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COMPOSITIONAL EFFECTS ON MECHANICAL PROPERTIES OF HAFNIUM-CARBIDE-STRENGTHENED MOLYBDENUM ALLOYS

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16 Abstract

The mechanical properties of swaged rod thermomechanically processed from arc-melted Mo-2Re-Hf-C alloys containing as much as 0 9-mol% HfC have been evaluated. The low-temperature ductilities of these alloys were not influenced by the amount of HfC present but by the amount of Hf in excess of stoichiometry Maximum ductility occurred at 0.2- to 0 3-at % excess Hf. At 0 3- to 0 5-mol% HfC, alloy strength varied directly with the Mo content of extracted carbide particles, both decreasing as the amount of excess Hf increased Additions of 2-at. % Re had little effect on strength or ductility Tensile and creep strengths of Mo-2Re-0 7Hf-0.5C alloy equaled or exceeded those of other high-strength Mo alloys

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COMPOSITIONAL EFFECTS ON MECHANICAL PROPERTIES OF HAFNIUM-CARBIDE-STRENGTHENED MOLYB DENUM ALLOYS

by Walter R. Witzke

Lewis Research Center

SUMMARY

Arc-melted Mo-2Re-Hf-C alloys containing as much as 0.9-mol% hafnium carbide (HfC) were thermomechanically processed to swaged rod for the evaluation of compositional effects on low-temperature ductility and high-temperature strength. Tensile ductile-brittle transition temperatures of these alloys were independent of HfC content in the range studied but varied with the amount of Hf in excess of stoichiometric requirements. Maximum low-temperature ductility occurred at excess Hf contents of 0.2 to 0.3 at.%. High-temperature strengths at 0.3- to 0.5-mol% HfC content increased as the molybdenum (Mo) content of the extracted carbide particles increased. The Mo content of the particles, in turn, increased with a decrease in excess Hf in the alloy. Maximum strengthening for creep at 1315° C was projected for no excess Hf in the alloy, corresponding to about 6-wt% Mo in the HfC particles. Additions of 2-at.% rhenium (Re) to the Mo-Hf-C alloys had little effect on either strength or ductility. At 1315° C the tensile strengths of the Mo-2Re-0.7Hf-0.5C alloy exceeded those of the Mo alloys TZC and Nb-TZC, while the creep strength of this alloy was comparable to that of Nb-TZC.

INTRODUCTION

The development of molybdenum (Mo) alloys during the last decade has shown the usefulness of carbides of titanium (Ti) and zirconium (Zr) in providing high strength at high temperatures (refs. 1 and 2). Because of the potential of greater stability with hafnium carbide (HfC) as a dispersed phase at high temperatures, Raffo studied the Mo-Hf-C alloys and reported outstanding mechanical properties for various composi-

tions, particularly for thermomechanically processed materials (refs. 3 to 5). The present study extends this work for the purpose of examining the effects of excess Hf on the mechanical properties of HfC-strengthened Mo alloys. An additional aim was to determine if any ductilizing effect would result from the addition of 2-at. % rhenium (Re) to Mo-Hf-C alloys. The effectiveness of dilute concentrations of Re in lowering the ductile-brittle transition temperature of unalloyed Mo has been demonstrated (ref. 6).

In this investigation the effects of HfC and of excess Hf on both the low-temperature ductility and high-temperature strength of Mo alloys were examined. Swaged Mo-2Re-Hf-C alloys were evaluated by tensile tests at low temperatures (-185° to 425° C) and at high temperatures (980°, 1315°, and 1650° C) and by creep tests at 1315° and 1650° C. The annealing behavior of these alloys was examined, and the tensile and creep properties of recrystallized Mo-2Re-Hf-C alloys were determined. The relations between alloy strength and extracted carbide particle composition are discussed.

EXPERIMENTAL PROCEDURE

Materials and Processing

The seventeen Mo-2Re-Hf-C alloys in this study contained from 1.6- to 2.4-at. % rhenium, 0.2- to 1.1-at. % hafnium, and 0.1- to 1.9-at. % carbon. Oxygen and nitrogen impurities in the fabricated alloys ranged from 31 to 143 ppm and 2 to 18 ppm by weight, respectively. The content of HfC in mole percent was assumed to be equal to the content of either Hf or C in atomic percent, whichever was lower. Hafnium contents were determined by a spectrophotometric method (ref. 7). Carbon determinations were made by a combustion chromatographic procedure (Leco) and are considered to have a precision of about ±5 percent.

Materials used for this investigation included commercial Mo, Re, and Hf powders and spectrographic-grade graphite powder. The Hf powder contained about 3-wt% zirconium (Zr). The alloy materials were prepared by a thorough blending of the metal and graphite powders, hydropressing them, and then sintering them into electrode shapes followed by vacuum arc melting into 5.8-cm-diameter ingots weighing about 3500 g. Billets machined from these ingots were solution annealed for 30 minutes at 2200° C and immediately extruded at 2040° to 2200° C with a reduction ratio of 8:1. The extruded rods were further processed at 1370° C by swaging to 89 percent reduction in area, This thermomechanical processing procedure follows that described in reference 4, which resulted in the best strength properties in the Mo-Hf-C alloys.

For use only in the low-temperature-ductility study, binary Mo alloys containing as much as 1.1-at. % Hf or 6-at. % C were prepared from 90-g-button arc melts. Follow-

ing triple button melting, each alloy was cast into a 1.5-cm-diameter cylindrical shape, canned in Mo tubing, and swaged at an initial temperature of 1700° C and a final temperature of 1480° C to 0.47-cm-diameter rod, about 90 percent reduction in area.

