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IRIDIUM ALLOY

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ABSTRACT OF THE DISCLOSURE

Iridium-base alloy containing columbium has good workability for production of wrought products such as sheet, strip, wire and the like and provides enhanced characteristics, including high strength and high recrystallization temperature, in comparison to characteristics of pure iridium.

The present invention relates to iridium alloys and, more particularly, to iridium alloys having high recrystallization temperatures.

It is well known that iridium has a very high melting point (about 2443° C.), good resistance to oxidation at temperatures up to 600° C. or 1000° C. and excellent resistance to corrosion by a wide variety of media, including the common mineral acids, the halogens, molten optical glasses of the leaded types, many fused salts or oxides and a number of molten metals. However, unalloyed iridium, such as commercially pure iridium, has undesirably low strength and recrystallizes at undesirably low temperatures, e.g. 800° C. Such recrystallization often results in coarse-grained microstructures and accompanying poor ductility, particularly in weldments.

Iridium is obtained from concentrates of metals of the platinum group, themselves often obtained as by-products in the recovery of nickel from its ores. Commercially pure iridium may accordingly contain small amounts of impurities including palladium, platinum, rhodium, iron and nickel in amounts up to 0.5% each, though normally it is at least 98% pure and indeed generally 99.8% pure. A typical analysis of commercially pure iridium is 0.02% platinum, 0.04% palladium, 0.03% rhodium, 0.0001% lead, 0.05% iron, 0.001% nickel and the balance iridium.

In order to gain greater benefit from the corrosion resistance and other useful characteristics of iridium, it would be desirable to provide a new iridium alloy that could be worked into wrought products such as sheet, rod, wire, etc. having a greater tensile strength and a higher recrystallizing temperature than are characteristic of unalloyed forms of iridium, e.g., commercially pure iridium. Additional considerations, for some fabricating purposes, are needs for iridium alloy wrought products having good weldability and weld ductility. Heretofore, attempts have been made to overcome some of the undesired characteristics of irridum by incorporating alloying elements therein. While some success has been achieved heretofore along these lines, a number of problems still remain, including need for improvement in mechanical properties or other physical characteristics and for overcoming contamination difficulties arising where particular alloying elements for iridium have adverse effects upon materials which are processed in iridium articles and apparatus.

Although many attempts were made to overcome the foregoing difficulties and other disadvantages, none, as far as I am aware, was entirely successful when carried into practice commercially on an industrial scale.

It has now been discovered that an iridium-base alloy having new and enhanced characteristics of utility, including improved tensile strength, is obtained with columbium and iridium alloyed together in special proportions.

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It is an object of the present invention to provide a new iridium-base alloy.

Another object of the invention is to provide new iridium-base alloy wrought products including sheet, strip, rod, wire and the like.

Other objects and advantages will become apparent from the following description.

Generally speaking, the present invention is directed to a new alloy containing about 0.1% to about 2% columbium and balance essentially iridium. The alloy can be made by combining columbium with commercially pure iridium and thus may contain small amounts of impurities typically present in commercially pure iridium, e.g. platinum, palladium, rhodium, iron and nickel. However, certain impurities which are sometimes, but not typically, found in commercially pure iridium are detrimental to ductility of iridium. Lead, gold, silver and bismuth are such detrimental impurities and the amount of each of these elements, if present, in the new alloy should not exceed 0.05% each in order for good ductility to be obtained.

Minimum temperatures for recrystallizing the new alloy are higher than required for recrystallizing commercially pure iridium and the new alloy has improved buttweldability. Advantageously, the alloy contains at least 0.25% columbium, e.g. 0.25% to 1% columbium, since then the temperature of recrystallization, i.e. the minimum temperature for recrystallization, is about 1000° C. or higher. Embodiments containing about 1% columbium are particularly suitable.

The new alloy is a single phase alloy. If the columbium content is excessively greater than 2%, a two-phase alloy, which is not in accordance with the invention, with inferior properties may be obtained and be unworkable.

