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H1D 32 7X
(56) Documents cited
GB 1494345
GB 1422461
GB 1308752
GB 1298293
GB 1295413
GB 1064056
GB 1032411
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GB 943613
GB 864447
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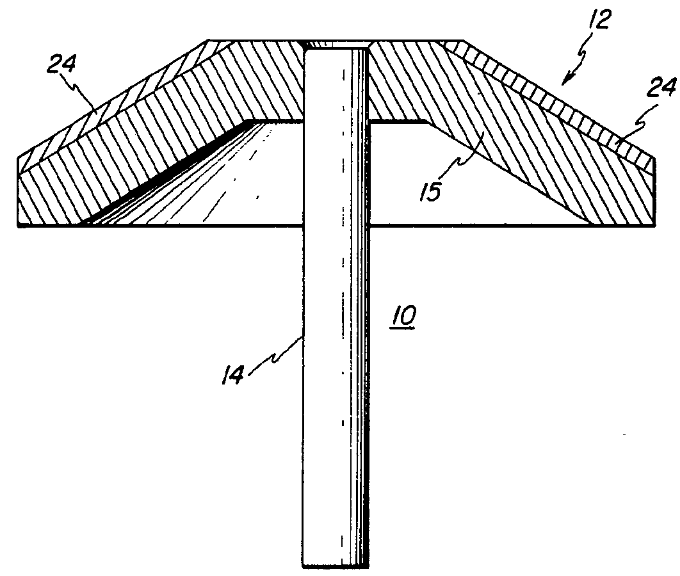
(54) X-ray tube targets

(57) In rotary targets for X-ray tubes warping is a problem which causes X-ray deficiency. A rotary target is described in which warping is reduced by using alloys of molybdenum with 0.05–10% iron, silicon, cobalt, tantalum, niobium, hafnium, stable metal oxide or mixture thereof. Suitable mixtures are 0.5 to 10% of tantalum, niobium or hafnium with from 0.5 to 5% yttrium oxide, or 0.05 to 0.3% of cobalt or silicon.

Optionally 0.1–5% by weight of additional material may be alloyed with the molybdenum, such as tantalum or hafnium carbides.



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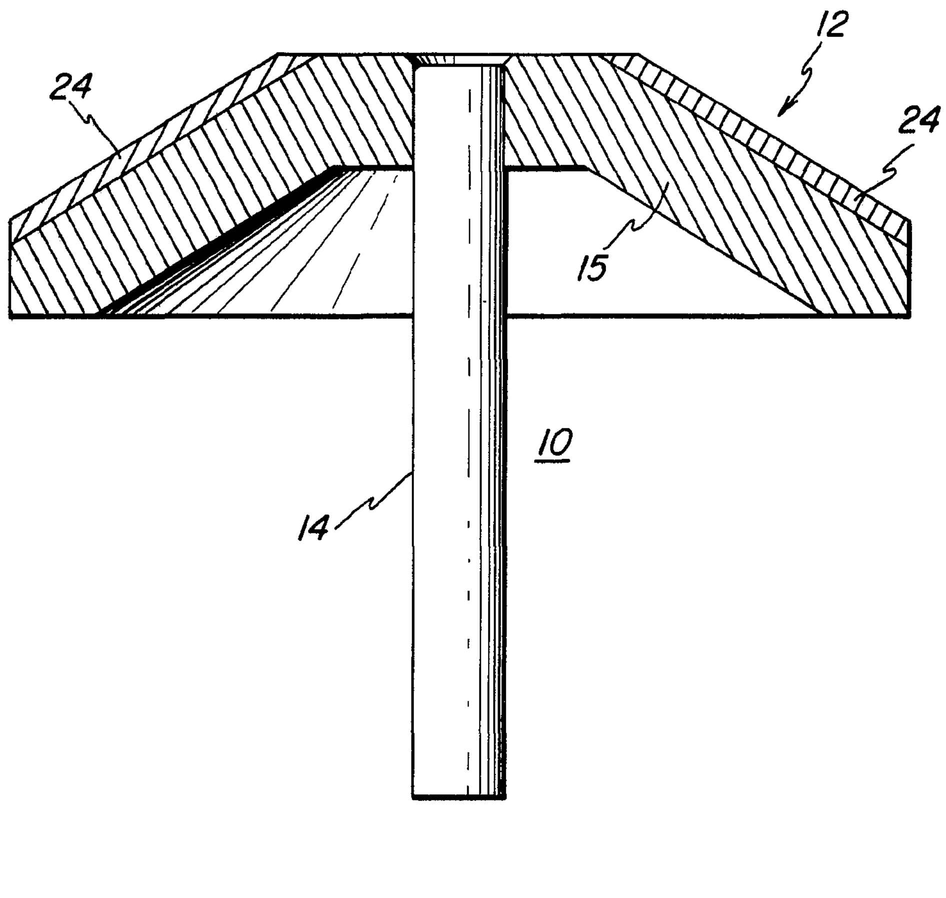
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SPECIFICATION