Specimens were annealed to determine recrystallization behavior. Annealing treatments were conducted in a vacuum furnace at less than 10^{-3} -N/m² pressure with a tungsten wire-mesh resistance heating element. Specimens were wrapped in Mo foil to reduce decarburization during annealing.

Electropolishing of tensile specimens to remove surface cracks was conducted at about 10 volts in concentrated sulfuric acid containing a few drops of water. A cooling bath was required to keep the temperature of the acid below about 50° C.

Evaluation

The Mo-2Re-Hf-C alloy materials were evaluated by tensile and creep tests in the as-swaged and recrystallized conditions. Following mechanical testing, the specimens were examined microstructurally and analyzed chemically.

Mechanical tests. - The Mo-2Re-Hf-C alloy specimens for tensile and creep tests had a 2.5-cm gage length and a 0.25-cm reduced diameter. The binary Mo-C and Mo-Hf alloy specimens were subsize, having a 2.5-cm gage length with a 0.19-cm reduced diameter in the test section. Tensile tests were conducted in a vacuum chamber below 10^{-3} N/m² at 980° , 1315° , and 1650° C. Other tensile tests were performed on electropolished specimens in air at lower temperatures, ranging from -180° to 425° C. A constant crosshead speed of 0.13 cm/min was used.

Creep tests were conducted under vacuum conditions at 10^{-4} N/m 2 in a conventional beam-loaded machine. Step-load creep and creep rupture tests were determined from the movement of tungsten extension rods which connected the specimen button-heads to a linear variable differential transformer. Test temperature was measured and controlled by a W/W-26Re thermocouple attached to the reduced section of the specimen. Stress values in step-load tests were based on initial cross-sectional area, and total strain was limited to 10 percent. Estimated stresses for 10^{-7} -sec $^{-1}$ creep rate were determined from log-log data plots of stress as a function of minimum creep rate.

Microstructural studies - Optical and electron microscopy were employed in examining alloy microstructures - Thin films for transmission electron microscopy were prepared from disks about 0.03 cm thick taken from the transverse cross section of each material. The disks were thinned electrolytically in a solution of sulfuric acid and ethyl alcohol at 30 volts.

The second-phase particles were extracted from the unstressed button-heads of tensile and creep specimens by chemical dissolution of the matrices, using a solution with a typical composition of 90-cm³ methanol, 10-cm³ bromine, and 10-gtartaric acid. The carbide particles were isolated by centrifuging, decanting, rinsing in methanol, and drying. X-ray diffraction analyses provided lattice parameter values of the particles. Emission spectrographic analyses of the carbide particles yielded quantitative data on the metallic elements present, with a precision of ±10 percent. Considering the minuteness of the spectrographic sample, analyses resulting in recovery of less than 50 wt% of the total metallics were not considered. Analyses were also held to be invalid when Zr content ranged beyond 2 to 4 wt% of the Zr + Hf content. The Mo contents of the extracted particles were calculated from the spectrographic results by using the ratio Mo/(Mo + Hf + Zr) and were expressed as the weight percentage of total metallics present in the carbide particles.

RESULTS AND DISCUSSION

Microstructure and Annealing Behavior

A typical microstructure of an as-extruded Mo-2Re-Hf-C alloy is shown in figure 1. The general appearance of the extruded alloys consisted of alternating layers of recrystallized and worked material. Twenty to 90 percent of the total area was recrystallized following this hot-working operation. Swaging at 1370° C yielded a fully worked structure. Table I lists the alloys investigated and the hardnesses measured for the extruded and swaged conditions.

Typical effects of 1-hour annealing on the microstructure of a swaged alloy, Mo-1.93Re-0.60Hf-0.49C (at.%), are shown in figure 2. Little change in microstructure occurred at 1370° C except for slight increases in fiber width. At 1650° C about 5 percent of the structure was recrystallized, and after annealing at 1930° C, the alloy was completely recrystallized.

The 1-hour recrystallization temperatures of the swaged Mo-2Re-Hf-C alloys are given in table I. Also included, as a base line, are data for an arc-melted Mo - 3.48-at. %-Re alloy. The 100-percent recrystallization temperatures for those alloys containing excess Hf are shown in figure 3. In general, the recrystallization temperature increased with the calculated content of HfC.

In figures 4 and 5, transmission electron micrographs of two of the swaged, HfC-precipitate-strengthened Mo-2Re alloys display a uniform dispersion of fine particles as small as 5 nm in diameter and a network of pinned dislocations. After tensile testing at 1315° C an increase in dislocation density was observed. Although the fine-particle dispersion appeared to be unchanged, there was a decided increase in the number of large platelet particles. The creep-tested structure (fig. 4(c)) with its long-term

exposure to the 1315° C test temperature differed from its tensile-tested counterpart (fig. 4(b)) in that the average size of the dispersed fine particles was apparently increased and the dislocation density was less. Tensile testing at 1650° C (fig. 5(c)) produced an overall change from a fine to a coarser particle size, indicating rapid growth of the second-phase particles at this temperature. Creep testing at 1650° C generally resulted in complete recrystallization of the matrix and growth of the uniformly dispersed, fine particles into a sparse and erratic dispersion of large (40 to 1200 nm) particles.

Low-Temperature Ductility

The effect of alloy composition on the low-temperature ductility of HfC-strengthened molybdenum alloys was studied by determining the tensile ductile-brittle transition temperature (DBTT) of swaged and electropolished alloy materials. The materials evaluated included unalldyed Mo, Mo-C, and Mo-Hf binary alloys, three Mo-Hf-C alloys from previous investigations (refs. 3 and 4), and Mo-Re-Hf-C alloys from the present study. The DBTT value for each alloy was designated as the lowest temperature at which tensile testing would result in a 5 percent reduction in area.

