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METHOD OF HEAT TREATING MOLYBDENUM AND MOLYBDENUM ALLOYS

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This invention relates to a new method of heat treating molybdenum and its alloys.

It has been found that molybdenum and its alloys are more easily fabricated and produce a more desirable finished product with respect to such properties as hardness, ductility and tensile strength, if the metal, prior to fabrication, has a high heat treated density, preferably above 10.00 gms./cc., and has a relatively low grain count, preferably below 5000 grains per square millimeter, as determined by standard procedure. Fabricating data indicate that grain counts below 5000 grains/mm.^{2} are extremely desirable. Ingots having a heat treated density of over 9.8 grams per cc. have not generally been produced in production quantities prior to this time, and particularly ingots of large cross-sectional dimension.

It is therefore an object of this invention to provide a method of heat treating molybdenum and its alloys which produce a fully coalesced material which is easily fabricated, has a high heat treated density and has a comparatively low grain count.

It is another object of this invention to provide a method of heat treating molybdenum and its alloys which produces a product having suitable hardness properties.

It has been found that the above and other objects are accomplished by heating the metal over a two weeks' period and including the following steps in the schedule: During the first week heating is applied between 1200–1250° C. over a period of 20 to 40 hours followed by a soaking period of 20 to 50 hours between 1600–1650° C. For about the last five hours of the first week of treatment the metal is heated at a temperature of 1700° C. or slightly over. During the second week of treatment the metal may be heated to 1600° C. within about 20 hours, then the temperature is raised slowly to 1700° C. and maintained above 1700° C. for about the last five hours.

Use of the above heat treatment schedule produces a

product having an average density of over 10.00 gms./cc. for production loads of molybdenum and molybdenum alloy containing up to .3% cobalt. For production loads the minimum soaking time is twenty hours at 1600–1650° C. during the first week of treatment to insure density values of 10.00 gms./cc. or better. There are two very critical temperatures during the heat treatment of the larger ingots. The first is a slow heating through the range of 1200–1250° C. to minimize cracking due to the fact that the rate of linear shrinkage is maximum in this range. The second is a very slow heating between 1600–1650° C., since if the ingot temperature exceeds 1650° C. too rapidly the density value will not reach 10.00 gms./cc. regardless of subsequent treatment. Smaller ingots can be heated to 1600° C. in swaging type furnaces as rapidly as the capacity of the power supply permits.

The second week of treatment both increases the density and reduces the grain count. Treatment of molybdenum alloy containing various percentages of cobalt according to the two week schedule produced average grain counts below 5000 grains per square millimeter and average densities of over 10.00 gms./cc.

The results on five production furnace loads obtained with the process of this invention are given in the following table. The procedure used to obtain the results on Load No. 1 was as follows: During the first week, the rate of heating through 1200° C. was slow, followed by a long soaking period of 30–40 hours between 1600–1650° C., with the temperature raised to 1700–1730° C. the last four or five hours. During the second week a soaking period of over 40 hours between 1600–1650° C. was used followed by 15 hours of heating between 1700–1750° C. For the results on Load No. 2 the same schedule as that for the results of Load No. 1 was used with the exception that the temperature used for the last four hours of the second week was slightly lower than for the same period of the first week. For the results of Load No. 3 essentially the same schedule used to obtain the results on Load No. 1 was used with the exception that the total time over 1700° C. was approximately 10 hours. For the results on Load No. 4 the temperature of the ingots was never above 1600° C. the first week and passed through the range of 1600–1650° C. in three hours during the second week. The results on Load No. 5 are for a one week treatment of molybdenum alloy containing .1% cobalt. The alloy was treated in a small swaging furnace built to operate at temperatures over 1900° C. The bars were subjected to a soaking period of 22 hours between 1600–1650° C., followed by 18 hours' treatment at 1750° C. and 4 hours at 1900° C.

LOAD NO. 1 (TOTAL LOAD 347.05 LBS.)

Sample Number	Percent Cobalt	Wt., Lbs.	Furnace Time, Wks.	Grain Count [1]	Density, gms./cc.	Average Density, gms./cc.	Hardness (V. P. N.) [2]
1	.035	74.25	1		10.18	10.03	164
2	.2	14.6	1		10.17	9.98	216
3	.2	14.6	1		10.11	9.97	216
1 wk. sample—Average					10.15	9.99	198.66
4	.1	97.4	2		10.10	9.89	163
5	.2	14.6	2		10.01	10.08	219
6	.2	14.6	2		10.20	10.14	219
7	.2	14.5	2		10.06	10.14	220
8	.2	14.6	2		10.04	10.14	220
9	.2	14.8	2		10.07	10.16	221
10	.2	14.7	2		10.20	10.13	218
11	.2	14.6	2		10.12	10.11	220
12	.2	14.6	2		10.19	10.11	225
13	.2	14.6	2		9.82	10.01	218
14	.2	14.6	2		10.12	10.05	220
2 wk. sample—Average					10.08	10.08	214.8

LOAD NO. 2 (TOTAL LOAD 307.1 LBS.)