X-ray target with substrate of molybdenum alloy

One of the principal problems found with medical X-ray targets is one of warpage of the focal track. While some slight warpage, whether of the concave or convex type, can be tolerated, less than can be detected with the naked eye causes an undesirable drop-off in X-ray output. With warpage of the focal track, X-rays are cut off at the periphery of the X-ray window in the surrounding tube enclosure. As detected by the center position of the X-rays on the external film, if this spot shifts as little as 1°, an X-ray deficiency can exist. With certain target designs and exposures, this can occur in less than 1,000 exposures whereas the X-ray tubes are typically guaranteed for 10,000 exposures. This warpage becomes more severe or occurs earlier as the target diameter increases and the overall temperature of the substrate rises. It is this problem to which this invention is directed and it has been found that this warpage can be minimized by providing certain alloys of molybdenum as the substrate.

British Patent 1,121,407 issued July 24, 1968 and assigned to Metallwerk Plansee Aktiengesellschaft discloses the use of an X-ray base comprising molybdenum alloyed with titanium and/or zirconium and optionally carbon. Other patents such as U. S. Patent 3,649,355 issued March, 1972 and assigned to Schwarzkopf Development Corporation disclose use of a graphite base, U. S. Patent 3,660,053 issued May 2, 1972 to Palme discloses use of a molybdenum base and a commercial X-ray tube is marketed with a base of molybdenum alloyed with 5% tungsten.

In accordance with this invention, there are provided rotary targets for X-ray tubes which have improved resistance to warpage and which comprise a molybdenum base body alloyed with a stabilizing proportion of iron, silicon, cobalt, tantalum, niobium, hafnium, stable metal oxide or mixture of the preceding.

The present invention will be further described, by way of example only, with reference to the accompanying drawing which is an elevation view, in cross-section, of a disk assembly.

Referring now to the drawing, there is shown an anode assembly 10 suitable for use in a rotating X-ray anode tube. The anode assembly 10 includes a disk 12 joined to a stem 14 by suitable means such, for example, as by diffusion bonding, welding, mechanical joinder and the like. The disk 12 comprises a molybdenum substrate 15 which is alloyed with a stabilizing proportion of iron, cobalt, tantalum, niobium, hafnium, stable metal oxide or mixture of the preceding. What is meant by stable is that the metal oxides do not decompose, volatilize or grow in particle size to any appreciable extent in the sintering and other heat treatments to form the rotary target and during its service and less than that which results in warpage beyond tolerable limits. Exemplary of suitable stable metal oxides are thorium oxide, zirconium oxide, titanium dioxide, aluminum oxide, magnesium oxide, silicon dioxide, yttrium oxide, cerium oxide and the rare earth metal oxides such as La_2O_3, Nd_2O_3, and Pr_6O_{11}. A stabilizing proportion of the alloying material or materials is enough sufficient to inhibit or reduce warpage of the focal track. The amount will depend on the materials employed, but generally from about 0.05% to about 10% is sufficient.

