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URANIUM ALLOY DEVELOPMENT FOR NON- NUCLEAR APPLICATION PROGRESS REPORT NO. 4

by

H. M. SKELLY, C. F. DIXON AND N. S. SPENCE

PHYSICAL METALLURGY DIVISION

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URANIUM ALLOY DEVELOPMENT FOR NON-NUCLEAR APPLICATION

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H.M. Skelly*, C.F. Dixon** and N.S. Spence***

SUMMARY OF RESULTS

A heat treatment procedure was developed for uranium - 2% molybdenum alloy which hardens 2-inch thick sections to 400-590 VHN. The hardness at the centre of the 2-inch sections is about 400 VHN and it increases to 500-590 VHN at the surface.

The heat treatment consists of heating the alloy to above the temperature at which it transforms to the gamma phase (about 700°C (1290°F)), cooling rapidly to 300-400°C (570-750°F), and finally water quenching or cooling in air.

The following heat treatment procedure produced a surface hardness of up to 528 VHN: the sample was coated with colloidal graphite and heated at 755°C (1390°F) for 1/2 hour in argon; the sample was then transferred quickly to a molten lead-antimony bath and held at 405-430°C (760-805°F) for 15 minutes and finally air-cooled from 405°C (760°F).

Spontaneous breaking-up of two samples after hardening was attributed to high internal stress resulting from the heat treatment. Such an occurrence could be a problem in heat treating thick sections of the alloy.

The mechanical properties of the alloy, both as-cast and heat treated, were not outstanding, the ductility in particular being very poor.

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INTRODUCTION

This report describes work which has been carried out as part of a program aimed at developing uranium-base alloys of high strength for non-nuclear applications. Three earlier Progress Reports (1, 2, 3) dealt mainly with the preparation, properties and heat treatment of laboratorysize melts of experimental alloys. This Progress Report describes work carried out on the heat treatment and properties of uranium-2% molybdenum alloy material that had been prepared on a production scale. The main purpose of the work described in this report was the development of heat treatments that would harden comparatively thick sections (about 2 inches diameter) of uranium-2% molybdenum alloy.

TEST MATERIAL

Two kinds of sample were received for testing -- as-cast billets and machined specimens. The billets had the original cast surface on them and measured 2 inches diameter by 6 inches long; they are identified as NM-2-1, 2, 3, 4, 6, 7 and 8. The machined specimens (also as-cast) had been turned to close tolerances from the same size of billet, and measured 1-3/4 inches diameter by 6 inches long and were tapered at one end; these specimens are identified as NM-3-1, 2, 3, 4 and 5.

Complete casting details for the billets are not known, but they were cast in vacuo into graphite moulds and then cut into 6-inch lengths. All samples originated with Eldorado Mining and Refining Limited, Port Hope, Ontario.

EXPERIMENTAL PROCEDURE AND RESULTS

Heat Treatment and Hardness

Two-Inch Diameter Cast Billets

Five of the seven 2-inch diameter billets were heat treated as detailed in Table 1. Billet NM-2-7 was heat treated twice. Two billets (NM-2-3 and NM-2-4) were not heat treated but were hardness-tested in the as-cast condition. Billets NM-2-7 and NM-2-8 were also hardness-tested prior to being heat treated.

The hardness measurements made on billets NM-2-1, NM-2-2, NM-2-3, NM-2-4 and NM-2-6 were carried out on a transverse section, 2-1/2 inches from one end of the billet, the measurements being made at five locations along a radius from the centre to just under the surface of the billet. In the case of the other billets, the hardness was measured on the surface only.