Sample Number	Percent Cobalt	Wt., Lbs.	Furnace Time, Wks.	Grain Count [1]	Density, gms./cc.	Average Density, gms./cc.	Hardness (V. P. N.) [2]
1	.1	70.6	1	10,640; 13,105	9.81	9.92	175
2	.1	157.3	1	8,465; 9,850	10.00	9.88	
3	.1	79.2	1	8,580; 10,445	10.07	10.00	178
Average (1 wk.)				10,180.8	9.96	9.93	176.5
1	.1	70.6	2	4,485; 6,600	10.11	10.06	169
2	.1	157.3	2	2,600; 4,300	10.10	10.05	166
3	.1	79.2	2	3,400; 5,570	10.09	10.09	173
Average (2 wk.)				4,492.5	10.10	10.07	169.5

LOAD NO. 3 (TOTAL LOAD 385 LBS.)

1	.3	15.4	2	5,400	10.09	10.12	229
2	.3	15.4	2	5,350	10.15	10.01	229
3	.3	15.4	2	5,450	10.15	10.03	255
4	.3	15.4	2	4,560	10.15	10.04	237
5	.3	15.4	2	4,340	10.15	10.02	222
6	.3	15.4	2	5,910	10.11	10.00	229
7	.3	15.4	2	5,380	10.19	9.99	226
8	.3	15.4	2	4,000	10.18	9.98	220
9	.3	15.4	2	5,190	10.19	10.01	226
10	.3	15.4	2	4,400	10.20	10.03	230
Average (.3% Co)				4,998	10.156	10.023	230.3
11	.1	15.4	2	4,220	10.12	10.06	175
12	.1	15.4	2	4,500	10.15	10.08	180
13	.1	15.4	2	3,540	10.17	10.05	180
14	.1	15.4	2	4,050	10.20	10.06	178
15	.1	15.4	2	5,570	10.18	9.98	185
16	.1	15.4	2	4,110	10.15	10.00	187
17	.1	15.4	2	4,780	10.18	10.15	192
18	.1	15.4	2	2,870	10.18	10.13	185
19	.1	15.4	2	3,880	10.19	10.09	185
20	.1	15.4	2	2,200	10.19	10.14	181
21	.1	15.4	2	3,430	10.17	10.16	184
22	.1	15.4	2	4,900	10.19	10.03	180
23	.1	15.4	2	4,390	10.19	10.12	183
24	.1	15.4	2	5,800	10.15	10.04	180
25	.1	15.4	2	3,820	10.13	10.09	169
Average (.1% Co)				4,137.3	10.17	10.08	181.6
Average (All samples)				4,481.6	10.16	10.05	201.01

LOAD NO. 4 (TOTAL LOAD 302 LBS.)

1	.05	151.0	2	1,969; 6,356	10.19	9.88	
2	.05	151.0	2	1,406; 4,894	9.96	9.88	
Average				3,656	10.09	9.88	

LOAD NO. 5

1	.1	12.5	1	4,305; 4,905	10.07	10.07	169
2	.1	13.8	1	4,765; 4,905	10.06	10.09	161
Average				4,720	10.065	10.08	165

[1] Center edge (grains/mm.²).
[2] Vickers Pyramid number.

Referring to the table covering the results on Load No. 1, a typical production load of 347.05 pounds of molybdenum-cobalt alloys of .035%, .1% and .2% cobalt, it is noted that those bars having only one week of treatment had an average density of 9.99 gm./cc. and those subjected to two weeks of treatment had an average density of 10.08. The average density for the entire load including all samples was 10.07 (not shown). The density values after two weeks of treatment were not dependent upon location in the furnace, i. e., uniform density throughout the furnace was obtained. The average hardness value for the ingots treated two weeks was 214.8 as compared to 198.66 for the load treated only one week. The average hardness value for molybdenum alloy samples containing .2% cobalt was 220 after two weeks of treatment as compared to an average value of 216 for samples of this alloy treated only one week and the average density of .2% cobalt alloy samples after one week of treatment was 9.975 as compared to an average density of 10.10 for .2% cobalt samples treated two weeks (not shown in the table).

The results obtained on Load No. 2, a total load of 307.1 pounds of molybdenum alloy containing .1% cobalt, show an average grain count of 10180.8, an average density of 9.93 and an average hardness value of 176.5 after the first week of treatment as compared to an average grain count of 4492.5, an average density of 10.07 and an average hardness value of 169.5 after two weeks of treatment.

