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⑥ CHARACTERIZATION OF THE HEXAGONAL-CLOSE-PACKED
INTERMETALLIC COMPOUND FROM THE Mo-Rh,
Mo-Ir, AND Mo-Pt SYSTEMS.

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CHARACTERIZATION OF THE HEXAGONAL-CLOSE-PACKED INTERMETALLIC COMPOUND FROM THE Mo-Rh, Mo-Ir, AND Mo-Pt SYSTEMS

ABSTRACT

The results of an investigation of the hexagonal-close-packed intermetallic compound found in the Mo-Rh, Mo-Ir, and Mo-Pt systems is reported. The compound was characterized by determining room and elevated-temperature hardness values, room-temperature electrical resistivity, oxidation resistance during exposure to air at 538 C (1000 F) and 982 C (1800 F), and corrosion resistance to aqueous solutions containing 41.6 or 70.9% nitric acid. The room-temperature hardness of the compound is reported to range between 300 and 675 DPH and is inversely proportional to the molybdenum content of the alloy. The molybdenum-iridium alloys are harder than the sapphire indentor at test temperatures between 1093 and 1649 C (2000 and 3000 F). The oxidation resistance of the alloys at 538 C (1000 F) decreases as the molybdenum content increases. Only the 80% Rh - 20% Mo alloy resisted oxidation at 982 C (1800 F) during a 10-hour exposure. All the alloys resist corrosion by dilute and concentrated nitric acid.

INTRODUCTION

The search for structural materials for elevated-temperature service has centered around refractory metal systems. Most of the investigations in this search have been concerned with the primary solid solutions of columbium, tantalum, tungsten, and molybdenum, and have yielded important commercial alloys such as the molybdenum-base alloys TZM and TZC. The Climax Molybdenum Company has a continuing interest in this search for new alloys, and believes that progress might be made by pursuing a study of a different class of materials than the primary solid solutions cited above. One such class of materials, which has not received significant attention, is the intermetallic compound in the binary systems containing molybdenum and an element from the platinum-group metals* rhodium, iridium, and platinum that has a hexagonal-close-packed (hcp) crystal structure. The melting temperature for this compound ranges between 1900 and 2200 C (3452 and 3992 F), high enough to make it a candidate for elevated-temperature service. Melting temperature is a necessary, but not sufficient condition for an elevated-temperature material. It is known, however, that the platinum-group metals markedly increase the hardness of molybdenum.[1,2] Thus, it was concluded that the refractory metal compounds warrant further investigation. This report presents the results of an investigation of the preparation and characterization of the hcp intermetallic compound found in the Mo-Rh, Mo-Ir, and Mo-Pt systems.

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* The term "platinum-group metals," which is used throughout this report, includes only the three elements rhodium, iridium, and platinum. A general classification for the alloys of this investigation is the molybdenum-platinum group metal alloys; a term that is also used in the report.

A critical review of the literature revealed that little is known about the hcp compound found in the molybdenum-platinum group metal systems. More importantly, the phase diagrams for these systems are still not completely defined. For example, Raub,[3] Greenfield and Beck,[4] and Knapton[5] attempted to identify the phases occurring in the binary systems of molybdenum and the platinum group metals including rhodium and iridium, as well as platinum itself. All reported the existence of a phase having a hcp crystal structure and a wide range of solid solubility. "Epsilon" became the accepted designation for this phase because of the similarity with the epsilon phase in the Cu-Zn system. Anderson and Hume-Rothery confirmed the existence of the epsilon phase in their study of the Mo-Rh system,[6] as did Michalik and Brophy for the epsilon phase in the Mo-Ir system.[7] Jaffee et al.[8] concluded, on the basis of their own phase-diagram investigations, as well as the investigations cited here, that the hcp crystal structure may occur if the average group number for the alloy is between 6.8 and 8.5.

More definitive studies of the Mo-Pt system have been made by Maldonado and Schubert,[9] Rooksby and Lewis,[10] Selman,[11] and Ocken and Van Vucht.[12] Giessen et al.[13] have reinvestigated the Mo-Ir and Mo-Rh systems. These investigations showed that the epsilon phase reported by others cannot be characterized simply as having a hexagonal crystal structure and a wide range of solid solubility. In particular, the Mo-Pt system contains four intermediate compounds approximated by Mo_4Pt, Mo_3Pt_2, $MoPt$, and Mo_2Pt_3.[12] (The Mo_3Pt_2 compound is an ordered hcp phase of limited solid solubility and not a disordered epsilon compound as suggested previously.) Giessen et al.[13] found that the epsilon phase in the Mo-Rh system decomposes at 1100 C (2012 F) to an ordered orthorhombic phase having a composition of MoRh. Giesson and co-workers also reported that the iridium-rich epsilon phase is better described as $MoIr_3$ with an ordered hcp crystal structure. A MoIr compound with an ordered orthorhombic crystal structure is also reported. This phase transforms to the disordered epsilon phase above 1610 C (2930 F). These investigators argued that, by analogy, $MoRh_3$ should exist as an ordered hexagonal phase. It would not be possible to detect ordering in this system, however, because of the small difference in the atomic scattering factor between molybdenum and rhodium. Phase diagrams based on all the published data, including the inferences by Giesson et al.[13] on the Mo-Rh system, are presented in Figures 1, 2, and 3.

The goal of the research program, undertaken by the Research Laboratory of the Climax Molybdenum Company of Michigan, was to characterize single-phase samples of the hexagonal intermetallic compounds defined in the phase diagrams of Figures 1, 2, and 3. The samples were characterized by determining the hardness of the alloys at room and elevated-temperatures, the resistance to oxidation at 540 C (1000 F) and 980 C (1800 F), the resistance to corrosion in oxidizing acids, and room-temperature electrical resistivity. The characterizations were made on several compositions within the limits of the hcp compound. The selection of the compositions was complicated, however, by the ambiguity in the phase diagrams. Therefore, a few selected compositions outside the anticipated hcp phase boundaries were included in the preliminary stages of the program. The compositions selected for study from each system are shown by circled crosses on the phase diagrams. The compositions of the alloys are presented in the procedures section of this report.

EXPERIMENTAL PROCEDURES AND RESULTS

The high inherent cost of the raw materials dictated a prudent approach to sample preparation. It was decided, therefore, to cast a single, small button of each composition that would be just large enough to provide a single hardness sample. These samples would be used later for the oxidation and corrosion testing. It was found that 30 grams of each alloy was sufficient for the characterization program outlined previously. Casting was selected as the procedure for consolidation of the alloys to circumvent the problems of voids that might remain in the samples after a pressing-and-sintering operation. The disadvantage of casting the alloys is that contaminants, such as oxides, agglomerate into small inclusions instead of remaining as uniformly dispersed, fine particles in the matrix. The inclusions are generally found along grain boundaries and thus, increase the brittleness of the material.

The alloys chosen for this study were cast in a water-cooled copper mold that had a rectangular cross section approximately 1 by 0.38 by 0.2 inch. Each button was nonconsumably arc-cast three times, turning top-for-bottom between each remelt. The melting was performed under a slight positive pressure of argon purified by melting a Mo-Ti-Zr getter button prior to casting the experimental alloys.

The 30-gram cast buttons were cut and ground to the exact size of the hardness sample, 0.8 by 0.36 by 0.2 inch. One end of the button was removed prior to testing, and was then annealed and submitted for metallographic examination. Each alloy was submitted for X-ray diffraction analysis and then annealed to reduce coring and develop the equilibrium crystal structure for the particular alloy. The alloys were heat-treated at 1400 C (2552 F) for 10 hours under a pressure of 5×10^{-5} torr, except for three alloys that were heat-treated in hydrogen for 10 hours. The 1400 C (2552 F) temperature was chosen since it was high enough to order the atomic arrangement, but not so high as to greatly increase the as-cast grain size. The heat-treated samples were resubmitted for X-ray diffraction analysis. The compositions* chosen for this program, and the crystal structure in the as-cast as well as the heat-treated condition, are presented in Table 1. The density of the hcp alloys was calculated from the X-ray diffraction data. Only those alloys that had a hcp crystal structure after heat-treatment were studied further.

All the alloys with the hexagonal crystal structure were submitted for metallographic examination. As described above, a small piece of each button not required for the hot-hardness sample was examined. All the alloys were polished in the conventional manner. Etching the alloys with aqua regia, aqua regia plus HF, and Murakami's etch proved unsuccessful. An electrolytic etching procedure using an electrolyte of 20% hydrochloric acid saturated with sodium chloride was found to etch the platinum alloys. The anisotropy of the hcp crystal structure makes it possible to view the microstructure with dark field illumination using polarized light.

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* weight percent unless indicated otherwise.

Table 1

X-Ray Diffraction Analyses of Several Mo-Pt, Mo-Rh, and Mo-Ir Samples

Heat No.	Aim Composition (weight percent)		Average Group Number	Density[a] (g/cm³)	X-Ray Diffraction Analysis			
					As-Cast		After Heat Treatment[b]	
	Mo	X			Phases	c/a	Phases	c'/(a'/2)
X = Rhodium								
H-205	20 (21.1)[c]	80 (79.8)	8.5	12.2	hcp a = 2.716, c = 4.367	1.608	Unchanged	
H-218	28.5 (30.0)	71.5 (70.0)	8.1	11.7	hcp a = 2.737, c = 4.365	1.595	Unchanged	
H-219	38.3 (40.0)	61.7 (60.0)	7.8	11.6	hcp a = 2.740, c = 4.392	1.603	Unchanged	
H-220	48.2 (50.0)	51.8 (50.0)	7.5	11.4	hcp a = 2.751, c = 4.417	1.606	Unchanged	
X = Iridium								
H-209	15 (26.1)	85 (73.9)	8.2	19.5	hcp a = 2.745, c = 4.381	1.60	Ordered hcp a' = 5.490, c' = 4.394	1.601
H-216	17.6 (30.0)	82.4 (70.0)	8.1	19.0	hcp a = 2.740, c = 4.392	1.603	Unchanged	
H-217	19.7 (33.0)	80.3 (67.0)	8.0	18.7	hcp a = 2.737, c = 4.404	1.60	Ordered hcp a' = 5.475, c' = 4.404	1.608

a Calculated from X-ray diffraction data.
b 10 hours at 1400 C (2552 F) and 5 x 10^{-5} torr pressure except Heats H-213, H-214, and H-215, which were treated in 1 atmosphere of hydrogen.
c Numbers in parentheses are atomic percent.

Table 1 (Continued)

X-Ray Diffraction Analyses of Several Mo-Pt, Mo-Rh, and Mo-Ir Samples

X = Platinum

Heat No.	Aim Composition (weight percent)		Average Group Number	Density^a (g/cm³)	X-Ray Diffraction Analysis			
					As-Cast		After Heat Treatment^b	
	Mo	X			Phases	c/a	Phases	$\frac{c'}{(a'\sqrt{2})}$
H-204	29 (45.3)	71 (54.6)	8.2		fcc, fct, hcp^d		Not heat treated	
H-208	35 (52.5)	65 (47.5)	7.9		hcp a = 2.786, c = 4.488	1.61	Ordered hcp a' = 5.572, c' = 4.488 Orthorhombic a = 2.75, b = 4.90, c = 4.49	
H-213^b	38 (56.0)	62 (44.0)	7.8		hcp a = 2.748, c = 4.518		Same as H-208	
H-214^b	42 (59.0)	58 (41.0)	7.6	14.9	hcp a = 2.748, c = 4.518	1.64	Ordered hcp a' = 5.590, c' = 4.484	1.604
H-215^b	45 (62.5)	55 (37.5)	7.5	14.3	hcp a = 2.784, c = 4.518		Ordered hcp a' = 5.606, c' = 4.497	1.604
H-221	50 (67.0)	50 (33.0)	7.3		hcp a = 2.748, c = 4.518		Ordered hcp, cubic Mo$_4$Pt	

^{d} fcc = face-centered cubic, fct = face-centered tetragonal, hcp = hexagonal-close packed.