An anode target 24 is affixed to a selected surface area of the substrate 15. The material of the anode target 24 can be any suitable material such as tungsten or an alloy of tungsten and rhenium. The rhenium content can vary from about 1 up to about 25 weight percent but is typically from 3 to 10 weight percent.

Exemplary of suitable mixtures which can be employed are from 0.5 to 10% and preferably from 1.25 percent to 2.25 percent of tantalum, niobium, or hafnium with from 0.5 to 5% and preferably from 1% to 2% of yttrium oxide, or from 0.05 to 0.3% and preferably from 0.08% to 0.13% of cobalt or silicon with the aforementioned amounts of yttrium oxide or from 0.05 to 0.3% and preferably from 0.08% to 0.13 weight percent of iron with the aforementioned amounts of yttrium oxide.

In a preferred embodiment, an additional material is alloyed into the molybdenum base such as carbides of tantalum or hafnium. An amount of between 0.1 to 5% by weight of the molybdenum is sufficient.

Although the various materials alloyed with the molybdenum can have a wide particle range such as, for example, from 0.01 microns to 30 microns, it is preferred that the average particle size range be between about 0.1 and about 10 microns and that the materials be milled and preferably dry milled so as to limit agglomeration.

The following examples will serve to illustrate the invention. All parts and percentages in said examples, and elsewhere in the specification and claims, are by weight unless otherwise indicated.

EXAMPLES

Rotary X-ray targets comprising a focal track of tungsten alloyed with 5% rhenium and a molybdenum base body alloyed with a metal as reported in the following table, were prepared by the following general method in which the rotary target base body was composed of molybdenum alloyed with 1% of yttrium oxide.

A sample of 986 grams of molybdenum was blended with 9.85 grams of yttrium oxide, or a decomposable yttrium salt, such as yttrium oxalate, acetate, or nitrate, for one hour in a Patterson-Kelly twin shell blender. The mixture was then removed and dry milled in a 1 quart carbide mill for three hours. Although the ingredients can be wet milled, the mixture is preferably dry milled.

Alternatively, the alloys can be prepared by the solution method in which, such as for example, 24.9 grams of yttrium acetate dissolved in deionized water was blended with 908 grams of molybdenum. More water was added if necessary to completely cover the molybdenum and then the mixture slowly evaporated to dryness on a hotplate while constantly kneading to avoid pockets of yttrium acetate salt crystals as it precipitated out of the saturated water solution. Two pounds of the above mixture was then ball milled dry in a 1 quart carbide mill for three hours. This procedure results in a finely divided oxide phase uniformally distributed in the molybdenum matrix. Iron and cobalt is desirably added as a decomposible, reducible salt in a manner similar to the yttrium salts although the powder metal can be employed. Hafnium is desirably added as a hydride powder.

X-ray rotary targets were prepared by heating the powder to decompose and reduce it and then the tungsten-rhenium focal track layer was placed in a compacting die, the mixture leveled and one of the invention base layers placed on top of the focal track layer. The base layer was then leveled and the composite pressed and sintered.

Representative examples of the invention were tested by measuring the warpage after being subjected to 52 Ciné exposures, namely 200 mA at 100 kVp for 8 seconds every 3 minutes. This is a severe exposure that will produce excessive warpage in all targets tested up to this time. In the following Table I, four compositions of the invention are compared with two commercial compositions 5 and 6. Example 1 is seen to be more than 10 times better than commercial composition 5 and all of examples 1 through 3 are seen to be at least five times better than commercial composition 6. Example 4 of the invention is seen to be at least twice as good as either of the commercial compositions with respect to warpage.