Tensile ductility test results are presented in table 11. From previous work (ref. 6) it was observed that increasing the Re content from 1.6 to 2.4 at. % in recrystallized molybdenum decreased the DBTT by about 15° C. In swaged Mo-Re alloys this variation in Re content might be expected to yield larger changes in DBTT. However, for this analysis the differences in Re for the quaternary alloys were neglected. In figure 6 the tensile DBTT values of the HfC-strengthened molybdenum alloys are seen to have no apparent relation to the HfC content, as indicated by the scattered results.

For comparison, as-swaged binary Mo-C and Mo-Hf alloys were also evaluated. Test results shown in table 11 and in figure 7 indicate that the addition of C to Mo increased the tensile DBTT for C contents to 6 at. %. In Mo-Hf alloys the initial effect of Hf was to lower the DBTT to a minimum at about 0.3-at. % Hf. Approximately 1-at. % Hf was required to raise the tensile DBTT back to the original value for unalloyed Mo. Fiber width measurements of the Mo-Hf binary alloys could not be correlated with the changes in DBTT. Similar DBTT effects for Hf and C have been observed in binary tungsten (W) alloys, and, moreover, the low-temperature ductility of the HfC-strengthened W alloys was found to be a function of the amount of Hf or C in excess of that required for formation of stoichiometric HfC (ref. 8). This latter relation was explored in the Mo-2Re-Hf-C alloys also.

Figure 8 shows, for the Mo alloys, the effect on tensile DBTT of Hf or C in excess of stoichiometric HfC. The band enclosing the Mo-2Re-Hf-C alloy data indicates that

excess Hf reduced the DBTT to a minimum at about 0.2-at. % Hf, while excess C increased the DBTT continuously to 0.3-at. % C. The three Mo-Hf-C alloy data points are in good agreement with the quaternary alloy results. The minimum shown for the HfC-strengthened Mo alloys and the Mo-Hf binary alloys occurred at about the same concentration of excess Hf. The differences in DBTT between these two data plots are believed to result mainly from differences in processing rather than from the presence of the second-phase particles.

Tensile Strength

Tensile test results for the Mo-2Re-Hf-C alloys at room temperature and at elevated temperatures are given in tables III and IV. The effect of calculated HfC content on the ultimate tensile strengths of swaged alloys at 1315° and 1650° C is presented in figure 9. The ultimate tensile strength σ increased with increasing HfC content in the range studied. The curves as drawn represent a parabolic relation, $\sigma \propto (\text{HfC})1/2$, as was found earlier for solution-annealed Mo-Hf-C (ref. 5). No maximum in strength was apparent as the HfC content increased to 0.88 mol%, the highest level of HfC present in the alloys investigated. The data shown in figure 9 are for alloys containing Hf in excess of the calculated stoichiometric HfC contents. The few alloys that contained excess C were generally stronger than those containing excess Hf. For example, at 1315° C for HfC contents ranging from 0.2 to 0.6 mol%, the excess-C alloys were more than 30 percent stronger than the average for the excess-Hf alloys.

Data for recrystallized Mo-2Re-Hf-C alloys presented in table IV similarly indicate, for HfC contents to 0.5 mol%, that an increase in HfC caused an increase in tensile strength at 1315° C. The recrystallized-alloy strengths at 1315° C averaged about $150 \, \text{MN/m}^2$ lower than swaged-alloy strengths for comparable HfC contents.

Creep Behavior

The creep rupture properties of swaged Mo-2Re-Hf-C alloys at 1315° C are presented in table V. Based on a creep rate of $10^{-7}~\rm sec^{-1}$, the effect of HfC content on the creep strengths of these alloys is shown in figure 10. For simplicity the data have been enclosed in a band which suggests a linear strengthening rate of about 200 MN/m² per mol% of HfC present, but the spread of the data indicates that differences in strength at a given HfC content can approach 250 MN/m². Examination of a segment of the data, as plotted in figure 11, showed that a major part of the strength differences can be attributed to the presence of excess Hf. For alloys containing 0.33- to 0.53-mol% HfC the

trend of the data indicates the relatively large influence that small amounts of excess Hf have in reducing strength. The strength of these alloys dropped at the rate of about 350 MN/m² per at. % of excess Hf. Therefore, the large range of creep strengths in figure 10 was probably due to compositional differences, namely, differences in excess Hf.

The effect of excess C on strength could not be determined from the few alloys available, but based on references 3 and 8, excess C could also be expected to cause some reduction in strength. This would further indicate that stoichiometric compositions of these alloys provide the best condition for maximum strength.

At 1650° C the step-load creep data for Mo-2Re-Hf-C alloys listed in table VI followed trends similar to those shown for creep rupture tests at 1315° C. Considering only those tests wherein the unstressed button-head microstructures indicated complete recrystallization on post-test examination, the 1650° C creep strength increased with increasing HfC and decreased with increasing amounts of excess Hf. Test results for the recrystallized Mo-2Re-Hf-C alloy at 1315° C using the step-load creep procedure are listed in table VII. Similar trends of increased strength with increased HfC content and decreased excess Hf are indicated.