There is one disadvantage to dark field illumination since small pits and inclusions appear as extremely bright spots in photomicrographs. The photomicrographs prepared for this report are subject to an overestimation of the quantity of surface defects as a result of emphasis of a white area on a dark background. Photomicrographs of all the alloys are presented in Figures 4 through 7.

The room-temperature resistivity of the alloys was measured on the heat-treated material using the four-point-probe technique.[14] The instrument was calibrated by comparing the resistivity determined for silver, molybdenum, chromium, Type 304 stainless steel, and bismuth with the published values for each of the metals.[15] The calibration curve is presented in Appendix A. The measured values of resistivity for the experimental alloys, which have been adjusted according to the calibration curve, are presented in Table 2.

Standard techniques were used to determine the room and elevated-temperature hardness values of the alloys.[16,17] A 10-kg load and a diamond indentor were used between room temperature and 871 C (1600 F), and a 5-kg load and a sapphire indentor were used between 1093 and 1649 C (2000 and 3000 F). The data are presented in Table 2 and plotted in Figure 8; the hardness of sapphire[18] between 816 and 1649 C (1500 and 3000 F) is also drawn in Figure 8. All three of the molybdenum-iridium alloys are harder than sapphire between 1093 and 1649 C (2000 and 3000 F). It was noted that the sapphire indentors flattened when the load was applied on Mo-Ir alloys--the alloys themselves showed no hardness impressions.

The oxidation resistance of the alloys was determined by exposing them to air at 538 C (1000 F) and 982 C (1800 F). The corrosion resistance was evaluated at room temperature in aqueous solutions containing 41.6 and 70.9% nitric acid (a 1 to 1 volume ratio of water to nitric acid, and concentrated nitric acid, respectively). A comparable molybdenum sample was included in the tests as a control sample.

The tests for evaluation of oxidation resistance were performed on segments of the hot-hardness sample. Each of the hot-hardness samples was cut into pieces approximately 0.2 by 0.2 by 0.37 inch; the exact dimensions of each sample were determined prior to the test. One small piece of an alloy was placed in a porcelain crucible, 1-1/2 inches in diameter and 1-inch high, and the sample and the sample-plus-crucible were weighed. The combined weight of sample-plus-crucible was used to determine changes in weight during the oxidation test to minimize extraneous losses resulting from handling the samples. The crucibles plus samples, along with a blank crucible, were placed in resistance-heated furnaces at the desired temperatures. Air was circulated through the furnace at the rate of 2.25 liters/min to ensure an adequate supply of oxygen. Changes in weight were measured at regular intervals. These weight changes were converted to weight gains or losses per unit of original area on the un-oxidized sample. Data on the change in weight per unit of original area for the two oxidation temperatures are presented in Table 3 as a gain in weight and in Table 4 as a loss in weight. The data are plotted in Figures 9 and 10. The sample dimension and weights as a function of time at temperature are given in Appendix B.

Table 2

Hardness and Resistivity of Hexagonal-Close Packed Alloys from the Mo-Rh, Mo-Pt, and Mo-Ir Systems

Heat No.	Aim Composition[b] (%)		Electrical Resistivity (μΩ-cm) at 20 C	Hardness (DPH)[a] at Indicated Temperature									
	Mo	X		Room	260 C (500 F)	538 C (1000 F)	704 C (1300 F)	871 C (1600 F)	1093 C (2000 F)	1232 C (2250 F)	1371 C (2500 F)	1510 C (2750 F)	1649 C (3000 F)
X = Rhodium													
H-205	20 (21.1)	80 (79.8)	135	518	498	489	464	379	262	250	160[c]	73.7	38.9
H-218	28.5 (30.0)	71.5 (70.0)	200	457	440[c]	415[c]	405	260	256	172	134	64.8	41.7
H-219	38.3 (40.0)	61.7 (60.0)	135	340	285	283	196	207	220	155	127	73.4	40[c]
H-220	48.2 (50.0)	51.8 (50.0)	91	370	340	300	309	225	175	145	104	61	46
X = Iridium													
H-209	15 (26.1)	85 (73.9)	68	672	630	595	561	546	--	--	--	--	--
H-216	17.6 (30.0)	82.4 (70.0)	74	565	507	449	442	431	>325[d]	>250[d]	>160[d]	>110[d]	>70[d]
H-217	19.7 (33.0)	80.3 (67.0)	120	599	516	516	462	388	--	--	--	--	--
X = Platinum													
H-214	42 (59.0)	58 (41.0)	92	276	246	223	217	202	208	158	124	108	63.2
H-215	45 (62.5)	55 (37.5)	145	365	326	285	254	242	164	142	117	104	63.9

[a] 10-kg load and diamond indentor used from room temperature to 871 C (1600 F), and 5-kg load and sapphire indentor used from 1093 to 1649 C (2000 to 3000 F).
[b] Numbers in parentheses are atomic percent.
[c] Interpolated value.
[d] All Mo-Ir alloys are harder than sapphire between 1093 and 1649 C (2000 and 3000 F); number given is the hardness of sapphire at indicated temperature (after Reference 16).

Table 3

Results of Oxidation Test Performed at 538 C (1000 F)

Heat No.	Aim Composition (%)		Weight Gain[a] (mg/cm^2) for Indicated Total Time at Temperature							Parabolic Rate Constant (mg^4/cm^2hr)
	Mo	X	0.5 hr	1.0 hr	2.0 hr	3.0 hr	5.0 hr	10.0 hr	25.0 hr	
--	100	--	0.238	0.333	0.523	0.760	1.663	4.80	11.6	--[b]
					X = Rhodium					
H-205	20.0	80.0	0.186	0.186	0.279	0.371	0.371	0.604	0.650	0.048
H-219	38.3	61.7	0.482	0.675	0.723	1.01	1.49	2.41	4.14	0.365
H-220	48.2	51.8	0.748	0.748	0.866	1.02	1.10	1.58	2.01	0.690
					X = Iridium					
H-209	15.0	85.0	0.158	0.197	0.276	0.354	0.394	0.473	0.788	0.048
H-216	17.6	82.4	0.512	0.085	0.342	0.427	0.427	0.726	1.07	0.063
H-217	19.7	80.3	0.241	0.241	0.337	0.482	0.578	0.771	1.40	0.084
					X = Platinum					
H-214	42	58.0	0.787	1.33	1.77	2.16	2.56	3.15	4.87	1.56
H-215	45	55.0	1.87	2.17	2.83	3.08	3.43	4.44	5.75	4.80

a Calculated using the original area of the unoxidized sample.
b Molybdenum does not follow a parabolic oxidation rate law.

Table 4

Results of Oxidation Test Performed at 982 C (1800 F)

Heat	Aim Composition (%)		Accumulated Weight Loss[a] (g/cm^2) for Indicated Total Time at Temperature							
	Mo	X	0.25 hr	0.5 hr	0.75 hr	1.0 hr	1.25 hr	1.5 hr	5.0 hr	10.0 hr
--	100	--	0.211	0.408	0.579	0.729	0.819	0.850	sample consumed	
X = Rhodium										
H-205	20.0	80.0	0.006	0.007	0.008	0.009	0.009	0.010	0.022	0.051
H-219	38.3	61.7	0.169	0.188	--b	--	--	--	--	--
H-220	48.2	51.8	0.195	0.344	--b	--	--	--	--	--
X = Iridium										
H-209	15.0	85.0	0.024	0.028	0.032	0.034	0.034	0.034	--b	--
H-216	17.6	82.4	0.053	0.061	0.072	0.078	--b	--	--	--
H-217	19.7	80.3	0.065	0.095	0.121	0.138	0.148	0.154	--b	--
X = Platinum										
H-214	42.0	58.0	0.243	0.389	--b	--	--	--	--	--
H-215	45.0	55.0	0.257	0.424	--b	--	--	--	--	--

[a] Calculated using original area of unoxidized sample.
[b] Test discontinued, see text for explanation.

All the 538 C (1000 F) oxidation samples were examined metallographically on completion of the exposure tests. Only the samples having relatively high molybdenum contents developed significant thickness of oxide layer. Photo-micrographs of the few visible oxide layers are presented in Figures 11 through 14.

The curve of weight gain per unit of original surface area for Heat H-219 (Figure 9) has an anomolous discontinuity. Examination of the sample after 5 hours of exposure revealed that a number of cracks had formed along the edges of the specimen. The net result was that the actual surface area being oxidized was greatly increased over the original area.

The change of weight per unit area data of Table 3 were plotted versus time on a log-log scale. The slope of the lines drawn through the data indicated that the oxidation of all the experimental alloys is closely approximated by a parabolic rate law. The parabolic rate constant for each alloy is given in Table 3. The rate constant for Heat H-219, given in Table 3, is based on data for the initial 2 hours of the test, since the crack formation noted above made it impossible to accurately calculate the surface area being oxidized.

The oxidation testing of several samples at 982 C (1800 F) was terminated after a short time of exposure. There were two reasons for discontinuing the test. For example, it was felt that Heats H-219, H-220, H-214, and H-215 were were oxidizing as rapidly as molybdenum. It was decided to save the remainder of the unoxidized sample for other testing programs. On the other hand, the oxidation of Heats H-209, H-216, and H-217 were discontinued because of the concurrent formation of a nonadherent, powdery black oxide, and volatilization of MoO_3; the former increased the weight of the sample, the latter decreased its weight. The net result of this competition was that the oxidation rate could not be measured.

The evaluation of the corrosion resistance of the hcp compound in two concentrations of nitric acid was also performed on small pieces cut from the hot-hardness specimen. Each small sample was measured and weighed prior to being immersed in 100 ml of either 41.6% nitric acid (a 1 to 1 volume ratio) or 70.9% nitric acid (concentrated). A small piece of molybdenum was included in each test as a control sample. Each sample was washed with water, rinsed with methanol, and air-dried after each time period in the acid solution. The results of the tests, including original sample dimensions, are presented in Tables 5 and 6. The results of exposure to the 41.6% nitric acid solution, which proved to be more severe than exposure to concentrated acid, are also plotted in Figure 15 as weight loss per unit of area on the original unexposed sample. The data for exposure to the concentrated nitric acid are inconclusive. No trend or pattern in weight change developed in the 255 hours of the test, except for Alloy H-215 (55% Pt and 45% Mo), which showed a slight decrease in weight.

Table 5

Results of Corrosion Testing of Experimental Alloys in an Aqueous
Solution Containing 41.6% Nitric Acid

Heat No.	Aim Composition (%)		Surface Area (cm^2)	Starting Weight (g)	Accumulated Weight Change per Unit Area (mg/cm^2) for Indicated Total Time		
	Mo	X			25 hr	98 hr	268 hr
--	100	--	1.895	1.8987	Sample completely dissolved in less than 2 hr		
X = Rhodium							
H-205	20	80	2.092	2.1330	nil	nil	nil
H-219	38.3	61.7	2.256	2.2436	nil	nil	nil
H-220	48.2	51.8	2.380	2.5155	nil	nil	nil
X = Iridium							
H-209	15	85	2.542	4.3498	nil	nil	nil
H-216	17.6	82.4	2.328	3.9860	-0.472 (-1.1)[a]	-0.601 (-1.4)	-0.601 (-1.4)
X = Platinum							
H-214	42	58	1.995	2.3868	-0.652 (-1.3)	-1.40 (-2.8)	-2.41 (-4.8)
H-215	45	55	1.992	2.2531	-1.76 (-3.5)	-4.32 (-8.6)	-7.38 (-14.7)

[a] Numbers in parentheses give accumulated weight change (mg) for indicated total time.