TABLE I

SUBSTRATE 11° WING TYPE TARGETS WARP TESTS ON MOLYBDENUM ALLOY

TUBE EXT. NO.	TARGET SUBSTRATE	*52 CINÉ WARP TEST DEGREES
1	125% Co/Mo	−0.30°
2	125% Co/Mo	−0.45°
3	1.0% Y_2O_3/Mo	−0.48°
4	1.25% Ta/Mo	−1.25°
5	100% Mo	−3.1° to 3.5°
6	5% W/Mo	−2.6°

*10,000 rpm, 100 KVP–200 mA–8 second exposures, one exp. every 3 minutes

To demonstrate the effectiveness of other alloys, tensile tests were conducted on rods formed from the alloys. The alloys were prepared by mixing the alloying elements (listed in Table II) with molybdenum, pressing and sintering rods from the powders and then hot swaging to densify. The rods, barbell in shape with a 0.1" diameter in the middle, were annealed at 1650°C for 1/2 hour and then pulled in tension in vacuum at 1100°C employing an Instron tension tester. The yeild strength, ultimate strength, total elongation and average reduction in area of the diameter are shown in Table II for the alloys of the invention and for the prior art unalloyed molybdenum, and molybdenum alloyed with 5% tungsten. A minimum of two rods were tested of each composition and the tensile results averaged.

TABLE II
Tensile Data on Bars at 1100°C

Composition	0.2% Y.S.-ksi	U.T.S. ksi	% Total Elong.	Percent Reduction in area
Unalloyed Mo	5-7	9-12	33	85
1-1/4% Ta	8.7	18	45	70
2-1/4% Ta	10	19	28	80
1% Y_2O_3	7.6	14	45	88
0.125% Co	14	26	69	77
0.125% Fe	11	19	43	90
0.25% Fe	14.5	25	60	85
0.1% Si	15.5	25	55	85
0.9% Hf	10	20	10&24	35&40
5% W	5.7	13.5	45	90
1/2% Y_2O_3	7	13.5	50	85
0.55% MgO	6-6.7	12-13.8	25-35	85
1% HfC	41	48	8.8	55
*0.085% Fe + 1% Y_2O_3	9.8	12.8	37	77

*tested at 1350°C

From an examination of the data reported in Table II, it can be seen that the compositions of the invention have both improved yield strength and ultimate tensile strength over the prior art compositions.

CLAIMS

1. A rotary target for X-ray tubes comprising a molybdenum base body alloyed with a stabilizing proportion of iron, silicon, cobalt, tantalum, niobium, hafnium, stable metal oxide or mixture thereof.

2. The target as claimed in claim 1 wherein the molybdenum is alloyed with from 0.05% to about 10% of said materials based on the weight of the molybdenum.

3. The target as claimed in claim 1 or claim 2 wherein the molybdenum of said base body is additionally alloyed with a proportion of inert carbide.

4. The target of any one of claims 1 to 3 wherein the molybdenum is alloyed with from 0.5% to 5% of yttrium oxide.

5. The target of claim 4 wherein the molybdenum is alloyed with from 1% to 2% yttrium oxide.

6. The target of any one of claims 1 to 3 wherein the molybdenum is alloyed with from 0.05% to 0.3% cobalt or silicon.

7. The target of claim 6 wherein the molybdenum is alloyed with from 0.08% to 0.125% of cobalt or silicon.

8. The target of any one of claims 1 to 3 wherein the molybdenum base is alloyed with from about 1.25% to about 2.25% tantalum, niobium or hafnium.

9. The target of any one of claims 1 to 3 wherein the molybdenum is alloyed with from 0.05 to 0.3% iron and from 0.5 to 5% yttrium oxide.

10. The target of claim 9 wherein the molybdenum is alloyed with about 0.0825% iron and about 1% yttrium oxide.

11. The target of any one of claims 1 to 10 wherein an electron impact area is applied to said base comprising tungsten alloyed with a proportion of rhenium.

12. The target of claim 11 wherein from 0.5% to 35% by weight rhenium is present in the tungsten alloy.

13. A rotary target for an X-ray tube as claimed in claim 1 substantially as hereinbefore described in the accompanying drawing.

14. A rotary target for an X-ray tube as claimed in claim 1 substantially as hereinbefore described in any one of the Examples.

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