combined with the very intimate mixing of the molybdenum compound with the alloying agents which results from the aqueous reduction technique of this invention, permits complete alloying of the molybdenum with alloyable metals, such as nickel, at temperatures well below the melting point of the metals simply by sintering the alloy powders in a strongly reducing atmosphere. Alloys are produced in particulate form by sintering the powder, as such, in a reducing atmosphere and in solid, finished or semi-finished shapes by conventional compacting and hot and/or cold working procedures carried out either before or after reduction of the molybdenum compound to metallic form.

In precipitating the molybdenum from solution as a complex compound coated on the core material, there are three important considerations:

(1) The provision of a suitable catalyst in the reduction solution or the catalytic activation of the core material;

(2) The control of reduction solution composition; and

(3) The control of operating conditions to effect substantially complete precipitation of the molybdenum in a minimum length of time.

The precipitation of molybdenum from solution by hydrogen is a catalytic reaction and the presence of a suitable catalyst is essential. In precipitating molybdenum as a complex compound coated on core materials dispersed in the reduction solution, it is thus essential to provide a catalyst in the solution or provide catalytically activated core materials, unless the selected core material is catalytically active in itself.

I have found that only a few core materials, including nickel and graphite, possess a surface which is sufficiently catalytic to precipitate molybdenum at a reasonably fast rate. The surface of most materials must be activated. I have found that this can be accomplished by treating the core material with any reagent which can be absorbed on the surface of the core and which is catalytically active in respect to molybdenum precipitation by hydrogen. Palladium chloride has been found to be the preferred reagent for this purpose. The core particles are activated simply by treating them with a dilute aqueous solution of the activation agent. In the case of palladium chloride, satisfactory results are obtained if the core particles are washed with an 0.5% solution of $PdCl_2$, then washed with pure water to remove excess $PdCl_2$.

Some materials, such as nickel and carbon powder, are catalytically active in themselves and complete precipitation of molybdenum compound as a coating on these materials can be attained under the conditions discussed hereinafter within about 30 minutes. However, because most core materials that will ordinarily be employed have low catalytic activity, it is generally desirable to adopt the treatment of core materials with $PdCl_2$ as a standard procedure.

As stated above, where one or more of the metals silver to cadmium inclusive in the E.M.F. series is selected as the core material, according to the present invention, powders suitable for forming molybdenum alloy compositions of molybdenum and one or more of such metals can be produced by co-precipitation of the complex molybdenum compound and any other metal or metals selected from this group which precipitate under the same conditions as the complex molybdenum compound. In this case, the reduction reaction can be catalyzed by providing palladium chloride catalyst directly in the solution. The molybdenum precipitation rate varies in direct proportion with the palladium chloride concentration in the solution. The precipitation reaction proceeds in the presence of a very small quantity of $PdCl_2$, in the order of 0.01 g.p.l. or less. However, it is preferred to use between about 0.01 and 0.05 g.p.l. The palladium chloride can be conveniently added as an 0.5% aqueous solution. The reaction can also be catalysed, in this case, by use of any of the catalytic nucleating agents which will initiate the reduction of the metals silver to cadmium inclusive in the E.M.F. series. Such nucleation catalysts are described in detail in United States Patents Nos. 2,767,081, 2,767,082 and 2,767,083. When the catalysts of these patents are utilized to initiate the reduction reaction, it is believed that the mechanism of the reaction is such that nuclei of the selected alloying

metal which is lowest in the E.M.F. series form first, then the molybdenum and the balance of the alloying metal or metals precipitate simultaneously on the nuclei to form a powder consisting essentially of an intimate mixture of complex molybdenum compound and the alloying metals in elemental form. For example, where nickel is the selected alloying metal which is dissolved in the reduction feed solution, the product is a powder consisting of nuclei of elemental nickel coated with an intimate mixture of elemental nickel and complex molybdenum compound.

The composition of the solution is a very important factor in the precipitation of molybdenum by gas reduction from the ammoniacal ammonium sulphate solution. The rate of molybdenum precipitation decreases with increases in the free ammonia (i.e. ammonia directly titratable with sulphuric acid) to molybdenum molar ratio. It is therefore desirable that the

$$\frac{NH_3(F)}{Mo}$$

molar ratio of the reduction solution be less than about 3.0 and preferably about 1.5–2.0. At this level of free ammonia, up to about 200 grams per litre of molybdenum will dissolve in the solution without difficulty and the rate of precipitation of molybdenum from the solution is satisfactory. During the precipitation reaction, about 0.4 mole of ammonia precipitates with each mole of molybdenum and thus the NH_3/Mo molar ratio increases as the reaction proceeds and the rate of precipitation decreases. Thus, for this reason alone, it is preferable that the free ammonia concentration of the reduction solution be maintained at the minimum level necessary to maintain the Mo in solution.