Billets NM-2-1, NM-2-2 and NM-2-6 were heat treated by immersion in molten salt (Houghton 980) and then in molten lead, the billets being first coated with colloidal graphite to protect them from attack by the salt and lead. The graphite-coated billets were first heated to solution heat-treating temperature in the molten salt, and after the required soaking time they were quickly transferred to molten lead-11. 2% antimony (or molten lead) held for a given time and then water-quenched. The lead-11. 2% antimony alloy, which has a melting-point of 252°C (486°F) compared to a melting-point of 328°C (622°F) for lead, was used in all heat treatments but one (i.e., NM-2-1). One billet (NM-2-2) was air-cooled after being heated in the molten salt.

On transferring the billets to the molten lead-antimony (or lead) the temperature of the latter rose, and the temperatures given in Table 1 indicate the range during this part of the heat treatment. The temperature underlined is the one from which the sample was quenched into water.

Although coating the billets with graphite was successful in protecting them from severe attack by the molten salt and the lead-antimony, there was some slight attack and, as it was required to heat treat some accurately-machined specimens without damaging them, it became necessary to devise a heat treating technique that would keep surface attack to a minimum. The first attempt at this was carried out on billet NM-2-7, which was wrapped in copper sheet and then sealed in a welded steel container; this billet was clean and unattacked after the heat treatment but, as can be seen from the results in Table 1, there was little hardening, the mean surface hardness being 370 VHN compared to 294 VHN for the ascast billet.

In the second attempt to protect the billets during heat treatment, billet NM-2-8 was wrapped in copper sheet. In this case the salt and molten lead-antimony alloy seeped through the copper sheet and contaminated the billet. The salt washed off readily, but there were patches of leadantimony alloy adhering tightly to the billet. As Table 1 shows, there was appreciable hardening of this billet, the surface hardness being 478 VHN versus 301 VHN as-cast. In the third attempt to protect the billets during heat treatment, billet NM-2-7 was given a second heat treatment in which it was coated with graphite, heated in an argon atmosphere in a specially-designed container, and then transferred to the molten lead-antimony. This billet was adequately protected, and it hardened to a mean of 570 VHN on the surface (See Table 1). Heating the billet in argon rather than in molten salt eliminated the possibility of any attack on the graphite coating, which was then able to protect the uranium alloy from attack by the lead-antimony. This billet later broke into several pieces due, presumably, to high internal stress resulting from the heat treatment.

The hardness values obtained for billets NM-2-1, NM-2-2, NM-2-3 and NM-2-6 are plotted in Figure 1 against distance from surface of the billet. It can be seen from Figure 1 that, whereas there was only slight hardening of billet NM-2-2, billets NM-2-1 and NM-2-6 were considerably hardened. It is interesting to note that there was a gradual decrease in the hardness of billets NM-2-1 and NM-2-6 from the surface to about half-way to the centre, at which point the hardness remained fairly constant through to the centre.

A hardness survey was carried out on longitudinal sections through billets NM-2-4 (as-cast) and NM-2-6 (heat treated). The hardness was uniform throughout billet NM-2-4, but there was a distinct trend in NM-2-6 towards greater hardness at the surface and the results for the latter are plotted in Figure 2. Billet NM-2-6 was sectioned longitudinally and eight hardness traverses were made on the longitudinal face. Each traverse was made along lines corresponding to diameters, starting at the surface and going through the centre to the diametrically opposite surface. The first hardness traverse was made at the surface and the other traverses were made at 1/2-inch intervals to beyond the centre-line of the billet. The top plot in Figure 2 is the traverse across the surface and the succeeding plots are the traverses made at 1/2-inch intervals to beyond the center-line. The hardness measurements were made at 1/4-inch intervals across each traverse.

It can be seen from Figure 2 that practically all the hardness values were in the range 400-500 VHN. The surface traverse exhibited uniformly high hardness and the other traverses showed decreasing hardness from the surface to about 1/2 inch below the surface, when the hardness became uniform.