Referring to the results obtained on Load No. 3, 10 samples of .3% cobalt alloy and 15 samples of .1% cobalt alloy comprising a production load of 385 pounds, it is seen that for all samples an average grain count of 448.6, an average density of 10.05 and an average hardness value of 201.01 was obtained. For the .3% cobalt samples the average grain count was 4998, the average density was 10.02 and the average hardness value was 230.3 as compared to an average grain count of 4137.3, an average density of 10.08 and an average hardness value of 181.6 for the .1% cobalt alloy.

Load No. 4 consisted of two 151 lb. ingots of .05% cobalt alloy. A two weeks' heating schedule gave an average grain count of 3656 and an average density of 9.88.

Load No. 5 consisted of two small ingots of .1% cobalt alloy. The average grain count was 4720 which was slightly higher than that obtained after two weeks of treatment at lower temperatures. An average density of 10.08 gms./cc. was obtained with an average hardness value of 165.

As the above results show, furnace loads of over 370 pounds and ingots weighing up to 157 pounds have been successfully treated by the process of the invention to yield average densities over 10.00 gm./cc. and average grain counts below 5000 grains per square millimeter. Prior to this invention these values have not been obtained on bars as large as four and six inches square. Over 1500 pounds of molybdenum and molybdenum-cobalt alloys were treated in accordance with the process of this invention over a period of three months with the result that an overall average density of 10.04 gms./cc. was obtained. The loads treated included ingots weighing as much as 157.3 pounds and up to four and six inches square.

While there has been disclosed the preferred embodiments of my invention it is obvious that many modifications and variations of the invention are possible in the light of the above teachings. It is therefore to be understood that within the scope of the appended claims the invention may be practiced otherwise than as specifically described.

What is claimed is:

1. In the process of heat treating molybdenum and molybdenum-cobalt alloys containing up to approximately .3% cobalt the improvement which comprises heating the metal at temperatures up to 1900° C. over a period of two weeks with soaking periods each week of from about twenty to about forty hours between about 1200 and 1250° C. and about twenty to about fifty hours between about 1600 and 1650° C.

2. In the process of heat treating molybdenum and molybdenum-cobalt alloys containing up to approximately .3% cobalt, the improvement which comprises including in the heat treating schedule at least one soaking period of about 20 to 40 hours between about 1200 to 1250° C., the temperature being gradually raised throughout said period.

3. In the process of heat treating molybdenum and molybdenum-cobalt alloys containing up to approximately .3% cobalt, the improvement which comprises including in the heat treating schedule at least one soaking period of about 20 to about 50 hours between about 1600 to about 1650° C., the temperature being gradually raised throughout said period.

4. In the process of heat treating bolybdenum and molybdenum-cobalt alloys containing up to approximately .3% cobalt, the improvement which comprises including in the heat treating schedule at least one soaking period of about 20 to 40 hours between about 1200 to 1250° C. and at least one soaking period of about 20 to about 50 hours between about 1600 to 1650° C., the temperature being gradually raised during each of said soaking periods.

5. The process of heat treating molybdenum and molybdenum-cobalt alloys containing up to approximately .3% cobalt, the improvement which comprises heating the metal at temperatures up to about 1900° C. within a two week period according to the following schedule: Bringing the temperature to about 1200° C., raising the temperature gradually over a period of about 20 to 40 hours between about 1200 and 1250° C., bringing the temperature slowly to about 1600° C., raising the temperature gradually from 1600 to 1650° C. over a period of between about 20 to 50 hours, raising the temperature to about 1700° C., heating at a temperature above 1700° C. for a period of between about 4 and 20 hours and repeating the schedule for the second week.

6. The process of heat treating molybdenum and molybdenum-cobalt alloys containing up to approximately .3% cobalt which comprises heating the metal over a two weeks' period at temperatures up to about 1750° C. which comprises the steps of bringing the metal to a temperature of about 1200° C., raising the temperature gradually over a period of about 20 hours between 1200 and 1250° C., raising the temperature slowly to about 1600° C., raising the temperature gradually from 1600 to 1650° C. over a period of about 40 hours, raising the temperature to 1700° C. and heating at a temperature from about 1700 to 1750° C. for a period of about 10 hours and repeating the above steps the second week.

7. In the process of heat treating molybdenum and molybdenum-cobalt alloys containing up to approximately .3% cobalt, the improvement which comprises heating the metal for at least 20 hours between about 1600° C. and about 1650° C. with a gradual increase in temperature.

8. The process of heat treating molybdenum and molybdenum-cobalt alloys containing up to .3% cobalt which comprises heating the metal over a period of two weeks and including in the schedule the steps of heating the metal during the first week of treatment for a period of about 20 hours between 1200–1250° C., for at least 20 hours between 1600–1650° C. and for a period of about 5 hours at a temperature above 1700° C.; during the second week of treatment heating the metal to 1600° C. within 20 hours, heating between 1600–1650° C. for at least 20 hours and heating for at least 5 hours above 1700° C.

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