The ammonium sulphate concentration in the reduction solution is also important because up to a concentration of about 350 g.p.l., the rate of molybdenum precipitation increases with increase of ammonium sulphate concentration. At concentrations below about 100 g.p.l., the rate of molybdenum precipitation becomes too slow to be useful for practical purposes, and at concentrations above about 350 g.p.l., there is very little increase in the rate of precipitation with increased ammonium sulphate concentration. The preferred range for ammonium sulphate concentration is thus from about 200 g.p.l. to about 300 g.p.l.

The hydrogen pressure and temperature for the reduction step also have a pronounced effect on the precipitation rate. This rate is directly proportional to the hydrogen partial pressure in the range between 100 and 325 p.s.i. Above this pressure, a rapid increase in the precipitation rate is observed; however, generally it is not desirable to operate with hydrogen partial pressures in excess of 500 p.s.i. as the cost of the high pressure equipment required becomes excessive. The preferred hydrogen partial pressure is within the range of about 300–400 p.s.i.

The molybdenum precipitation reaction can be satisfactorily conducted at any temperature above 250° F. The rate of molybdenum precipitation increases with increased temperature, and this effect becomes more pronounced at higher temperatures. The optimum temperature range is between 275° F. and 400° F.

Under optimum conditions of solution composition and reduction conditions, the molybdenum precipitation reaction goes essentially to completion in less than 30 minutes and less than 0.1 g.p.l. molybdenum remains in the reduced solution.

The molybdenum precipitates as a dark brown or black complex compound coated on the core particles.

The molybdenum is in the solution in a valency state of +6 (as $2NH_4^- + MoO_4^= \rightleftarrows (NH_4)_2 MoO_4$) and is precipitated as a complex molybdenum oxide, it is believed, in a valency state of +4. A typical chemical composition for this precipitate is: Mo=60%, NH_3=4%, S=0.5%.

After separation from the reduction end solution and washing to remove any water soluble contaminants, the product may be treated in a number of ways depending on the composition of the material and the end product desired. The powders can be heated, as discrete particles, in a reducing atmosphere at a temperature between about 1600° F. and about 1800° F. to produce discrete, individual molybdenum coated composite particles or to produce molybdenum-metal alloys in particulate form, depending on the nature of the core material.

Composite powders consisting of individual particles of core material coated with molybdenum can be produced with a very wide variety of metallic and non-metallic cores and in a wide range of core sizes from sub-micron to in excess of 500 microns. These particles can be used as such in applications such as plasma and flame spraying or may be compacted and hot and/or cold worked, such as by extrusion, to produce finished shapes having discrete particles uniformly dispersed through a molybdenum matrix. For example, molybdenum alloys containing dispersed carbides or silicides are used for heating elements, and molybdenum alloys containing a dispersed phase of finely divided refractory oxides, such as thoria, for example, find uses in other high temperature applications.

Powders in which the complex molybdenum compound is coated on another metal or other metals can be utilized to produce a wide variety of molybdenum alloys in particulate or solid form.

Powders consisting of complex molybdenum compound and one or more of the metals silver to cadmium inclusive in the E.M.F. series may be produced by the co-precipitation of molybdenum and the selected alloying metal or metals from the ammoniacal ammonium sulphate solutions as described hereinabove or by providing the alloying metals in the solution as discrete particles. The ratios of the various metals in the final product can be easily controlled by adjusting the concentration of the various constituent metals prior to the reduction.

In the case of alloys of molybdenum and metals such as aluminum, chromium, iron, tungsten, etc., which cannot be reduced from an aqueous ammoniacal solution under conditions under which molybdenum is precipitated, the alloying metals are supplied in discrete particulate form in the desired size range from an external source. They are provided in the reduction solution as a physical dispersion in the amount calculated to give the desired composition in the final product having regard to the amount of molybdenum in solution. With the exception of nickel and carbon, it is necessary to treat the core materials to make them catalytically active prior to conducting the molybdenum coating step. The preferred procedure is to treat the core powder with an 0.5% aqueous solution of $PdCl_2$, wash the powder with water to remove excess $PdCl_2$ and charge the powder into the reduction vessel with the molybdenum solution.

The procedure described herein can be utilized to produce molybdenum compositions containing both alloying metals and a dispersed phase of discrete, insoluble particles. For example, molybdenum and nickel can be co-precipitated on core particles of thoria. Also, of course, multi-constituent metal alloys, such as Mo-Ni-Co-Cu can be produced with or without a dispersed hard phase. It will be evident that a very great number of combinations of molybdenum with other metals and/or refractory or other particles are obtainable according to this invention.