Carbide Particle Effects

It has been assumed in this report that a stoichiometric HfC phase precipitates from the Mo or Mo-2Re matrix as fine, uniformly dispersed particles and that these second-phase particles are primarily responsible for the strength increases observed. As has been noted with other **second-phase-strengthened** materials, Raffo has shown that interparticle spacing can have a considerable effect on the minimum creep rate in Mo-Hf-C alloys (ref. 4). However, for this report, no statistical evaluation was made of the influence of such physical parameters as particle size, spacing, and morphology. Considering the large influence on strength shown by small changes in alloy composition, an attempt was made to evaluate the particle chemistry and its relation to alloy composition and strengthening.

While it can be assumed that HfC precipitates stoichiometrically, it is known that HfC and similar carbides can exist in a large range of carbon-to-metal ratios (ref. 9) and that their properties, such as lattice parameter and microhardness, vary with the carbon-to-metal ratio. Therefore, although this was not investigated because of the larger samples required for carbon analyses, it should be remembered that the carbide particles in the Mo alloys may be nonstoichiometric, which, in turn, may influence alloy strength.

Because of the large concentration of Mo surrounding the HfC particles in these alloys, this carbide-former could be expected to influence the composition of the second-phase particles. Nowotny and coworkers (ref. 10) have shown that Mo is soluble in HfC. Analyses of solvent-extracted particles from the Mo-2Re-Hf-C alloys indicated varying amounts of Mo to be present.

The results of emission spectrographic and X-ray diffraction analyses of the extracted carbide particles from unstressed portions of 1315° C creep specimens are given in table V. The lattice parameter values of this cubic structure ranged from 0.4604 to 0.4632 nm, compared to 0.46395 nm for HfC_{0.97} (ref. 11). Little or no Re was detected. The major metallic elements found were Hf, Mo, and Zr. The Mo contents were determined to represent 2 to 9 wt% of the total metallics present in the carbide particles. Because of the larger sample requirements, carbon analyses of these fine particles could not be made.

Figure 12 shows that the 1315° C creep strength increased as the Mo content of the carbide particles increased for those alloys containing 0.33- to 0.53-mol% HfC. It should also be noted (table V) that the lattice parameters for the extracted particles decreased with increased alloy creep strength. Figure 13 relates the Mo content of the carbide particles to the excess Hf in the alloy. Since the creep strength increased with increasing Mo content in the particles but decreased with excess Hf in the alloy composition, it was not unexpected that the Mo would decrease in the particles as the excess Hf content of the alloy increased. From figure 13 it can be seen that for calculated stoichiometric conditions in the alloy, that is, no excess Hf, about 6-wt% Mo would be found in the second-phase particles. Thus, the data indicate that the excess Hf in the alloy influences the Mo content of the carbide particles. The mechanism by which either or both of these parameters may affect the strength of the alloy is presently subject to conjecture.

The influence of excess C on the Mo content of the second-phase particles could not be determined because of inadequate data. The presence of excess C in the alloy should provide a greater potential for additional Mo or for a larger carbon-to-metal ratio in the particles. Both of these possibilities could reflect the generally higher tensile strength of Mo-2Re-Hf-C alloys containing excess C. It is interesting to note, however, that in the W-4Re-Hf-C system (ref. 8) the W content of the second-phase particles increased with increases in excess C content but was deleterious to strength.

Similar treatment of extracted carbide particle data from swaged 1315° C tensile specimens, given in table III, yielded the same trends of increased tensile strength with increased Mo content in the second-phase particles and increased Mo content with decreased excess Hf in the alloy.

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Comparison of Mechanical Properties with Those of Other

Thermomechanically Processed Molybdenum Alloys

The strength increases attainable by optimizing the distribution of the carbide phase in Mo-Hf-C alloys by thermomechanical processing were described in reference 4. The most advantageous procedure, solution treatment at 2200° C followed by direct extrusion, was used to prepare the Mo-2Re-Hf-C alloys in this investigation. Previous investigators have used other in-process solution treatments to develop strength improvements in the molybdenum alloys TZM, TZC, and Nb-TZM (refs. 2, 12, and 13). Tensile and creep strength comparisons of these Mo alloys with a Mo-2Re-0.7Hf-0.5C alloy are shown in figures 14 and 15.

Molybdenum alloys strengthened with 0.5-mol% HfC exhibited tensile strengths at 1315° C that were 50 to 60 percent greater than the tensile strength of TZM with inprocess solution treatment (fig. 14). Larger strength improvements, greater than $3\frac{1}{2}$ times, were noted at 1650° C. Materials from TZC and Nb-TZC alloys, which have carbide levels more quantitatively similar to that of the Mo-2Re-0.7Hf-0.5C (at.%) alloy but which were given a solution treatment following fabrication, were also weaker. The thermomechanically processed and HfC-strengthened alloys were 35 percent stronger than TZC at 1315° C and about 25 percent stronger than Nb-TZC at 1650° C.

Figure 15 shows a comparison of the 1315° C creep strengths of in-process solution-treated Mo alloys. The Mo-2Re-0.7Hf-0.5C and Nb-TZM alloys were the strongest, with Mo-0.6Hf-0.5C having slightly lower creep strength. The TZC alloy, with a somewhat higher carbide content than these alloys, had only an intermediate strength. The low carbon content of TZM was reflected in a strength level about one-half those of the stronger alloys.

CONCLUDING REMARKS

High strength and ease of fabrication are desirable characteristics in Mo alloys. The HfC-strengthened Mo alloys have been shown to possess excellent high-temperature tensile and creep strengths as well as good low-temperature ductility, provided the alloy composition is carefully controlled to limit the amount of excess Hf or C present. For good strength properties as well as below-room-temperature ductility, these alloys should contain less than 0.4-at. % excess Hf.