Table 6

Results of Corrosion Testing of Experimental Alloys
in Concentrated (70.9%) Nitric Acid

Heat No.	Aim Composition (%)		Surface Area (cm^2)	Starting Weight (g)	Accumulated Weight Change per Unit Area (mg/cm^2) for Indicated Total Time				
	Mo	X			2.0 hr	6.5 hr	23 hr	96 hr	255 hr
--	100	--	2.084	1.8847	-0.575 (-1.2)[a]	1.73 (-3.4)	-5.18 (-7.2)	-99.50 (-197)	
X = Rhodium									
H-205	20	80	2.092	2.1330	nil	nil	nil	nil	nil
H-219	38.3	61.7	2.256	2.2436	nil	nil	nil	nil	nil
H-220	48.2	51.8	2.380	2.5150	nil	nil	nil	nil	nil
X = Iridium									
H-209	15	85	2.542	4.3497	nil	nil	nil	nil	nil
H-216	17.6	82.4	2.329	3.9846	nil	nil	nil	nil	nil
X = Platinum									
H-214	42	58	1.995	2.3818	nil	nil	nil	nil	nil
H-215	45	55	1.992	2.2381	nil	nil	nil	nil	1.6 (-3.0)

[a] Numbers in parentheses give accumulated weight change (mg) for indicated total time.

DISCUSSION

One major problem encountered in this investigation would tend to limit the applicability of the molybdenum-platinum group metal hcp compounds--they are extremely brittle. The brittleness of the compound may be considered in two ways:

1. the grains of the alloy exhibited some fracture around hardness impressions, particularly at low temperatures [below 675 C (1250 F)], and high rhodium or iridium contents (greater than 70% rhodium or 80% iridium--the lowest iridium content in buttons cast for this study). The molybdenum-platinum hcp phase and the rhodium alloys containing 50 and 40% molybdenum appeared to be more ductile, as indicated by the deformation around the hardness impressions.

2. The grain boundaries of all the samples lack cohesion and can be considered to be the dominant factor in determining the apparent ductility of the alloys. Here again, the alloys with the higher iridium and rhodium contents are more susceptible to loss of grain boundary cohesion than the alloys with the higher molybdenum contents. One alloy, the 71% Rh - 29% Mo alloy, was so brittle that it could not be properly cut or ground to produce the hardness specimen because of grain boundary fracture.

Fractography provided a means for examining the grain boundaries of some of the compositions. Two typical fractographs are presented in Figure 16; one was taken on Heat H-205 (80% Rh and 20% Mo) the other was taken on Heat H-209 (85% Ir and 15% Mo). Both grain boundaries are densely populated with what appear to be oxide inclusions. Efforts to reduce the quantity of inclusions by recasting the alloy with a small carbon addition failed. It was found, however, that rapid cooling through the liquidus region of the phase diagram improved the apparent cohesion of the grain boundaries. It is reasoned that quenching the liquid alloy decreased the grain size of the solidified material, and increased the grain-boundary area over which the inclusion could be distributed. Samples prepared in this manner were easier to cut and polish than samples cooled slowly from above the liquidus temperature. A more thorough study of means for reducing grain boundary impurities is recommended.

The rapid cooling from above the liquidus temperature did introduce the question of coring during solidification. It had been anticipated that the 1400 C (2552 F) heat treatment would alleviate some of the effects of coring. However, remnants of the dendritic structure are observed in Heat H-205, 80% Rh and 20% Mo [Figure 4(a)], Heat H-216, 82.4% Ir and 17.6% Mo [Figure 5(b)], and Heat H-215, 55% Pt and 45% Mo (Figure 7). Semiquantitative analysis on the electron microprobe analyzer indicated that only the molybdenum-platinum alloy had a detectable difference in composition between the matrix and the boundary region. The grain boundary area is slightly richer in platinum than the matrix. Two areas, approximately 0.5 micron in diameter (one in the

matrix and one in the etched region, marked on Figure 7(a), were examined more precisely. It was found that there is a 3 atomic percent difference, equivalent to approximately one weight percent, in the platinum content between the two areas, grain boundary region 2 is higher in platinum. Cursory examination of other points confirmed that this difference existed at the same level throughout the sample.

The segregation or coring in Heat H-215 resulted in a small amount of second phase that appears as a small black phase in the etched sample [Figure 7(a)]; the amount of second phase present is less than about 5 volume percent, and was not detected in the X-ray diffraction pattern. The one photomicrograph of Heat H-215 [Figure 7(a)] showing the second phase was taken on a sample from the edge of the arc-cast button where segregation is highest. The second photomicrograph of Heat H-215 [Figure 7(b)], which shows no second phase, was taken from a more central location in the arc-cast button. Even though the quantity of second phase in Heat H-215 is small and the difference in composition between the two areas in the heat is also small, these two factors may have had an influence on the hardness of the sample, as will be discussed later in this report.

Although the heat treatment at 1400 C (2552 F) for 10 hours failed to homogenize several of the experimental alloys completely, the annealing treatment did order the two molybdenum-platinum alloys and two of the three molybdenum-iridium alloys. The ordered structure for MoIr$_3$ has been identified as MgCd$_3$ type, space group D^4-P6$_3$/mmc with 8 atoms per unit cell.[13] The X-ray diffraction pattern for the order hexagonal Mo$_3$Pt$_2$ (or Mo$_5$Pt$_3$) has been published,[10] but no further definition of space group is known.

It is interesting to note that the crystal structure of as-cast Mo-Pt alloys (Table 1), over a range of 35 to 50% molybdenum, was disordered hcp, the so-called epsilon structure. The crystal structures of the annealed alloys differ markedly from the as-cast structures, except for the two compositions at 42 and 45% molybdenum (Heats H-214 and H-215). It may be surmised that the investigators reporting a wide range of homogeneity for the disorderd epsilon phase in the Mo-Pt system did not anneal their sample properly. In other words, the results of this study of arc-cast and annealed materials confirm the phase relations suggested by Rooksby and Lewis,[10] and Selman.[11] The data of this investigation indicate that the range of solid solubility of molybdenum and platinum in Mo$_3$Pt$_2$ is smaller than estimated by Ocken and Van Vucht[12] (Figure 3).

The trends in lattice spacing, as influenced by the average group numbers of the alloys studied, are presented in Figure 17. The lattice constant, $\underline{a}$, of the normal hexagonal structure is plotted, not the a' of the ordered structure. Since five of the nine compositions prepared have the normal, disordered structure, it was decided to use $\frac{1}{2}$a' for the plot. The data are taken from Table 1. The interesting aspect of Figure 17 is that lattice spacing of the three systems studied are affected differently by changes in composition of the hexagonal phase, i.e., by changes in the average group number. Both the $\underline{a}$ and $\underline{c}$ lattice constants for the Mo-Pt alloy increase as the platinum content increases; the ratio of c/a, however, remains constant. The lattice constants for the Mo-Rh alloys decrease as the rhodium content increases, but at different rates. Thus, the ratio c/a goes through a minimum at some average-group number

between 8.0 and 8.25. These trends for the Mo-Rh hcp phase are in agreement with the trends noted by Anderson and Hume-Rothery,[6] although the actual constants determined in this study are from 0.1 to 0.5% larger than those reported by Anderson. In contrast to both the Mo-Pt and Mo-Rh alloys, the a for the Mo-Ir alloys increases with increasing iridium content, but the c decreases. The c/a ratio for the MoIr₃ composition range decreases fairly sharply with increasing iridium content. In summary, it is concluded that the average group number of the alloys has little or no effect on lattice constants, and that some other physical factor, such as differences in atomic radii, must determine lattice constants.

It may also be noted that the hardness of the molybdenum-platinum group alloys between room temperature and 1093 C (2000 F), as well as their resistance to oxidation at 538 C (1000 F) and corrosion in nitric acid, varies more as a function of atomic percent molybdenum in the alloy than any other factor. The hardness of the alloys at all test temperatures with few exceptions decreases as the molybdenum content increases from 26 atomic percent in Heat H-209 to 60 atomic percent in Heat H-214. Similarly, the parabolic rate constant for oxidation at 538 C (1000 F) decreases markedly as the molybdenum content of the alloys decreases over the same range of atomic percents. The shaded band (Figure 18) is drawn to show the general trend of the parabolic rate constant as a function of molybdenum content. The weight loss per unit area of the alloys during exposure to dilute nitric acid (Figure 15) also decreases markedly.

There are some exceptions to the trend noted for hardness of the hcp alloys with respect to molybdenum content. First, Heat H-205 (21.1 atomic percent Mo and 79.8 atomic percent Rh) has less molybdenum than Heat H-209 (26.1 atomic percent Mo and 73.9 atomic percent Ir) and yet is softer than Heat H-209. Part of this behavior may be attributed to the ordering of the Mo-Ir alloy and to differences in atomic radii. The other exceptions occur at higher molybdenum contents. Specifically, Heat H-215 has a higher atomic percent molybdenum than Heat H-214 for the Mo-Pt alloys (62.5 atomic percent molybdenum in Heat H-215 versus 59 atomic percent molybdenum in Heat H-214), but is harder than Heat H-214. Similarly, Heat H-220 has a higher atomic percent molybdenum than Heat H-219 for the Mo-Rh alloys (50 atomic percent molybdenum in Heat H-220 versus 40 atomic percent molybdenum in Heat H-219), but is harder than Heat H-219. These two exceptions to the general trend of hardness as a function of molybdenum content may be a result of the coring and minute amount of second phase; the latter being observed only in Heat H-215.

It is interesting to compare the hardness of the molybdenum-platinum group metal hcp phase with the hardness values of other refractory metal systems. The hardness of TZC, a molybdenum-base alloy,[19] is plotted in Figure 8, and may be seen to fall below all the alloys prepared for this study. Hardness data for other refractory metal systems are not as plentiful as would be desired for this comparison; however, Semchyshen and Barr[20] have reported room-temperature hardness values of 340 to 400 DPH for a wide range of arc-cast tungsten-base alloys. The hardness of these alloys decreases to approximately 80 DPH at 1540 C (2800 F) as shown by the hardness band in Figure 8. Field et al.[21] have reported room-temperature hardness values of 100 to 400 DPH for tantalum-base alloys, with one or two experimental alloys reaching 500 DPH. Therefore, if hardness

can be used as an indicator of other mechanical properties, it might be expected that the molybdenum platinum-group metal hcp compounds may be able to surpass the strength properties of some of these refractory metal alloys.

SUMMARY AND CONCLUSIONS

An investigation has been conducted on the hcp intermetallic compound found in the binary systems that include molybdenum and one metal from the group rhodium, iridium, and platinum. The compound can be broadly characterized as having a hcp crystal structure and melting at temperatures in excess of 1900 C (3452 F). The compositions that are characterized ranged from 50 to 80% rhodium, a disordered hcp phase; from 80 to 85% iridium, centered around $MoIr_3$, an ordered hcp compound; and 55 to 58% platinum, the limits of the ordered hcp compound Mo_3Pt_2. Until this investigation, little else was known about the compound. This investigation has characterized small arc-cast samples of several compositions within limits of the hcp compound by determining the room and elevated-temperature hardness, the resistivity, the oxidation resistance when exposed to air at 538 C (1000 F) and 982 C (1800 F), and corrosion resistance to aqueous solutions containing 41.6 and 70.9% nitric acid.