The new alloy can be butt-welded with a tungsten arc and thus-welded specimens in 0.02 inch sheet can subsequently be bent around a mandrel of 1¼" radius whereas an identical joint in pure iridium fractures. Sheets of iridium alloys according to the invention have tensile strengths of about 20 long tons (2240 pounds) per square inch and higher in the as-rolled state. After being welded, sheets of the alloy have shown a strength of about 15 to 18 long tons per square inch (t.s.i.). Wire of iridium alloy according to the invention has excellent tensile strength at an elevated temperature, for example 1000 C°.

In carrying the invention into practice it is advantageous that melting, and also any sintering, of the alloy be done with exclusion of oxygen. Thus, the alloy may be made by melting iridium in a zirconia crucible in an atmosphere of argon, adding to the molten iridium a sintered compact of mixed iridium and columbium powders containing the requisite amount of columbium, and reheating the crucible to above the melting point of the metal in it.

The resulting ingots may be forged or otherwise hotworked in the temperature range of 1500° C. to 1600° C. Once the alloy has been forged, working at lower temperatures is possible and the heavily worked alloy is readily worked at room temperature.

Good results can also be obtained from arc-melting the alloy in an argon atmosphere.

For the purpose of giving those skilled in the art a better understanding of the invention and a better appreciation of the advantages of the invention, the following illustrative examples are given.

Five iridium-columbium alloys of the invention, referred to hereinafter as alloys 1 through 5, were prepared from high purity iridium powder and columbium powder by mixing the requisite proportions of powders, compressing the mixtures isostatically, sintering at 1700° C. for 5 minutes each in a hydrogen-nitrogen atmosphere and then melting the sintered powder mixtures into ingot form in a tungsten-electrode arc furnace with a titanium-gettered argon atmosphere surrounding and protecting the alloy. The ingots which were in the form of round buttons, were turned over and remelted with intermediate gettering of the argon until smooth buttons were obtained. Metal working procedures for converting the ingots to sheet were to edge forge at 1500° C., then hot roll at 1500° C. and thereafter roll at 1100° C. All reheatings were in hydrogen. Alloys 1 through 4, containing columbium in amounts from 0.1% to 1%, exhibited good workability and were successfully worked to 0.5 millimetre (mm.) thick sheet. Total reduction in thickness during rolling to 0.5 mm. sheet at 1100° C. was about 80%. In order to have good malleability characteristics for working the alloy into sheet, the new alloy should be of an advantageously workable composition containing 0.1% to about 1.75% columbium and the balance iridium. Alloy 5, containing 2.0% columbium, was worked to 0.6 mm. sheet but not without some cracking during forging and rolling.

Two other alloys, namely, alloys A and B, which contained 3.0% and 5.0% columbium respectively, with the balance being iridium, and which were not in accordance with the alloy composition of the invention, could not be worked to sheet form after being made into ingots by the same techniques used for alloys 1 through 5. Alloy A was severely cracked after forging and broke up in rolling. Alloy B was very brittle and broke on forging.

Chemical compositions of alloys 1 through 5 and room temperature tensile test results pertaining thereto are set forth in Table I. The stress-relief treatment referred to in Table I was a one-half hour heating at 1100° C. When in either of the conditions referred to in Table I, the new alloy had very substantially higher tensile strength than is obtained with pure iridium in a corresponding condition. Thus, the new alloy had an ultimate tensile strength of at least about 70 t.s.i. in the 1100° C. stress-relieved condition whereas the ultimate tensile strength of pure iridium when worked and stress-relieved in the same manner was less than 45 t.s.i. When annealed one-half hour at 1500° C., all five of the alloys became fully annealed and had ultimate tensile strengths of about 30 t.s.i. to 32 t.s.i. with much finer grain structures than obtained with unalloyed iridium after the same annealing treatment. It is accordingly apparent that working the new alloy at 1100° C. has the effect of warm working with little or no recrystallization and thus increases the strength of the alloy.