The conversion of the complex molybdenum compound precipitated as a coating on the core particles in the reduction reaction is effected by treatment with hydrogen at a temperature between about 1600° F. and about 1800° F., preferably about 1750° F. Sufficient hydrogen is supplied to combine with the oxygen contained in the molybdenum compound, and treatment is continued until the compound is substantially 100% converted to pure

molybdenum. This reduction can be carried out on the composite powder, as such, or the powder can be compacted or otherwise hot and/or cold worked before being treated with hydrogen as above described.

An important characteristic of the powder compositions produced by the hydrogen precipitation technique of this invention is the very high reactivity of the molybdenum compound during conversion to the metallic form by reduction with hydrogen at elevated temperatures. This high reactivity and the intimate mixing of the various constituents which result from the molybdenum precipitation process permits complete alloying of molybdenum with molybdenum soluble metals at temperatures well below the melting point of molybdenum.

The following examples will further illustrate the invention:

EXAMPLE 1

The following test was conducted to produce discrete composite particles consisting of an iron core coated with molybdenum, such composite powder having a composition of 70% molybdenum and 30% iron.

Iron powder was treated with an aqueous solution of 0.5% $PdCl_2$ and then washed on a filter to remove the excess $PdCl_2$ salt. The so-treated powder and molybdenum solution were charged into a gas heated, agitator equipped autoclave and the concentrations of the constituents adjusted to: 50 g.p.l. Mo; 21.3 g.p.l. Fe; 20 g.p.l. free NH_3; and 200 g.p.l. $(NH_4)_2SO_4$. The autoclave was purged with hydrogen, sealed and 350 p.s.i. hydrogen pressure applied and maintained during the reduction period. The autoclave was then heated to 325° F. and kept at that temperature for 30 minutes, after which the consumption of hydrogen ceased. The autoclave was then cooled and the product removed, filtered, washed and dried. The end solution contained 0.1 g.p.l. Mo and 0.2 g.p.l. Fe. The product powder consisting of discrete particles of iron coated with a black molybdenum compound was placed in a stainless steel boat and heated in a muffle furnace at 1750° F for 18 hours, hydrogen being constantly passed through the furnace during this period. The weight loss in the furnace was 24.0%. The composite molybdenum-iron powder produced contained 70.5% Mo and 28.8% Fe with 99.8% pure molybdenum.

EXAMPLE II

The above procedure was repeated exactly except that the iron core was not treated with $PdCl_2$ and the reduction reaction was continued for 120 minutes. The powder recovered from the autoclave contained 3.2% Mo and 96.4% Fe. 30.0 g.p.l. of Mo remained in solution. This result clearly illustrates the necessity of catalytic activation of those core materials, such as iron, which are not catalytically active in themselves.

Table 1 illustrates a number of other compositions produced by the method of this invention.

In each case, the solution contained, in addition to the Mo and core material shown, 200 g.p.l $(NH_4)_2SO_4$ and 20 g.p.l. NH_3 (free). The powders were treated under hydrogen at 1750° F. for 18 hours to convert the complex molybdenum compound to molybdenum metal. It can be noted that in the case of nickel and graphite powders, essentially complete reduction of molybdenum from the solution was obtained without activation of the powder; however, unactivated aluminum powder would not initiate the reduction reaction.

Table 1

Composition	Conditions of reduction			Charge		Type	$PdCl_2$ treatment	Product percent after H_2 reduction		Mo left in reduced solution, g.p.l.
	Temp., ° F.	H_2, p.s.i.	Time, min.	Mo, g.p.l.	Core (g.p.l.) amount			Mo	Core	
Mo/Ni	325	530	30	53	22	Ni powder, 10μ	No	69.4	29.1	0.4
Mo/Al	375	350	120	40	12	Al powder, 10μ	No	n.a.	n.a.	39.7
Mo/Al	325	480	60	50	21	Al powder, 10μ	Yes	59.4	39.7	2.2
Mo/W	325	530	45	50	21	W, 7–9μ	Yes	58.0	41.4	0.7
Mo/Si	350	500	15	45	11	Si, −325 mesh*	Yes	64.0	32.5	0.03
Mo/C	375	350	60	40	22	Graphite, 38μ	No	69.9	27.5	0.02
Mo/Al_2O_3	325	540	30	50	21	Al_2O_3, −150 mesh*	Yes	68.8	30.9	0.2
Mo/ZrO_2	375	370	35	50	21	ZrO_2, >1μ	Yes	51.3	43.7	7.6
Mo/TiO_2	325	570	35	50	21	TiO_2, >5μ	Yes	60.0	39.6	18.9
Mo/TiC	325	580	20	50	21	TiC, 3μ	Yes	60.6	38.2	0.07
Mo/Cr_3C_2	325	560	45	50	21	Cr_3C_2, −325 mesh*	Yes	70.4	26.0	0.8
Mo/SiC	325	500	40	50	21	SiC, 11μ	Yes	63.5	35.5	0.06
Mo/WC	325	570	90	50	21	WC, 200/325 mesh*	Yes	51.4	48.5	12.8
Mo/glass	325	560	70	50	21	Glass, −400 mesh*	Yes	63.5	35.8	0.03
Mo/Si_3N_4	325	500	15	50	21	Si_3N_4, −325 mesh*	Yes	63.5	35.7	0.1
Mo/asbestos	325	540	20	50	6	Asbestos fibres	Yes	66.6	32.7	0.02