All the alloys were arc-cast and then annealed at 1400 C (2552 F) for 10 hours. The annealing treatment developed the equilibrium crystal structure without greatly increasing the grain size of the samples. Most of the samples were homogeneous and single phase, as determined by metallographic and X-ray diffraction analyses. One sample, containing 45% Mo and 55% Pt, did have a small amount of unidentified second phase in the grain boundaries.

The molybdenum-platinum group metal hcp compound is embrittled by oxides that are dispersed along the grain boundaries during solidification. The alloys having the higher molybdenum contents are less prone to the effect of grain boundary impurities.

The hcp compound has a room-temperature hardness between 300 and 675 DPH. The hardness of the compound decreases as the atomic percent molybdenum increases. The hardness of the molybdenum-rhodium and molybdenum-platinum hcp compound decreases to about 50 DPH at 1649 C (3000 F). The molybdenum-iridium alloys studied are harder than sapphire (the material used as the indentor) between 1093 and 1649 C (2000 and 3000 F). On the basis of hardness data, it is concluded that iridium is most effective in hardening the hcp phase for a fixed molybdenum content, followed in decreasing order by rhodium and platinum.

All the alloys gain weight during oxidation at 538 C (1000 F) following a parabolic rate law. The parabolic rate constant increases as the atomic percent molybdenum in the alloy increases. All the alloys except one lose weight at 982 C (1800 F) at approximately the same rate as pure molybdenum. Only the 80% Rh - 20% Mo alloy exhibited oxidation resistance at 982 C (1800 F) for periods up to 10 hours.

The alloys investigated are all resistant to corrosion by dilute and concentrated nitric acid. The molybdenum-platinum alloys do lose a small amount of weight in 250 hours of exposure.

The hcp compound has resistivity in the range of 100 μohm-cm at 20 C.

-16-

RECOMMENDATIONS

The research program has provided a limited characterization of a hcp intermetallic compound containing molybdenum and a metal from the group rhodium, iridium, and platinum. It is noted that the room and elevated-temperature hardness of the compound is generally greater than molybdenum, tungsten, or tantalum base alloys. It is believed that the hcp alloys are worthy of further investigation. In particular, a better characterization of mechanical properties is required to evaluate the usefulness of the alloys. In addition, it is necessary to establish a procedure for increasing the purity of the alloys by decreasing the grain boundary impurities. No fair evaluation of properties is possible with the grain boundaries as brittle as they were in the samples prepared for this investigation. Finally, it is recommended that consideration be given to finding a ternary alloying element that could be substituted for the expensive platinum-metal elements used for this study.

Data presented in this report may be found in Laboratory Records Book 780, pp. 9-32.

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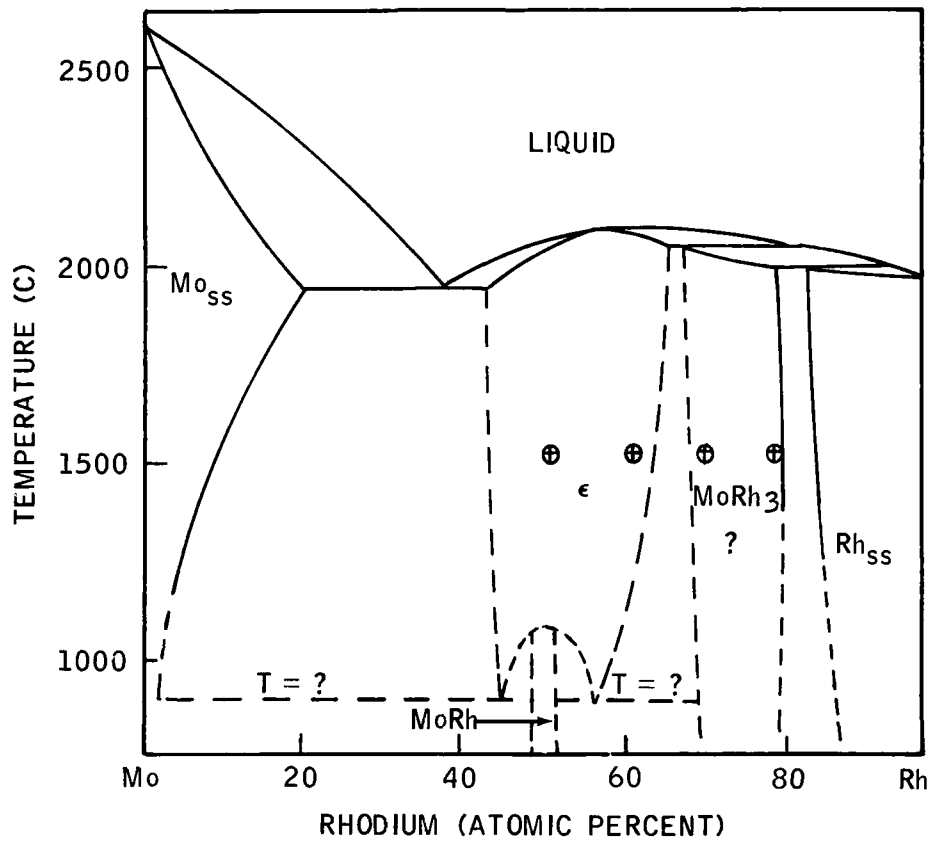
Figure 1   Phase Diagram for the Mo-Rh System

after   E. Anderson and W. Hume-Rothery, J. Less Common Metals 2 (1960),
19-28; modified with data from B. C. Giessen, U. Jaehmigen, and
N. J. Grant, J. Less Common Metals 10 (1965), 147-150.

⊕ Represents the compositions of molybdenum-rhodium alloys arc-cast
for this investigation.

-21-
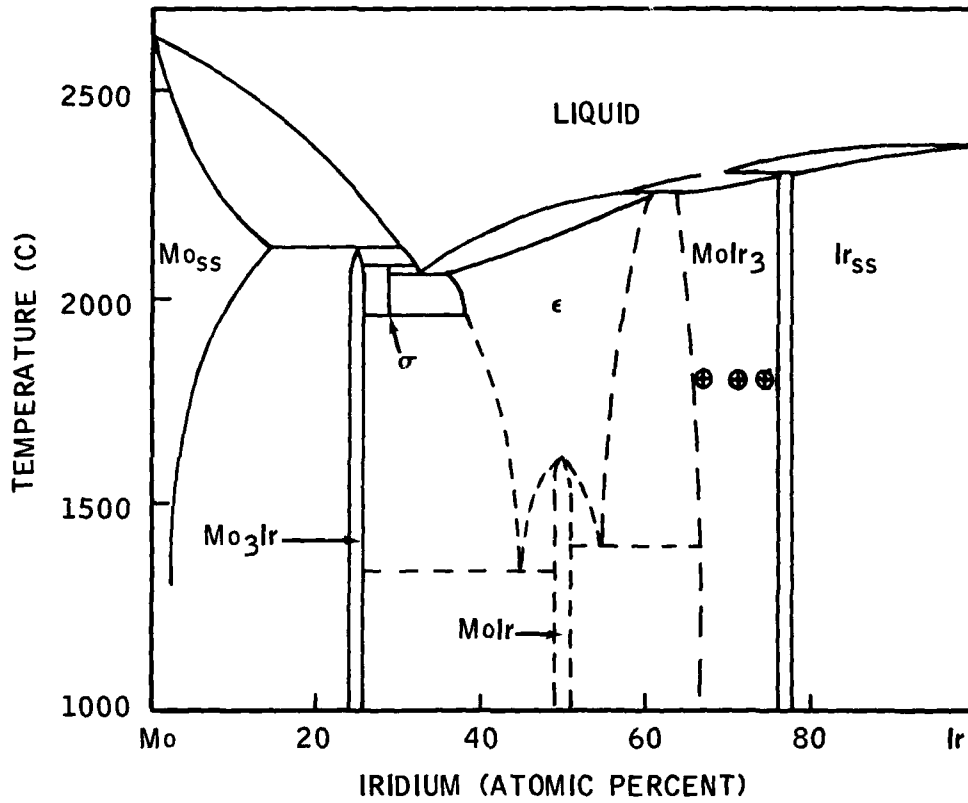
Figure 2 Phase Diagram for the Mo-Ir System

after B. C. Giessen, U. Jaehnigen, and N. J. Grant, J. Less Common Metals 10
(1965), 147-150.

⊕ Represents the compositions of the molybdenum-iridium alloys arc-cast
for this study.
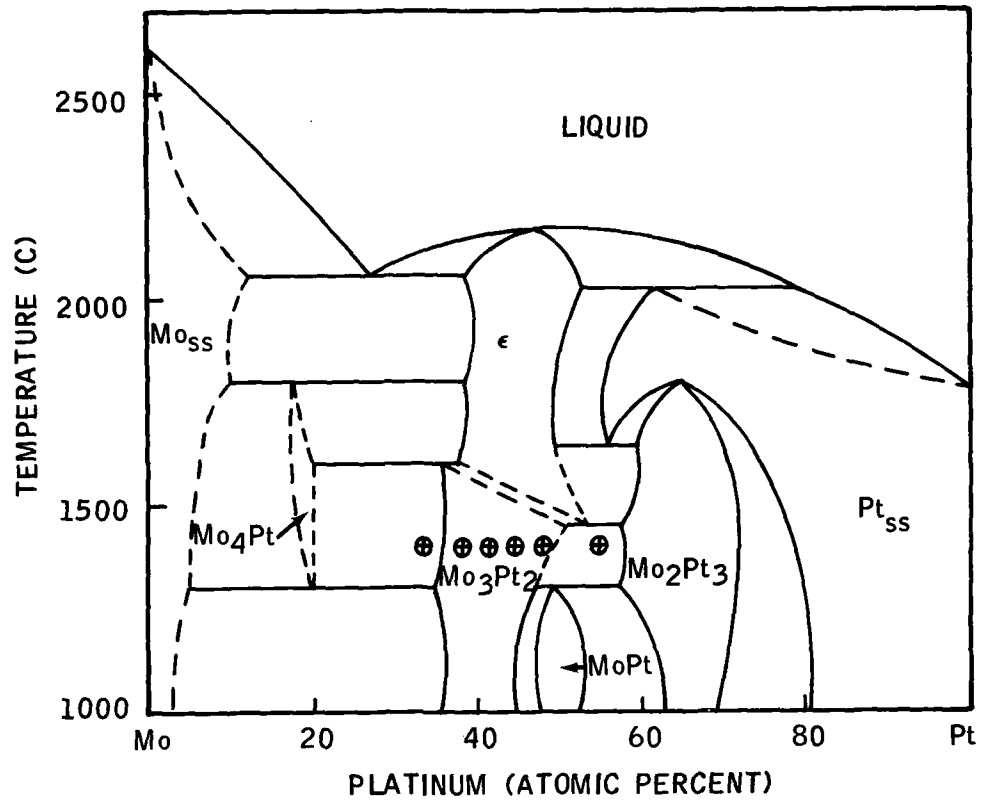
Figure 3 Phase Diagram for the Mo-Pt System

after H. Ocken and J. H. N. Van Vucht, J. Less Common Metals 15
(1968), 193-199.