The value of adding 2-at. % Re to Mo-Hf-C alloys appears questionable. From the few comparative data available, little improvement due to this Re addition has been observed in the low-temperature duetilities and high-temperature strengths of these alloys. One qualitative observation, however, is that an improvement in product yield generally

accompanies the addition of Re.

For good high-temperature strength and for ductility properties similar to those of lower strength commercial molybdenum alloys, an alloy composition such as Mo-0.7Hf-0.5C (at.%) should provide satisfactory performance.

SUMMARY OF RESULTS

Thermomechanically processed Mo-2Re-Hf-C alloys containing as much as 0.9-mol% hafnium carbide (HfC) were examined to evaluate the effects of excess Hf on mechanical properties. The results from this study were as follows:

- 1. The low-temperature ductility of HfC-strengthened molybdenum (Mo) alloys was not dependent on the amount of HfC present but was related to the amount of Hf in excess of stoichiometry. The low-temperature ductility was maximum at an excess Hf content of about 0.2 at. %.
- 2. At 0.3- to 0.5-mol% HfC content, alloy strength increased with the Mo content of the carbide particles. The Mo content, in turn, increased as excess Hf decreased. With no excess Hf, the Mo content of the particles is projected to be about 6 wt%, and alloy strength should be maximized.
- 3. The addition of 2-at. % Re to Mo-Hf-C alloys contributes little to low-temperature ductility or high-temperature strength.
- 4. The 1315[°] C tensile strengths of Mo alloys containing 0.5-mol% HfC exceeded those of the molybdenum alloys TZC and Nb-TZC by 35 percent. The 1315[°] C creep strengths of Mo-2Re-0.7Hf-0.5C and Nb-TZC alloys were comparable.

Lewis Research Center,

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TABLE I - COMPOSITION, HARDNESS, AND IEECRYSTALLIZATION

CHARACTERISTICS OF Mo-2Re-Hf-C ALLOYS

Alloy	Re	Hf	С	Calculated	As extruded	As swaged	One-hour				
		ntent o	of swaged %	HfC content, mol%	Wielront a handwage						
68	3 48	0	(0 19)	0	196		1200				
88	1 79	76	11	11	189	322	<1760				
87	2 25	89	13	13	207	342	1650				
100	1 64	21	24	21	182	425	< 1930				
96	1 64	66	22	22	205	483	1870				
95	1 99	82	23	23	206	351	1760				
97	2 18	1 05	34	34	240	319	>1930				
105	1 68	61	40	40	260		< 1930				
86	2 20	95	40	40	227	304	1760				
74	1 93	60	49	49	230	413	1930				
94	2 40	59	50	50	256	366	1870				
104	2 27	70	50	50	266	342	< 1930				
75	1 96	56	1 88	56	232	493	1790				
73	1 97	58	70	58	219	370	1650				
102	2 23	82	66	66	268	390	<u><</u> 1930				
101	1 93	97	82	82	222	360	< 1930				
98	2 31	85	98	85	212	429	1700				
103	2 05	1 00	88	88	276	483	>1930				

TABLE Π DUCTILE-BRITTLE TRANSITION TEMPERATURES OF SWAGED AND ELECTROPOLISHED MOLYBDENUM ALLOYS

Alloy	Re Solute	Hf conte	C (after test)	Calculated HfC con. tent, mo1%	Hf Excess at		Ductile-brittle transition temperature for ⁵ per cent reduction in area,
				Unalloy	ed Mo		
					.=		-82
				Mo-C a	lloys		
			0.81				-18
	_,		1 45				1
			3 52				4
			6.05				7
			— · · · · ·	Mo-Hf a	illoys		
		0. 10		-: :-			157
		21					157
		31				,	184
		40					-146
		51				,-	-168
-,		78		·			126
		1 10					-73
			· · · · · · · · · · · · · · · · · · ·	Mo-Hf-C	alloys		
39	<u>-</u>	0.60	0 62	0. 60		0.02	32
40		1 04	26	26	0.78		.9
42		1 09	1 13	1 09		04	< -32
	<u> </u>	<u> </u>		Mo-Re-Hf	C alloys	3	and the state of t
88	1 79	0.76	0 06	0.06	0 70	-,,	0
100	1 64	21	40	21		0.19	121
87	2 25	89	24	24	65		-26
96	1 64	66	29	29	37		-27
95	1 99	82	34	34	48		42
105	1 68	61	40	40	21		- 87
86	2 20	95	44	44	51		93
74	1 93	60	50	50	10	;	12
94	2 40	59	56	56	03		32
75	1 96	56	2 59	56		2 03	42
73	1 97	58	85	58		27	260
97	2 18	1 05	59	59	46		27
103	2 05	1 00	84	84	16		12
101	1 93	97	85	85 or	12		18
98	2 31	85	1 05	85		20	38

TENSILE PROPERTIES OF SWAGED Mo-2Re Hf-C ALLOYS AND LATTICE PARAMETERS AND CHEMISTRY OF EXTRACTED CARBIDE PARTICLES TABLE III