* Standard Tyler Screen,

EXAMPLE III

This example shows how the method of this invention may be utilized in the preparation of molybdenum-metal alloys. Molybdenum-nickel alloy strip was prepared as follows: 10 litres of solution containing 31 g.p.l. Ni; 150 g.p.l. $(NH_4)_2SO_4$ and 18 g.p.l. NH_3 (free) were mixed with 0.10 litre of solution containing 143 g.p.l. Mo; 200 g.p.l. $(NH_4)_2SO_4$ and 38 g.p.l. NH_3 (free). The blended solution was charged into an agitator equipped, gas heated autoclave, and 0.023 g.p.l. $PdCl_2$ was added to catalyze the initial reduction reaction. The reduction was carried out at a temperature of 350° F. and under 350 p.s.i. hydrogen overpressure. The reduction was complete in 10 minutes. The reduction end solution was discharged and fresh solution of the same composition as the above-described blend was fed into the autoclave. The product of the first reduction was utilized as the catalyst for a second reduction at 350° F. and under 350 p.s.i. hydrogen overpressure. The reduction was complete in 14 minutes. A third reduction was conducted following the same procedure and was complete in 20 minutes.

The precipitated composite powder was an intimate mixture of metallic nickel and a complex molybdenum compound containing about 60% Mo and 4% NH_3.

The Ni-Mo powder was heat treated for 2 hours at 1500° F. and pulverized to improve the compactibility of the powder.

The powder was then mixed with 1 weight percent water and roll compacted at 0.010 inch roll setting. Strong, 0.026–0.028 inch thick green strip was obtained. The strip was sintered for 3 hours at 2000° F. in hydrogen, hot rolled 30%, cold rolled 30% and annealed under hydrogen for 1 hour at 1400° F.

The properties of the finished strip were as follows:

Composition	UTS (p.s.i.)	YS (p.s.i.)	E, percent	Rockwell 30T hardness
94.3 Ni/Mo 4.9	74,300	26,900	33	60

The metallographic examination of the Ni-Mo finished strip indicated complete alloying of the nickel and molybdenum.

It will be understood that modifications may be made to the improved process of this invention without departing from the scope of the invention defined by the appended claims.

What I claim as new and desire to protect by Letters Patent of the United States is:

1. The method of producing molybdenum alloy compositions which comprises the steps of: preparing an ammoniacal ammonium sulphate solution which contains, in solution, molybdenum and at least one other metal selected from the group consisting of silver, nickel, cobalt and copper, about 1.5 to about 2.0 moles of free ammonia per mole of dissolved metals and at least 100 grams per litre of ammonium sulphate; providing in said solution a nucleation agent capable of initiating the reduction of said other metal; reacting said solution with hydrogen at a temperature above about 250° F. and under a partial pressure of hydrogen above about 350 pounds per square inch to co-precipitate molybdenum and said other metal as an intimate mixture of complex molybdenum compound and elemental metal in powder form; separating and recovering the resulting precipitate from said solution and reacting said precipitate at a temperature of about 1750° F. with hydrogen to reduce the molybdenum compound to molybdenum metal and effect substantially complete alloying of said molybdenum metal and said other metal.

2. The method according to claim 1 in which said other metal is nickel.

3. The method according to claim 1 in which the nucleation promoter is palladium chloride and is provided in solution in amount equivalent to about 0.01 to 0.05 gram per litre.

4. A composition suitable for producing molybdenum base alloys, said composition consisting essentially of particles of a complex molybdenum salt containing about 60% molybdenum and about 4% ammonia, said particles having dispersed therein particles of a metal selected from the group consisting of silver, nickel, cobalt and copper, said composition being produced by hydrogen reduction from an aqueous ammoniacal ammonium sulphate solution containing, in solution, molybdenum and at least one other metal selected from the group consisting of silver, nickel, cobalt and copper, at least 1.5 moles of free ammonia per mole of dissolved metal and at least 100 grams per litre of ammonium sulphate.

5. A composition of claim 4 in which said other metal is nickel.

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DAVID L. RECK, *Primary Examiner.*