⊕ Represents the composition of the molybdenum-platinum alloys arc-cast
for this study.

-23-

Contract No. N00014-66-C0338
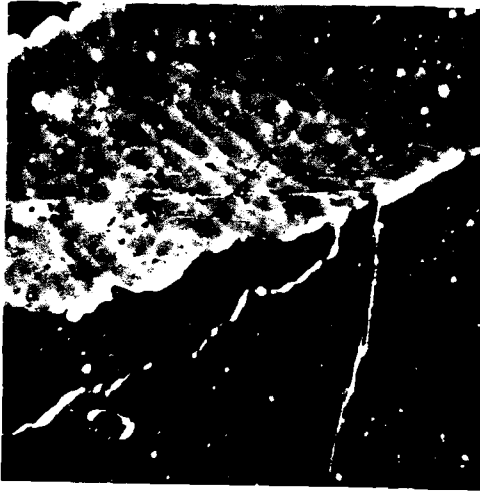
N6593 Dark Field Illumination X250
(a) Heat H-205 (80% Rh and 20% Mo)

N6590 Dark Field Illumination X250
(a) Heat H-218 (71.5% Rh and 28.5% Mo)
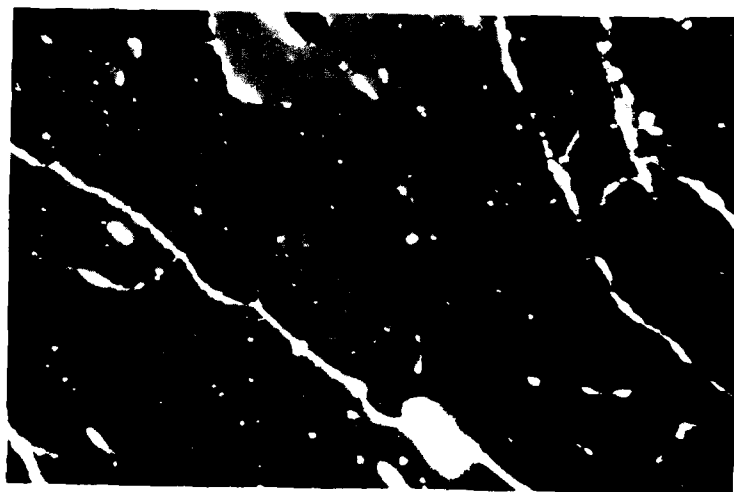
N6591 Dark Field Illumination X250
(c) Heat H-219 (61.7% Rh and 38.3% Mo)
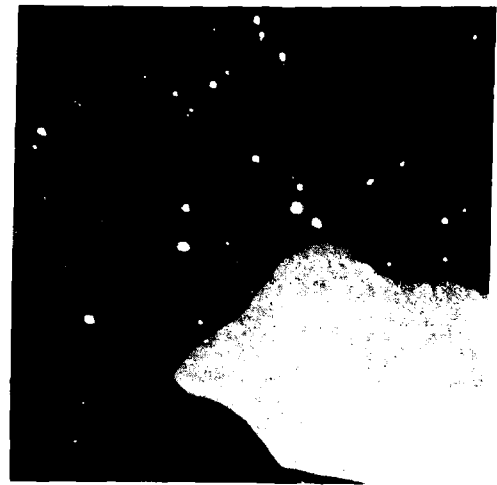
N6592 Dark Field Illumination X250
(d) Heat H-220 (51.8% Rh and 48.2% Mo)

Figure 4 Photomicrographs of the Molybdenum-Rhodium hcp Alloys Heat-Treated
at 1400 C (2552 F) for 10 Hours
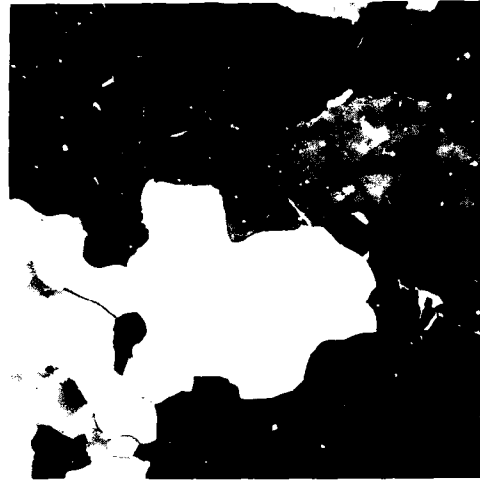
N6597 Dark Field Illumination X250

(a) Heat H-209 (85% Ir and 15% Mo)

N6588 Dark Field Illumination X250

(b) Heat H-216 (82.4% Ir and 17.6% Mo)
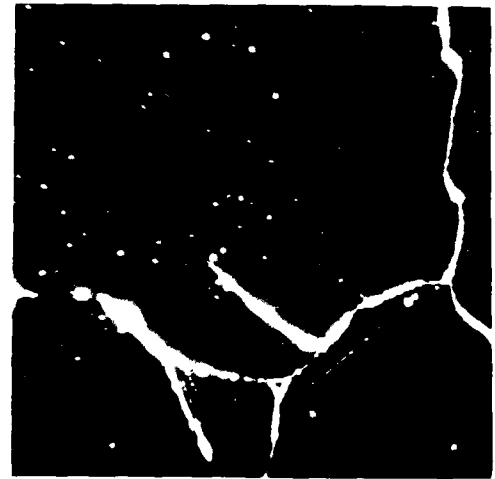
N6594 Dark Field Illumination X250

(c) Heat H-217 (80.3% Ir and 19.7% Mo)

Figure 5   Photomicrographs of the Molybdenum-Iridium hcp Alloys Heat-Treated
at 1400 C (2552 F) for 10 Hours

-27-
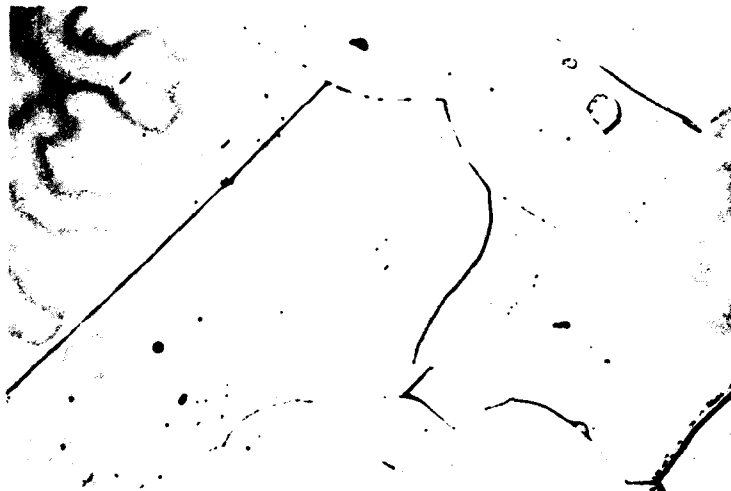
N6601   NaCl Saturated Solution of 20% HCl   X500

(a) Etched Condition

N6586   Mechanical Polish   X250

(b) Dark Field Illumination

Figure 6   Photomicrograph of Heat H-214 (58% Pt and 42% Mo) Heat-Treated
at 1400 C (2552 F) for 10 Hours
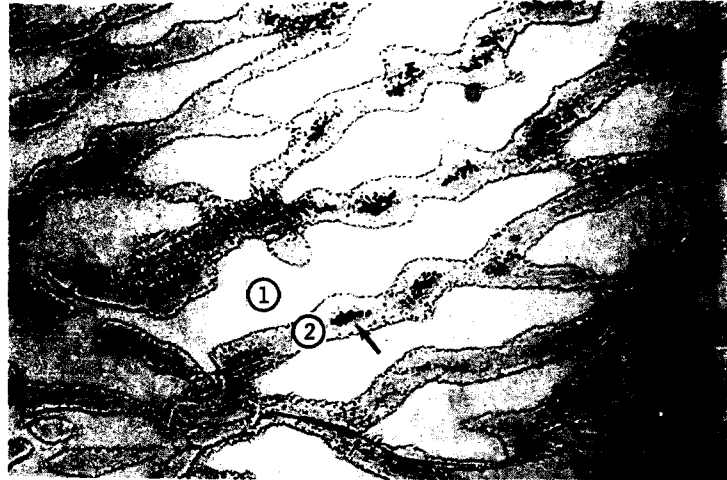
N6602   NaCl Saturated Solution of 20% HCl   X500

(a) Etched Condition

Microprobe analyses were made at Locations 1 and 2. A
second phase is present in the microstructure and indicated
by an arrow.
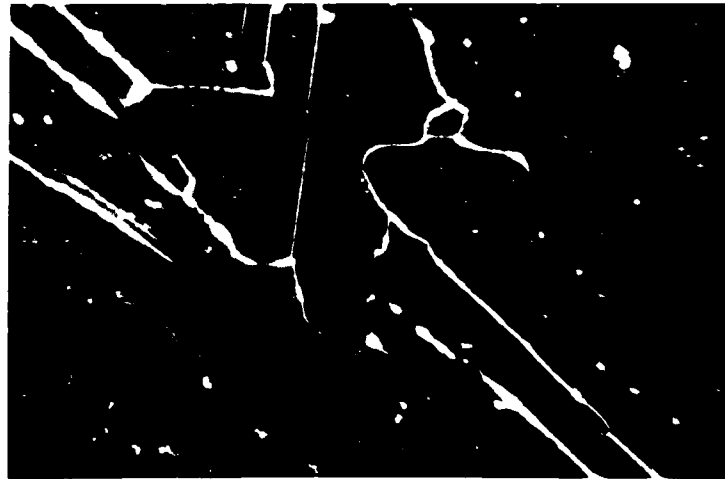
N6600   Mechanical Polish   X250