The part of the				· · · · · · · · · · · · · · · · · · ·														,																			
Comparison Com	cles	hic analysis	Mo content,	wt% of total metallic content		!!	į	i		ļ	;	i :		į	i	1		į	į	į	ŀ	1	}	1	ł	-	1	1	ı		!	3.6	,	:		(g)	
Comparison Com	parti	ograp	Zr	29		i	•	i		i	1	l		i	ł	1	i	1	i	1	i	1	1	7	1	!	1	ŀ	i.	!	į			ŀ	l	29	ī
Comparison Com	arbide	spectr	Re	at, wt 9			i			i	!	1 1		1 0 1	1 1	1 8	ı	-	i	-	-			1							i	<0.1		1	!	0.1	
Comparison Com	cted c	ssion	Mo	Conte	İ		:								ï	į	!	į	į	1	1	ļ	!	-	1	1		,	ı					1	1	3 1	
A	Extra	Emi	Hf					i I						į	i	1	1			1		- !	i	İ			,	i 		,				-	!		I
The part of the		Lattice	param- eter	a _o , nm					1			1				0.4626	1		-	0.4612		1			-				-	ı	t 1		ı	[]		0.4630	:
Test He C Calculated H C Test term Yield strength Fracture Ultimate Heath Leath Leat	匚	in area, percent	**************************************		n,	1 8	n i	n m	ę	7 1	6 G		3	13	83	88	> 95	2	74	74	91	B2	8m	£	8E	8	85	82	4 6	3	88	73	90	5	84	83	92
Re Hf C Calculated Hf C Test tem Yield strength, Fracture 1.0 1.	Elongation,	percent			18	, F	CT C	92 46		1 (ထင	67 67		4	ω	14	20	2	10	10	16	10	6	16	17	န	80	11	25	က	8	6	34	2	6	ıA	49
Reconstruction Rec			MN/m ²		924	458	100	20			641	451		u	099	432	160	ı	848	199	258	1027	663	434	363	ŧ	663	511	209		800	26 8	18,	I I	0-8	9 _E G	233
Recontent, Action Actio						-	i I	ī	ų,	6 4 5		į		D/16			!	1220		î	1			1	1	1014	1	1	ı	1214	i	i	•	1269		i	£°
3 48	Yield strength	at 0.2 percent offset,	MN/m ²		841	433	202	40	000	903	627	1454		965	651	416	148	1151	834	655	100	910	658	423	346	822	649	200	185	1110	772	547	174	1186	772	567	229
He C Calculated He Exc. Exc. Est	Test tem	perature. ^o C			25	G G	00 1	1450	ī	n 7 0	086	1650		25	980	1315	1650	25	086	1315	1650	28	08m	1518	1450	25	086	1315	1650	25	086	1315	1650	2B	9 80	# I H	B 50
He C Calculated He Exc. Exc. Est	ر	988	. %		;		!				!	ļ		1	<u> </u>	1	ì	ı			ŀ		!	į.	1			ı	•		!			ļ	í	j	T
Solute content, Action A	Ħ	Exc	at				ı			į				i	1		92		ı			1		0.44	19		!	0 59	51	ſ	ı	0.71	81	i	., .,	0 55	28
Solute cont at % 3 48 1 73 0 7\$ 1 64 0.8\$ 2 18 1 05 2 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 0 95 2 2 2 0 95 2 2 2 0 95 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	_		%low		0) 		->		7. 7		11 60	3				>	0.21	21	21	20	0.22	22	22	47	0.23	23	23	31	0.34	34	34	24	0.40	40	40	37
	ပ	(after test)	tent,		n	1				g ,	(a)	1 g	3	(g)	(a)	0. 13	£1	(a)	(a)	0.24	20	(a)	(a)	0.22	47	(a)	(a)	0 23	31	(a)	(a)	0.34	24	(a)	(a)	0.40	37
	Ħ		te con	at %				. —				-		68°.				0.21				0.88				0. 32			-					95			
	Re		Solu															•		****		64				66										-,-,-	\exists
	Alloy				89	3			r	n n				8.1			-	100				9 m				35			-	-							

-								
2 L	1 1 1	9 6	1 1	7 7	1 : 6	• e	e ! 9	1 ; & .
, , 8		ا ما ا	1 1		1 8 1	1 12	. O .	. ை i
1 1 63 1	1 1	- <0.01 1		0.2	. ' 12 '	<0.01	. 6	<0.01 1
1 0 1		~	•					
2	1 !	ما ا	1 1	່ ຜ່	4	2 2 2 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1		4 6 3
- 67	1 1	47		64.8	- 19	57	52.	63
0.0624	,	0.4620	0, 4604	0, 4623	0. 4617	0.460	0. 46∑0	0. 4613
21 - 74 83	2 E E E E	74 (76)	" E - 4, 5	0 - 61 78	5 73 64 88	10 76 37 85	2 86 72 94	22 73 73 68
3 8 11 16	41 8 9 78	14 22	8 12 27	<1 8 12 15	10 12 42	4 9 17 38	1 8 9 29	m 0 1 1 1 1 1 2 1 1 1 1 1 1 1 1 1 1 1 1 1
 848 576 343	738 569 250	772	979 827 207	910	862 636 209	1014 652 212	86 75 ⁷	3627 896 710 302
M204	71.2	i I	1036	979	1275	3 8 5	1013	!
1076 834 557 329	1020 731 553 235	752	587 738 192	1696 910 758 375	1207 841 612 186	5434 8 ₃ 9 6 ₀ 5 103	1310 841 731 184	14 4 8 7 6 7
25 980 1315 1650	25 980 1315 1650	135 148	25 380 1315 1c50	25 980 1315 1650	25 980 8 15 650	25 980 115 150	25 980 1315 1650	25 980 1315 1650
1 1 1	1 1	I I	1 32 1 c4	0. 12			0, 13	1 1 1
 0.11	- 0.09	0 20 23	I I	(0.16	 0 15 26 ·		0. 12 24
0.43 43 43	0 50 50 50	an a ! O	0 58	0, 58 58 58 54	0.66 66 66 47	0 82 82 82 71	0 35 35 35 34	83 83 70 70
(a) (a) 0.43	a m ?	(a) (a) 0,5°	(a) (a) 1 88 2 20	(a) (a) 0, 70	(a) (a) 0.66	(a) (a) 0.82 71	(a) (a) 0.98 34	(a) (a) 0.83
0	e 0	0 70	0 56	0.53	0.82	0.97	92 0	0
1 33	0 e 8	2 Z7	W T	£ M M	2 23	1 93	2 3 3 2	2 05
70 1	e m	e or	Les ea	73	102	101	m	- M

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^aNot determined; taken as equal to carbon content of specimen at 1315° C bwhere Zr/(Zr + Hf) did not equal 2 to 4 percent, Mo contents were considered to be invalid.