Machined Samples

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The machined samples were all heat treated in the same manner as the second heat treatment given to billet NM-2-7, namely, coating with graphite and heating in argon prior to immersion in the lead-antimony. The hardness of these samples was measured on the surface in the as-received and heat treated conditions, the measurements being made along the length. As can be seen from Table 2, the as-received hardness of the samples was of the order of 310 VHN and they hardened to 486-593 VHN. These machined samples were tapered at one end and in all cases except one (NM-3-3) maximum hardening occurred at the tip of the taper.

Sample NM-3-4 was air-cooled from 405°C (760°F) after immersion in the lead-antimony bath as compared with the other samples listed in Table 2, which were water-quenched. Air cooling still resulted in hardening (mean of 516 VHN) and there was no appreciable oxidation of the sample.

Within two days of being heat treated, sample NM-3-2 started to break-up and this continued for ten days until the sample was in several pieces. As in the case of billet NM-2-7, the breaking-up was attributed to internal stress.

Mechanical Properties

To determine how the heat treatment had affected the mechanical properties of the two-inch diameter billets, tensile and compression specimens were machined from billet NM-2-3 in the as-cast condition and from billets NM-2-1, NM-2-2 and NM-2-6 after heat treating. The tensile specimens, which were machined to Physical Metallurgy Division Drawing No. 107, had a gauge diameter of 0.178 inch and a gauge length of 0.632 inch. The compressive yield strength (CYS) was determined on specimens machined to 0.5 inch diameter by 1.5 inches long and the ultimate compressive strength (UCS) was determined on specimens 0.5 inch diameter by 0.62 inch long. The tensile and compression results are listed in Tables 3 and 4, respectively. The figures in brackets indicate the number of test results averaged.

Dimensional Check

Dimensional checks were carried out on a billet and a machined specimen, before and after heat treating, to determine the magnitude of any dimensional changes that might have occurred. The billet was first machined to remove the cast surface. The dimensions were measured on a Pratt and Whitney Standard Measuring Machine.

It was found that the dimensions increased very slightly on heat treating. The maximum change observed in the billet was an increase of 0.0016 inch on the length. The change in diameter for both billet and machined specimen was in all cases less than 0.001 inch.

Density Determinations

Density determinations were carried out by water displacement on machined specimens from billets NM-2-1, NM-2-2, NM-2-3 and NM-2-6, all being in the heat treated condition except NM-2-3, which was as-cast. A density determination was also carried out on a complete billet in the as-cast condition (NM-2-8) using both water displacement and measurement methods. Details and results of these determinations are given in Table 5.

Chemical Composition

The results of chemical and gas analyses carried out on some of the test samples are listed in Table 6.

DISCUSSION

The results listed in Table 1 show that the greatest hardening obtained in the 2-inch diameter billets came from the second heat treatment given to billet NM-2-7; this billet subsequently broke up due, presumably, to residual stress. Billets NM-2-1, NM-2-6 and NM-2-8 also hardened appreciably. Billet NM-2-2, which was air-cooled directly from the solution heat treating temperature exhibited only slight hardening. It is obvious that billet NM-2-7, which hardened only slightly after the first heat treatment, was then so well-insulated in the copper sheet and welded container that there was insufficient quenching effect on immersion in the leadantimony bath.

The procedure used in the second heat treatment given to billet NM-2-7 was the one adopted for the heat treatments carried out subsequently on the machined samples.

Figures 1 and 2 show that maximum hardness in the 2-inch diameter billets was confined to the surface and the hardness gradually decreased for a distance of 1/2-inch from the surface and then remained constant. This behaviour is probably related to the comparative values of the thermal conductivity of the uranium-2% molybdenum alloy and of the rate of the transformation reaction resulting in hardening.

From the heat treatments detailed in Table 2, it is interesting to note that a treatment of 755°C (1390°F) for 1/2 hour was sufficient to produce hardening on subsequent quenching into lead-antimony (see NM-3-4). The advantage of heating to this lower temperature rather than to 800°C (1470°F) is that there is less likelihood of damage to the alloy sample, the microstructure of which changes to the soft gamma phase at about 700°C (1290°F). Another interesting point is that air-cooling from 405°C (760°F) was sufficient to induce hardening in sample NM-3-4 without causing oxidation.