(b) Dark Field Illumination

Figure 7   Photomicrograph of Heat H-215 (55% Pt and 45% Mo) Heat-Treated
at 1400 C (2552 F) for 10 Hours
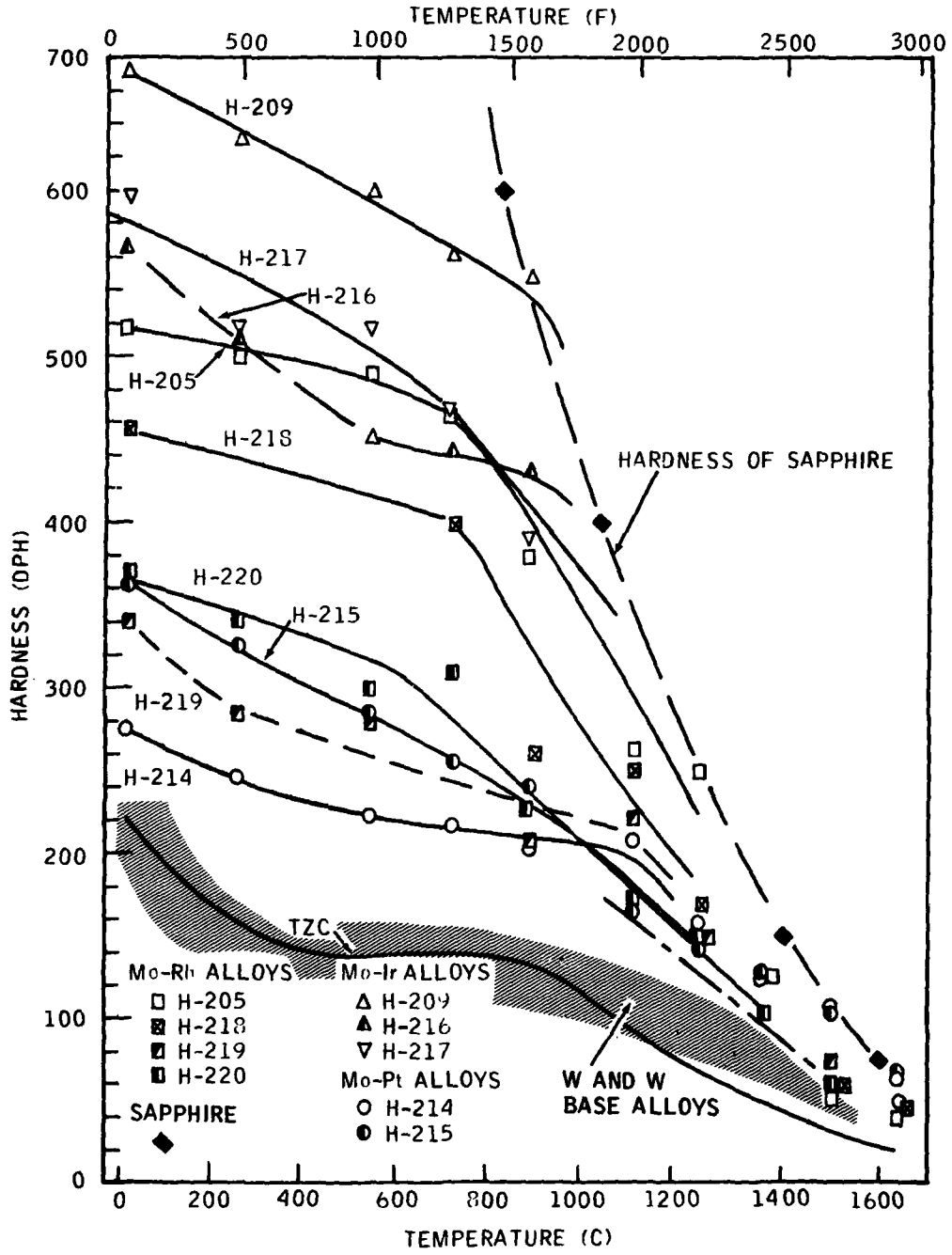
Figure 8   Hardness of Experimental Alloys As a Function of Test Temperature
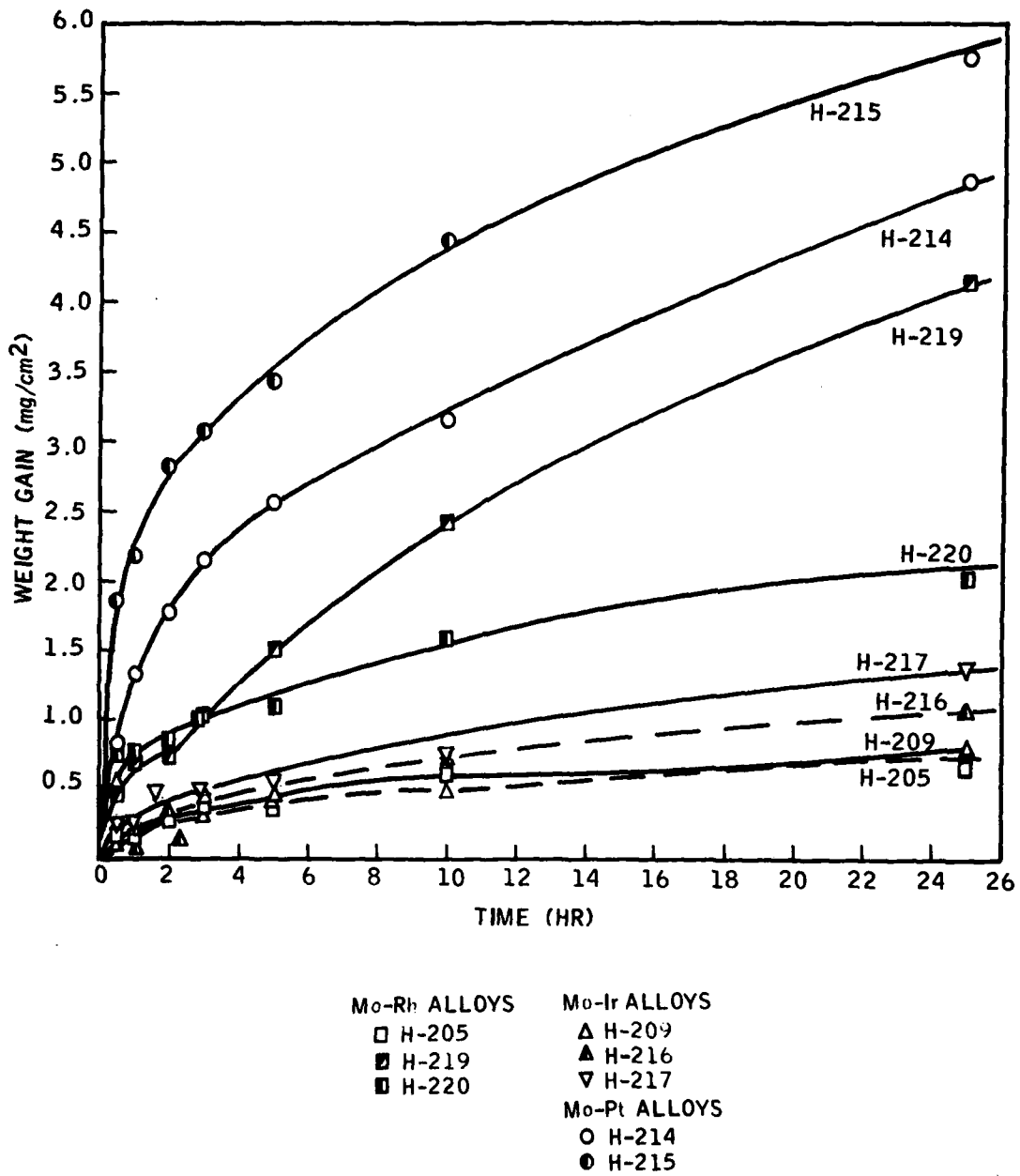
Figure 9 Accumulated Weight Gain of the Molybdenum–Platinum Group Metal hcp
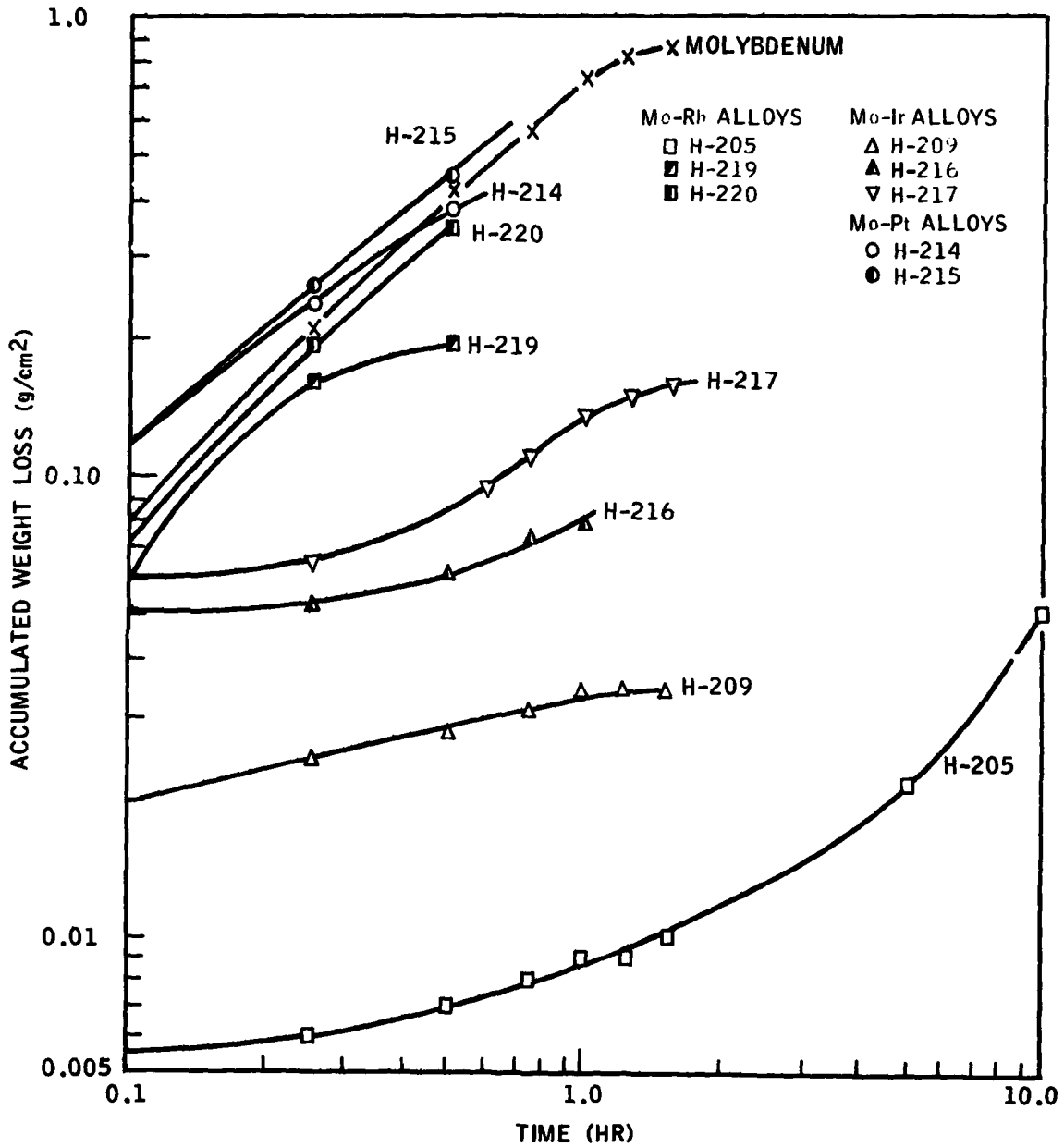Compounds on Exposure to Air at 538 C (1000 F)

Figure 10   Accumulated Weight Loss of the Molybdenum-Platinum Group Metal hcp
Compounds on Exposure to Air at 982 C (1800 F)

Contract No. N00014-66-C0338
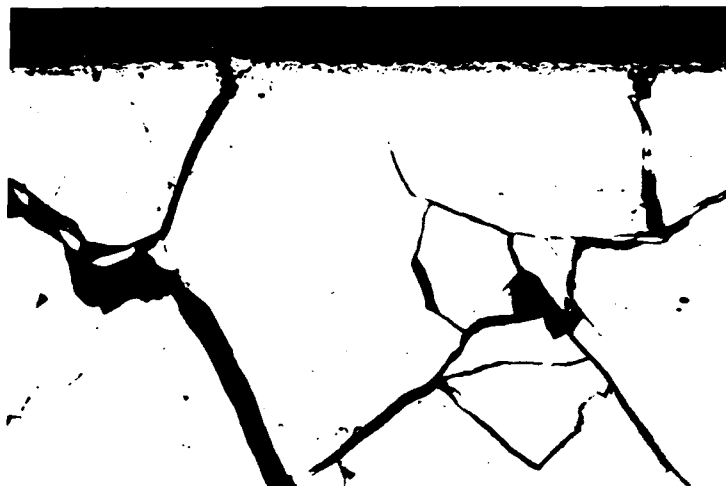

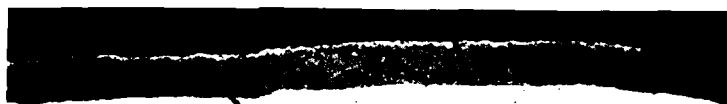N6577   Mechanical Polish   X500

Figure 11 Photomicrograph of the Oxide Layer on Molybdenum Exposed to
to Air at 538 C (1000 F) for 25 Hours