TABLE IV $\,$ - TENSILE PROPERTIES OF RECRYSTALLIZED $\,$ Mo-2Re-Hf-C ALLOYS AT 1315 $^{\rm O}$ C

Alloy	Calculated	Excess	Yield strength	Ultimate	Elongation,	Reductior	Average grain
	HfC con-	Hf,	at 0 2 percent	tensile	percent	in area,	diameter in
	tent,	at %	offset,	strength,		percent	button-head
	mol%		MN/m^2	MN/m ²			after test,
							$\mu {f m}$
100	0 002	0.21	40	115	28	95	46
103	01	99	134	192	16	54	182
104	03	67	152	318	12	40	24
96	06	60	164	321	14	84	20
88	06	70	127	228	36	95	72
101	11	86	165	367	14	67	22
87	12	77	174	241	17	84	35
102	17	65	164	40 1	15	72	25
95	18	64	183	350	24	93	59
74	22	38	225	361	17	57	50
98	26	59	163	370	18	90	37
94	31	28	208	311	22	90	43
73	32	26	148	339	24	69	88
75	33	23	220	341	23	69	83
86	45	50	166	347	19	93	47
97	52	53	194	507	17	72	46

TABLE + - CREET RUTHURE PROTERMIES OF BWAGED MO-2Re Hf-C ALLOYS AT 1315° C AND LATTICE PARAMETERS AND CHEMISTRY OF EXTRACTED CARBIDE PARTICLES

Alloy	Alloy Calculated average	JH	i I	Stres:,	Minimum creep	Rupture life,	Estimated stress for	Total elonga-	Carbi MN/m ² .	de part	cles leve	extrac	ted fr men	Carbide particles extracted from 241- $\rm MN/m^2$ -stress-level specimen button-heads
	HfC con- tent,	Average		Е	rate, sec ⁻¹	hr	10 ⁻⁷ -sec ⁻¹ creep rate,	tion, percent	Lattice	Emiss	ion s	ectro	graph	Emission spectrographic analysis
	%Iom	solute, at %	é, %				MN/m ²		param eter,	Ħŧ	Mo	Re	Zr	Mo content,
									a O	ပိ	ntent	Content, wt%		wt% of total metallic
														content
89	0	 	! ! !	41	4. 0×10 ⁻⁷	158	34	75	1	7	-		l I	1
				55	2 4×10 ⁻⁶			22	1 1	: :	<u>.</u> 		! ! !	!
				69	1 2×10 ⁻³	5 67		80		1	-	1	:	1
88	Ø0 0	0 70	1	103	2 6×10-7	62.7	78	28	-	1	!	!	1	
				172	1 6×10 ⁻⁶	7 24		18	1	1	1	1 1	1 1	
				241	4.0×10 ⁻⁶	2 14		-			-] 	
100	0.11	0 10	1	172	7 1×10 ⁻⁹	191	31\$	11	 			1	! !	
			· ; · ,,	207	2 0×10 ⁻⁸	172		9	1	:	1	!	!	
				241	2 9×10 ⁻⁸	110		12		!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!	1		1	
95	0 19	0.63	[]	138	1 0×10 ⁻⁷	72 8	157	21		1			1	
				172	6.6×10^{-8}	48 3		15	1 1	!	<u> </u>	1	1	
				241	5 2×10 ⁻⁷	10.8		13	~		-			
87	0.22	- 29 0	.1 	172	4. 0×10 ⁻⁷	18 5	13\$	01			I	 		!
				241	3 0×10 ⁻⁶	2 90		, ¦		1	:		!	
94	0 33	0 28 -	1	172	3 3×10 ⁻⁹	 	264	ı	 		!		1	1 1
				207		p35		18		1 '	1 1			1 1
		1	1	241	3 ×10 ×	m [9]		18	0.4627	68 5	3 7	<0.1	2 1	5 O
74	0 34	0 28	-	172	8 2×10 9	675	288	22	1		1	1	1	1 1
				241		72 4		11	0 4625	52 5	1 9	<0.2	1 5	3. 4.
·				276	9×10	109		16	 		<u>-</u>	: I : I : I	!	1
				414	6. 0×10 ⁺	4.24		7	1	!	-		1 1	1