The method used to heat treat the machined specimens resulted in little or no surface attack. All the specimens exhibited a "crazed" pattern on parts of the surface where there appeared to be a very slight attack; this was thought to be due to cracking of the graphite coating during heating in the argon, and the lead-antimony attacking the uranium slightly through the cracks. This attack was not considered to be serious.

The tensile properties of the as-cast 2-inch diameter billets (see Table 3) exhibited comparatively low ductility, which was further reduced on hardening the billets. Heat treating did produce an increase in tensile and yield strengths, especially in the case of NM-2-6, which was the hardest of those tested.

There was an appreciable increase in the compressive strength of the billets after heat treating (see Table 4), but only the hardest (NM-2-6) exhibited a definite ultimate compressive strength, the others being flattened.

The dimensional checks carried out showed that the changes in dimensions brought about by the heat treatment were not large enough to present a problem.

The density results listed in Table 5 indicate a slightly greater density for heat treated material, but more work would have to be done to check this. Apart from this slight variation, the values obtained are consistent and normal.

The chemical analyses (Table 6) showed that the carbon content of billet NM-2-3 was exceptionally high, and if this high carbon is typical of this batch of material, it would help to explain the low ductility in the ascast billets. The hydrogen, oxygen and nitrogen contents are satisfactorily low.

As already mentioned, one of the cast billets and one of the machined specimens broke into several pieces over a period of days after being heat treated. This spontaneous breakage, which was probably a result of internal stress arising from the heat treatment, could be a problem in hardening thick sections of the uranium-2% molybdenum alloy.

CONCLUSION

Two-inch thick sections of uranium-2% molybdenum alloy can be hardened to 400-500 VHN. Hardness as high as 590 VHN can be obtained on the surface of the sections, but the hardness drops to about 400 VHN at the centre.

Spontaneous breaking of hardened uranium-2% molybdenum alloy specimens of about 2-inch thickness could be a problem in future development of the alloy for non-nuclear application.

ACKNOWLEDGEMENTS

Thanks are extended to the Chemical Analysis Section of the Extraction Metallurgy Division for carrying out the molybdenum and carbon analyses.

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- N.S. Spence, H.M. Skelly and C.F. Dixon, "Uranium Alloy Development for Non-Nuclear Application. Progress Report No. 2", Mines Branch Investigation Report IR 64-40 (May 4, 1964).
- 3. H.M. Skelly, C.F. Dixon and N.S. Spence, "Uranium Alloy Development for Non-Nuclear Application. Progress Report No. 3", Mines Branch Investigation Report IR 65-75 (September 15, 1965).

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Heat Treatment and Hardness of 2-Inch Diameter Cast Billets (Uranium - 2% Mo Alloy)

Sample Ident.	Condition	Vickers Hardness No. (20 kg load)
NM-2-1	765°C (1410°F) for 1-1/2 hr, then into Pb bath of <u>400</u> -443°C (750- 830°F) for 20 min W.Q.	Outside
NM-2-2	800°C (1470°F) for 1 hr. Air cooled	325-314-312-312-312
NM-2-3	As-cast	305-298-297-292-302
NM-2-4	As-cast	289-286-298-298-288
NM-2-6	800°C (1470°F) for 1 hr, then into Pb/Sb at <u>300</u> -360°C (570-680°F) for 15 min. W.Q.	490-455-422-424-430 <u>Surface</u>
NM-2-7 NM-2-7 (lst heat treatment)	As-cast 805°C (1480°F) for 1 hr, then into Pb/Sb at 350- <u>400°C</u> (660-750°F) for 35 min. W.Q.	292-297 360-380
NM-2-7 (2nd heat treatment) NM-2-8	805°C (1480°F) for 1 hr, then into Pb/Sb at <u>300</u> -350°C (570-660°F) for 50 min. W.Q. As-cast	566-575 298-304
NM-2-8	805°C (1480°F) for 1 hr, then into Pb/Sb at 300- <u>350°C</u> (570-660°F) for 35 min. W.Q.	473-483