N6580   Mechanical Polish   X500

(a) Heat H-219 (61.7% Rh and 38.3% Mo)

N6581   Mechanical Polish   X500

(b) Heat H-220 (51.8% Rh and 48.2% Mo)

Figure 12   Photomicrograph of the Oxide Layer Developed on Two Molybdenum-
Rhodium Alloys after Exposure to Air at 538 C
(1000 F) for 25 Hours

Contract No. N00014-66-C0338

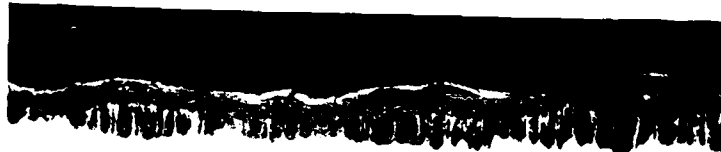N6579   Mechanical Polish   X500

Figure 13   Photomicrograph of Oxide Layer on Heat H-217 (80.3% Ir and 19.7% Mo)
            after Exposure to Air at 538 C (1000 F) for 25 Hours

Contract No. N00014-66-C0338

N6584   Mechanical Polish   X500

(a) Heat H-214 (58% Pt and 42% Mo)

N6585   Mechanical Polish   X500

(b) Heat H-215 (55% Pt and 45% Mo)

Figure 14   Photomicrograph of the Oxide Layer Developed on Two Molybdenum-Platinum Alloys after Exposure to Air at 538 C (1000 F) for 25 Hours
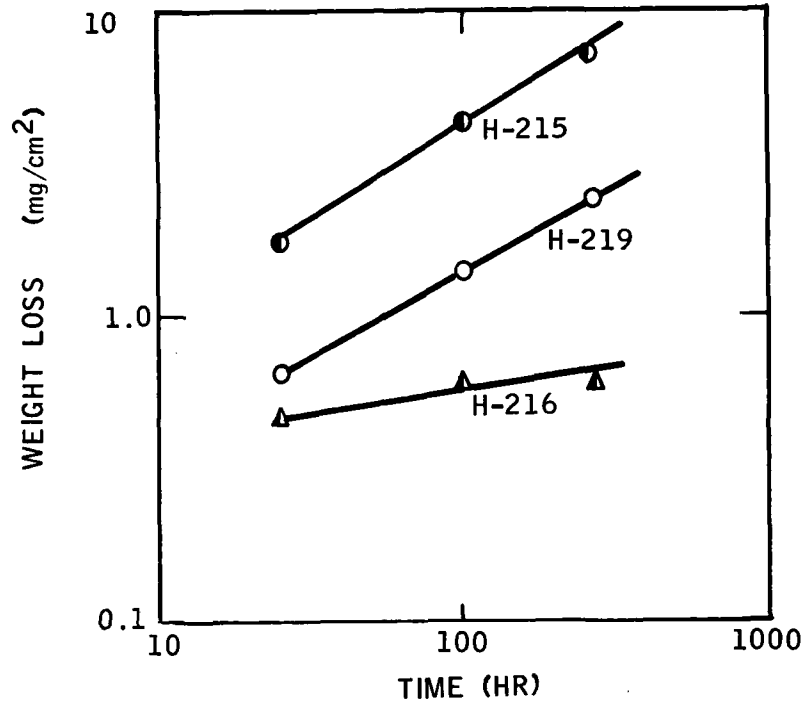
Figure 15   Weight Loss per Unit Area for the Molybdenum-Platinum Group Metal
hcp Compounds on Exposure to an Aqueous Solution
of 41.6% Nitric Acid

Contract No. N00014-66-C0338

N5935   Fractograph   X2000

(a) Heat H-205 (80% Rh and 20% Mo)

N5943   Fractograph   X2000

(b) Heat H-209 (85% Ir and 15% Mo)

Figure 16   Fractographs of a Rh-Mo and an Ir-Mo Alloy Showing
Grain Boundary Oxides
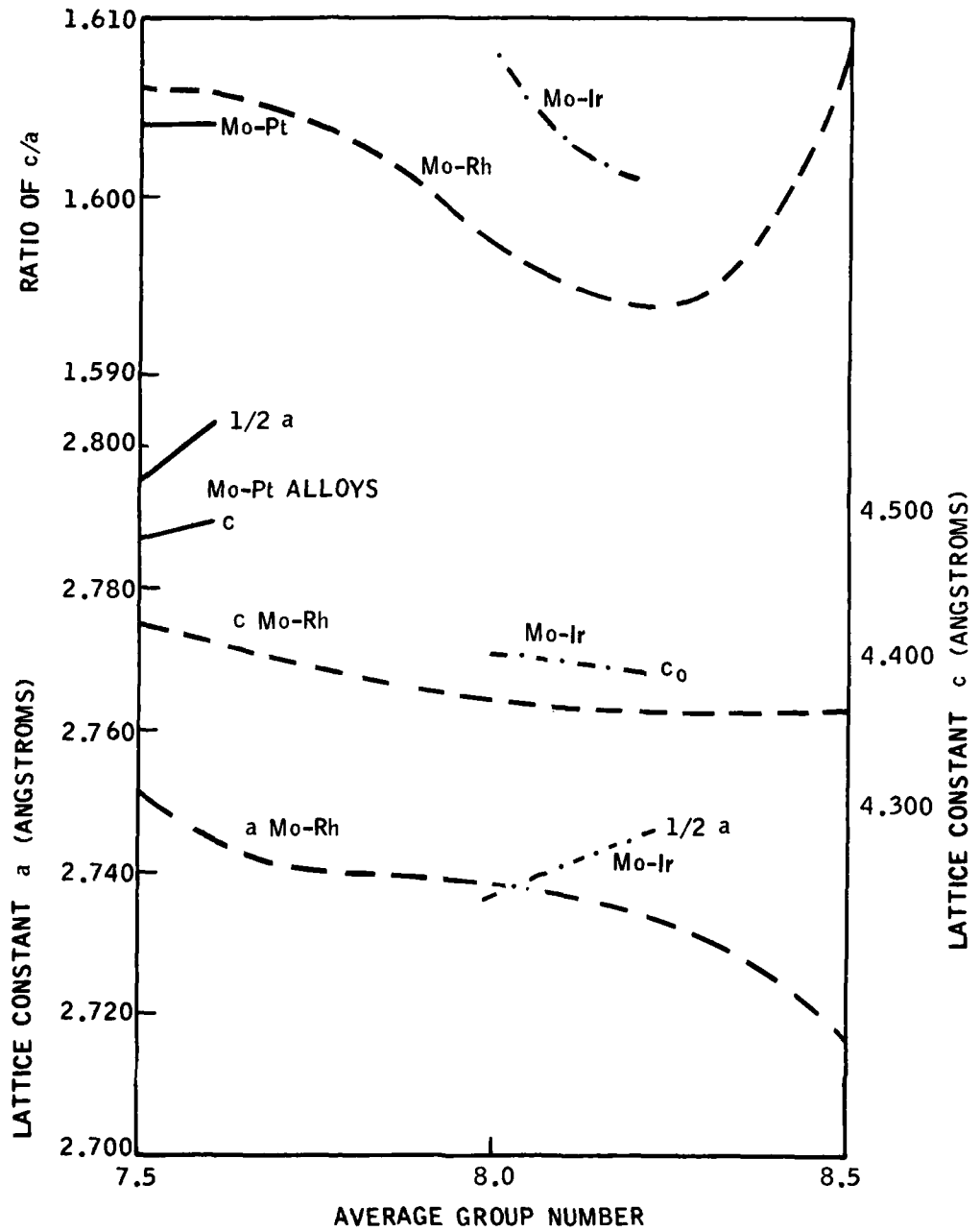
Figure 17   Lattice Spacings As a Function of Average Group Number for the
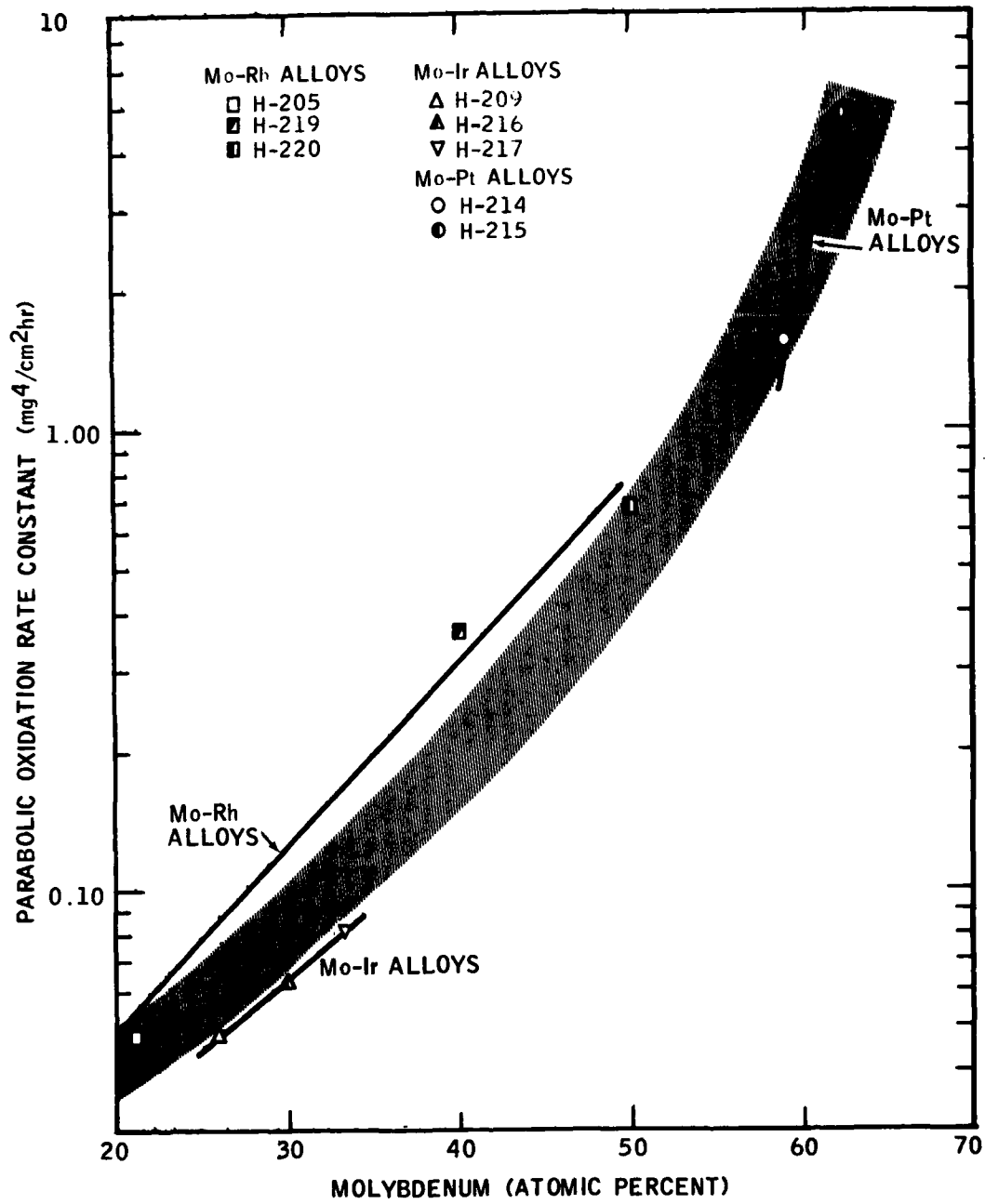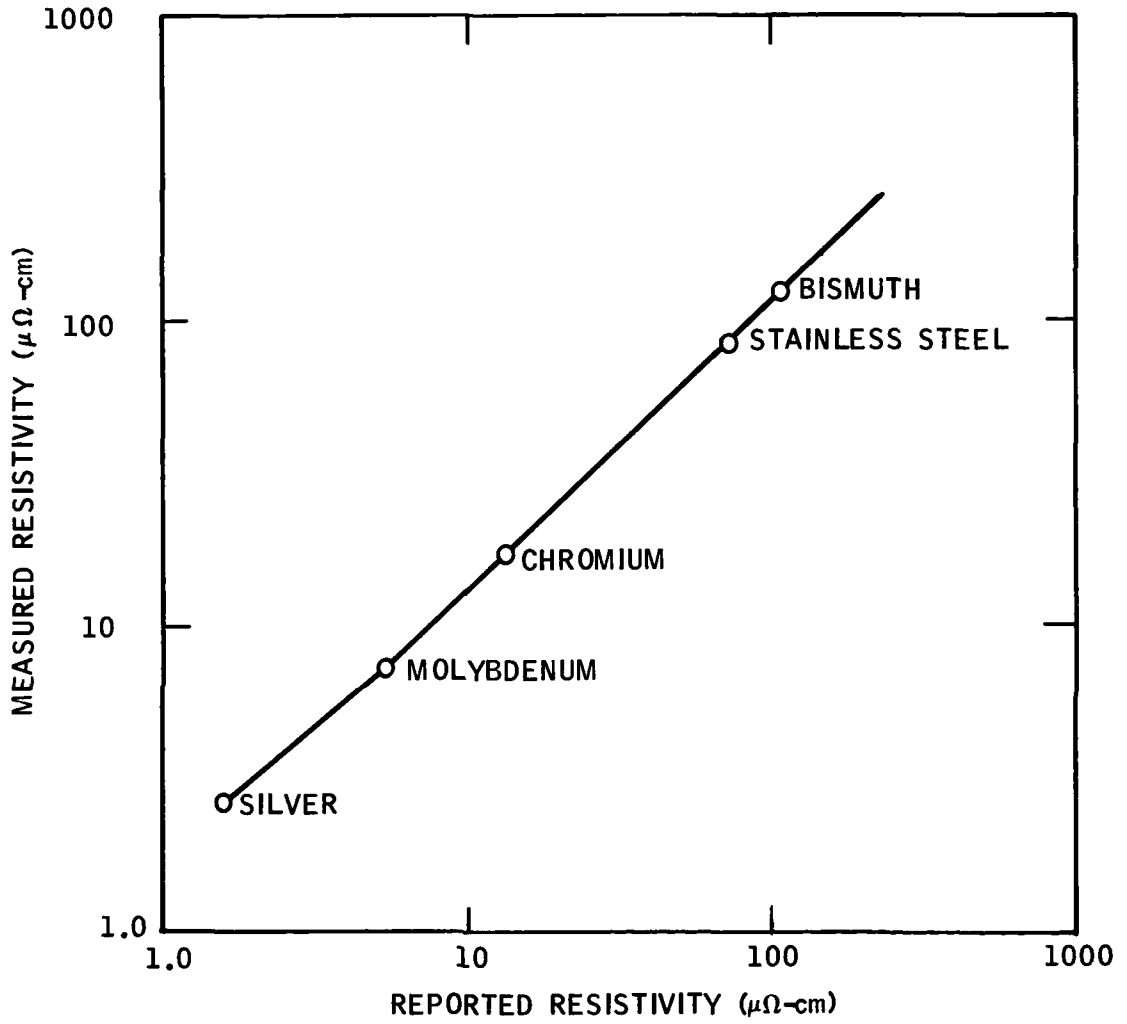Alloys Prepared for this Investigation