TABLE I

Alloy No.	Composition		Ultimate tensile strength (t.s.i.)	
	Cb (percent)	Ir	As-rolled	Stress relieved
1	0.1	Bal	81	77
2	0.25	Bal	81	73
3	0.5	Bal	83	77
4	1.0	Bal	100	88
5	2.0	Bal	120	102

Bal=Balance, which also includes tolerable amounts of impurities such as up to 0.1% of each of platinum, palladium, and rhodium.

Table II shows Diamond Pyramid Number (D.P.N.) hardness test results obtained in room temperature testing of the rolled sheet of alloys 1 through 5 when in the as-rolled condition and in heat treated conditions obtained by heating for one-half hour at the temperatures indicated in Table II.

TABLE II

Alloy No.	As rolled	Hardness, D.P.N. after heat treatment at—					
		900° C.	1,000° C.	1,100° C.	1,200° C.	1,300° C.	1,400° C.
1	470	420	390	370	200	190	190
2	510	470	450	420	390	220	200
3	515	500	500	450	420	260	230
4	570	525	550	510	490	390	250
5	640	590	620	610	570	470	340

Results of tensile tests and hardness tests, and also micrographic studies, have confirmed that the recrystallization temperature of the new alloy is higher than the recrystallization temperature of pure iridium. Results also show that when the new alloy contains 0.25% to 2% columbium, the recrystallization temperature is at least about 1000° C., e.g. about 1100° C., and warm-worked wrought products are resistant to softening and loss of strength up to 1000° C. or 1100° C. and higher.

Butt-weld joints were made successfully with sheet of each of alloys 1 through 5 using nonconsumable electrode argon-arc techniques. Improved weld ductility was obtained with alloys containing 0.1% to 0.25% columbium and balance iridium.

For obtaining an especially good combination of weldability, workability, strength and high recrystallization temperature, a composition containing about 1% columbium, e.g. about 0.75% to 1.5% columbium, is advantageous.

The high recrystallization temperature of the new alloy enables the alloy to be warm-worked at 1100° C. to increase the room temperature ultimate tensile strength to at least about 80 t.s.i., advantageously at least 100 t.s.i. when the alloy contains about 1% or more columbium, e.g. 1% to 1.5% columbium. Ultimate tensile strength of unalloyed iridium when worked at 1100° C. is as low as 10 t.s.i. Of additional benefit, when the new alloy is warm worked and then recrystallized, e.g. by annealing at 1500° C. or by welding, the microstructure thus developed is of smaller grain size than the microstructure usually present in unalloyed iridium.

The alloy of the present invention can be produced in the form of sheet, strip, rod and wire, which can be used for making crucibles, springs, electrodes for sparking plugs and similar devices for use at elevated temperature. The new alloy is particularly useful for making crucibles that can be employed in fusing high-purity semiconductor materials, e.g. semiconductors containing columbium oxide, of types which must be maintained essentially devoid of quadrivalent elements such as titanium and zirconium.

Although the present invention has been described in conjunction with preferred embodiments, it is to be understood that modifications and variations may be resorted to without departing from the spirit and scope of the invention, as those skilled in the art will readily understand. Such modifications and variations are considered to be within the purview and scope of the invention.

I claim:

1. A wrought iridium-base alloy product containing 0.1% to 1.75% columbium with the balance essentially iridium.

2. A wrought iridium-base alloy product as set forth in claim 1 containing 0.75% to 1.5% columbium.

3. A wrought iridium-columbium alloy product as set forth in claim 1 in the form of sheet, strip, bar, wire and like products.

4. A wrought product as set forth in claim 1 containing 0.25% to 1.75% columbium.

5. A wrought product as set forth in claim 1 containing about 1% to 1.5% columbium.

6. A wrought product as set forth in claim 1 containing about 1% columbium.

References Cited

WADD TR 60–132, Part III, Refractory Metal Constitution Diagrams, January 1964, relied on pp. 242–252 and 268–271.

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