Heat Treatment and Hardness Results for Machined Samples (1-3/4 in . dia x 6 in.) (U-2% Mo Alloy)

Sample Ident.	Condition	Vickers Hardness No. (20 kg load)
NM-3-1	As-cast	308-315
NM-3-1	805°C (1480°F) for 1/2 hr, then into Pb/Sb at <u>300-330°C (570-625°F)</u> for 20 min. W.Q.	505-524
NM-3-2	As-cast	308 - 31 3
NM-3-2	805°C (1480°F) for 1 hr, then into Pb/Sb at <u>300</u> -330°C (570-625°F) for 40 min. W.Q.	505-528
NM-3-3	As-cast	313-315
NM-3-3	805°C (1480°F) for 1/2 hr, then into Pb/Sb at <u>405</u> -430°C (760-805°F) for 15 min. W.Q.	520-564
NM-3-4	As-cast	312-315
NM-3-4	755°C (1390°F) for 1/2 hr, then into Pb/Sb at <u>405</u> -430°C (760-805°F) for 15 min. <u>Air cooled.</u>	505-528
NM-3-5	As-received	293-301
NM-3-5	805°C (1480°F) for 1 hr, then into Pb/Sb at 305- <u>345°C</u> (580-655°F) for 35 min. W.Q.	486-593

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Tensile Properties of Two-Inch Diameter Billets (U-2% Mo Alloy)

Sample Ident.	Condition	UTS (ksi)	0.1% YS (ksi)	0.2% YS (ksi)	%, El (4 VA)	%, RA
NM-2-3 NM-2-1 NM-2-2 NM-2-6	As-cast HT* HT* HT*	118(4) 142(3) 134(4) 178(1)	93(4) None(4) 121(4) 161(1)	101(4) None(4) 132(2) 171(1)	4.5(4) 1.0(4)	2(4) 0(4) 0(4) 2(1)

* Heat treated - see Table 1 for details.

TABLE 4

Compression Properties of Two-Inch Diameter Billets (U-2% Mo Alloy)

Sample	Condition	UCS	0.1%	•0.2%
Ident.		(ksi)	CYS(ksi)	CYS(ksi)
NM-2-3	As-cast	None(4)	54(4)	72(4)
NM-2-1	HT*	None(4)	135(4)	153(2),
NM-2-2	HT*	None(4)	103(2)	118(2)
NM-2-6	HT*	316(3)	164(3)	184(3)

* Heat treated - see Table 1 for details.

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Results of Density Determinations (U-2% Mo Alloy)