Figure 18 Parabolic Oxidation Rate Constant for the Alloys Investigated
[Samples exposed to 538 C (1000 F) in Air]

APPENDIX A

CALIBRATION OF THE FOUR-POINT PROBE FOR
ELECTRICAL RESISTIVITY
DETERMINATION

APPENDIX A CALIBRATION OF THE FOUR-POINT PROBE WITH FIVE MATERIALS
OF LOW RESISTIVITY DETERMINED AT 20 C

APPENDIX B

OXIDATION TEST DATA

Table B-1

Oxidation Data at 538 C (1000 F)

Heat No.	Composition (%) Mo	Composition (%) X	Dimensions (in.) Length	Dimensions (in.) Width	Dimensions (in.) Diameter	Surface Area (cm²)	Starting Weight Sample plus Crucible (g)	0.5 hr	1.0 hr	2.0 hr	3.0 hr	5.0 hr	10.0 hr	25.0 hr
--	Blank Crucible						11.6007	-0.7 (-0.7)a	-0.7 (0.0)	-1.1 (-0.4)	-1.1 (0.0)	-1.1 (0.0)	-0.4 (0.0007)	-0.8 (-0.4)
--	100	--	0.368	0.190	0.167	2.105	13.4957	0.5 (0.5)	0.7 (0.2)	1.1 (0.4)	1.6 (0.5)	3.5 (1.9)	10.1 (6.6)	24.5 (14.4)
X = Rh														
H-205	20.0	80.0	0.366	0.183	0.182	2.154	14.0877	0.4 (0.4)	0.4 (0.0)	0.6 (0.2)	0.8 (0.2)	0.8 (0.0)	1.3 (0.5)	1.4 (0.1)
H-219	38.3	61.7	0.366	0.184	0.170	2.076	13.9678	1.0 (1.0)	1.4 (0.4)	1.5 (0.1)	2.1 (0.6)	3.1 (1.0)	5.0 (1.9)	8.6 (3.6)
H-220	48.2	51.8	0.365	0.216	0.203	2.540	14.6870	1.9 (1.9)	1.9 (0.0)	2.2 (0.3)	2.6 (0.4)	2.8 (0.2)	4.0 (1.2)	5.1 (1.1)
X = Ir														
H-209	15.0	85.0	0.376	0.206	0.205	2.540	16.2835	0.4 (0.4)	0.5 (0.1)	0.7 (0.2)	0.9 (0.2)	1.0 (0.1)	1.2 (0.2)	2.0 (0.8)
H-216	17.6	82.4	0.368	0.195	0.195	2.342	15.9517	1.2 (1.2)b	0.2 (-0.1)	0.8 (0.6)	1.0 (0.2)	1.0 (0.0)	1.7 (0.7)	2.5 (0.8)
H-217	19.7	80.3	0.363	0.177	0.173	2.076	14.9532	0.5 (0.5)	0.5 (0.0)	0.7 (0.2)	1.0 (0.3)	1.2 (0.2)	1.6 (0.4)	2.9 (1.3)
X = Pt														
H-214	42.0	58.0	0.365	0.168	0.167	2.034	13.8962	1.6 (1.6)	2.7 (1.1)	3.6 (0.9)	4.4 (0.8)	5.2 (0.8)	6.4 (1.2)	9.9 (3.5)
H-215	45.0	55.0	0.365	0.172	0.169	1.981	14.2696	3.7 (3.7)	4.3 (0.6)	5.6 (1.3)	6.1 (0.5)	6.8 (0.7)	8.8 (2.0)	11.4 (2.6)

Accumulated Weight Gain (mg) for Indicated Total Time at 538 C (1000 F)

a Numbers in parentheses give the incremental weight gains for each period of exposure.
b Weighing believed to be in error.

B-1

Table B-2

Oxidation Data at 982 C (1800 F)

Heat No.	Composition (%) Mo	X	Dimensions (in.) Length	Width	Diameter	Surface Area (cm²)	Starting Weight Sample plus Crucible (g)	Accumulated Weight Loss (mg) for Indicated Total Time at 982 C (1800 F) 0.25 hr	0.5 hr	0.75 hr	1.0 hr	1.25 hr	1.5 hr	5.0 hr	10.0 hr
--	Blank Crucible 100	0	0.369	0.179	0.167	2.033	11.5982	(0.000)a	(0.000)	(0.000)	(0.000)	(0.000)	(0.000)	(0.000)	(0.000)
							13.4624	0.428 (0.428)	0.829 (0.401)	1.178 (0.349)	1.482 (0.304)	1.665 (0.183)	1.828b (0.163)		
X = Rh															
H-205	20.0	80.0	0.367	0.183	0.180	2.144	14.0611	0.012 (0.012)	0.015 (0.003)	0.017 (0.002)	0.019 (0.002)	0.021 (0.002)	0.023 (0.002)	0.047 (0.025)	0.109 (0.062)
H-219	38.3	61.7	0.366	0.189	0.183	2.203	13.9281	0.373 (0.373)	0.513 discontinued						
H-220	48.2	51.8	0.365	0.216	0.193	2.465	14.5043	0.480 (0.480)	0.848 (0.368) discontinued						
X = Ir															
H-209	15.0	85.0	0.375	0.205	0.205	2.527	16.3055	0.061 (0.061)	0.071 (0.010)	0.080 (0.009)	0.087 (0.007)	0.086 (0.001)c	0.086 (0.000)	discontinued	
H-216	17.6	82.4	0.369	0.194	0.187	2.283	15.7154	0.122 (0.122)	0.139 (0.017)	0.164 (0.025)	0.178 (0.014)	discontinued			
H-217	19.7	80.3	0.364	0.178	0.171	2.032	14.9022	0.133 (0.133)	0.194 (0.061)	0.246 (0.052)	0.279 (0.035)	0.299 (0.020)	0.312 (0.013)	discontinued	
X = Pt															
H-214	42.0	58.0	0.365	0.173	0.159	1.989	13.8739	0.484 (0.484)	0.774 (0.290)	discontinued					
H-215	45.0	55.0	0.365	0.168	0.168	1.947	14.2789	0.500 (0.500)	0.826 (0.326)	discontinued					

a Numbers in parentheses give the incremental weight losses for each period of exposure.
b Molybdenum sample completely consumed in 1.5 hours.
c A weight gain.

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The results of an investigation of the hexagonal-close-packed intermetallic compound found in the Mo-Rh, Mo-Ir, and Mo-Pt systems are reported. The compound was characterized by determining room and elevated-temperature hardness values, room-temperature electrical resistivity, oxidation resistance during exposure to air at 538 C (1000 F) and 982 C (1800 F), and corrosion resistance to aqueous solutions containing 41.6 or 70.9% nitric acid. The room-temperature hardness of the compound is reported to range between 300 and 675 DPH and is inversely proportional to the molybdenum content of the alloy. The molybdenum-iridium alloys are harder than the sapphire indentor at test temperatures between 1093 and 1649 C (2000 and 3000 F). The oxidation resistance of the alloys at 538 C (1000 F) decreases as the molybdenum content increases. Only the 80% Rh - 20% Mo alloy resisted oxidation at 982 C (1800 F) during a 10-hour exposure. All the alloys resist corrosion by dilute and concentrated nitric acid.

A-31408

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Molybdenum						
Rhodium						
Iridium						
Platinum						
Hardness						
Electrical resistivity						
Corrosion						
Oxidation						

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