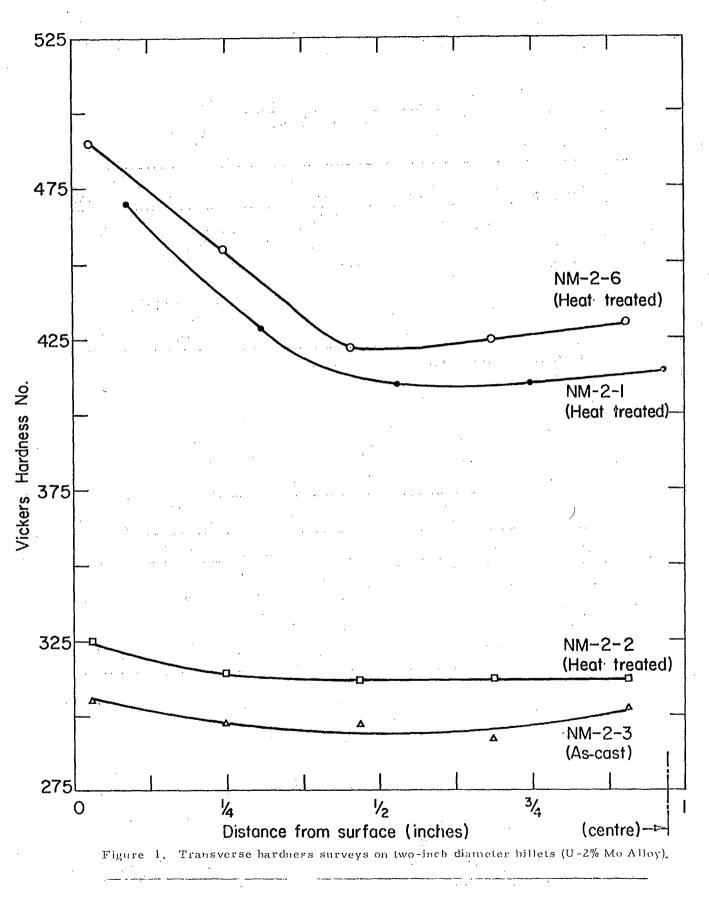
Sample Ident.	Sample Size and Condition	Method	Density (g/cc)
NM-2-1 NM-2-2 NM-2-3 NM-2-6 NM-2-8	1/2 in. dia x 1-1/2 in. H.T.* """H.T.* """"As-cast """H.T.* 2 in. dia x6 in. As-cast	Displacement "" " Displacement and Measurement	18.50 18.52 18.36 18.38 18.31

* Heat treated - see Table 1 for details.

TABLE 6

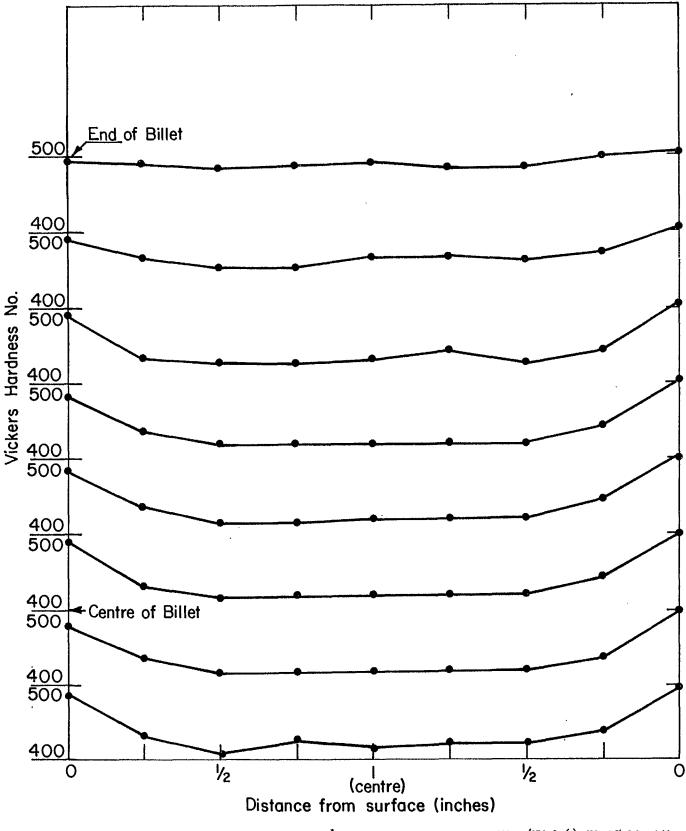
Chemical Composition of Test Material

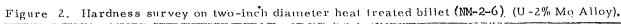
Sample	%	ppm			
Ident.	Mo	С	H ₂	0 ₂	N ₂
NM-2-3 NM-3-2 NM-3-5	2.02 2.02 2.06	1600 - -	0.70 0.65 0.60	52 35 50	30 25 25



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