į

2 - 1	5.1	 (a)	(a)	20	8.2	 	 (a)	(a)	5.1
2.3	1 3	4	C U	2 0	1 8 -	2 1	0.7	2 8	1 3
<0.1	<:0, 1	<0.1	<0.01	<0.2	<0.2	<0.1	<0.1	<0.2	<0 1
1.4	6 c u i	 2 6	4.1	3 9	5.8	3 6	4, 0	2 9	2 9
61 0	50 5		54.9	C	63 5	0 09			52 5
0 4632	0 4618	 0 0 4627	0. 4611	0 4611	0.4609	0.4610		0 4616	 0.4618
19 16 13	13	19 17 14	13 11	5 2 12 4	8 10 11	7 8	14 23 20	18	19 18 (7)
195	325	177	2,65	403	178	347	185	184	192
153 23 9 97	78 2 74 3	159 44. 5 6. 21	17B 5, 0	> 279 272 28 1 2 80	80.3 13.3 91	88 6 38 0	91 6 39 6 16 9	66.9	111 26.7 5.80
5 2×10 ⁻⁸ 3 0×10 ⁻⁷ 7 8×10 ⁻⁶	3 5×10 ^{-\$} 8.5×10 ^{-\$}	8 8×10 ⁻⁸ 2 1×10 ⁻⁷ 4 4×10 ⁻⁷	8 3×10 ⁻⁹ 4, 4×10 ⁻⁸	3 0×10 ⁻⁸ 3 1×10 ⁻⁸ 7 7×10 ⁻⁸ 1 4×10 ⁻⁶	6 8×10 ⁻⁸ 7 6×10 ⁻⁷ 1 1×10 ⁻⁵	1 4×10 ⁻⁸ 3 6×10 ⁻⁸	5 6×10 ⁻⁸ 1 8×10 ⁻⁷ 4 2×10 ⁻⁷	7 5×10 ⁻⁸ 3 3×10 ⁻⁷	1 7×10 ⁻⁷ 2 7×10 ⁻⁷ 8.5×10 ⁻⁷
172 241 414	241 310	172 207 241	241 276	241 414 483 552	172 241 414	172 241	172 197 241	172 241	207 241 276
	1	1		!	1 71]	
0.59	0.26	0.58	0 18	0 02		0. 22	0. 18	0.33	0.34
0.36	0. 44	0.47	0 45	0 53	0.56	0.63	0.64	0 64	99 '0
86	104	26	9 a,	73	75	86	102	101	103

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 $^{
m a}$ Where ${
m Zr}^{\,\prime}({
m Zr}$ + Hf) did not equal 2 to 4 percent, Mo contents were considered to be invalid.

TABLE VI - CREEP PROPERTIES OF SWAGED Mo-2Re-Hf-C

ALLOYS ST LOADED AT 1250° C

Estimated	stress for $10^{-7} \cdot \text{sec}$ creep rate, MN/m^2	Z 1	8	2≤ 3	9 02	21 5	27 9
2	rate, sec ⁻ 1	1 2×10-7 3 9×10-7 2 0×10-6 1 3×10-5	2 2×10 ⁻⁷ 5 8×10 ⁻⁷ 3 9×10 ⁻⁶ 2 3×10 ⁻⁵ 9 3×10 ⁻⁵	7 6×10 ⁻⁸ 4.8×10 ⁻⁷ 2 0×10 ⁻⁶ 1 1×10 ⁻⁵ 4.0×10 ⁻⁵	3 4×10 ⁻⁸ 3 0×10 ⁻⁷ 1 4×10 ⁻⁶ 6 8×10 ⁻⁶ 5 9×10 ⁻⁵	1 7×10 ⁻⁷ 3 0×10 ⁻⁷ 5 8×10 ⁻⁷ 1 1×10 ⁻⁶ 2 7×10 6	2 4×10 ⁻⁷ 6 7×10 ⁷ 2 6×10 ⁻⁶ 9 1×10 ⁻⁶ 2 6×10 ⁻⁵
Stress,		17 2 24 1 34 5 48 3	13 8 20.7 29.0 37 9 48 3	27 6 34 5 41 4 51 7 62 1	17 2 24 1 34 5 48 3 69 0	24. 1 27 6 33 1 37 9 45 5	31 0 41 4 51 7 62 1 72 4
ndition	Average grain diameter, μm	83	₩	1	41	32	39
Post test condition	Amount of recrystallization,	100	100	Lo	100	100	100
ပ	ess ite. %	1 1 1	-	1 I 1		- - - -	1
ΗĘ	Excess solute at %	Ø. 78	3	50	0 × 0	7× 0	25
	tent (after test), mol%	0 004	60 0	60. 0	60 0	0 15	0 28
Alloy		W	100	94	<i>1</i> .3	<u>ී</u> 6	W

					. '				
20.2	37 z	35 7	41 0	osi l	် ဂ ဗ	37.8	z 1 4	\$ 1	41 0
4 8×10 ⁻⁷ 1 4×10 ⁻⁶ 5 3×10 ⁻⁶ 2 3×10 ⁻⁵	2 8×10 ⁻⁷ 9 5×10 ⁻⁷ 3 1×10 ⁻⁶ 9 0×10 ⁻⁶	2 3×10 ⁻⁷ 8 6×10 ⁻⁷ 2 3×10 ⁻⁶	1 1×10-7	2 9×10 ⁻⁷ 1 1×10 ⁻⁶ 2 5×10 ⁻⁶ 5 1×10 ⁻⁶ 1 0×10 ⁻⁵	23× 10 ⁻⁶ 47× 10 ⁻⁶ 74× 10 ⁻⁶ 15× 10 ⁻⁵	1 4×10 ⁻⁷	8 4×10 ⁻⁸ 7 2×10 ⁻⁷ 3 3×10 ⁻⁶	1 5×10 ^{-\$} 6. 5×10 ^{-\$} 1 3×10 ⁻ B	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$
26.2 33.1 41.4 52.4	44.8 55.2 69.0 82.7	41 4 51 7 62 1	41 4	13 8 20 7 28 3 37 9 48 3	17 2 24.1 31 0 41 4	41 4	20 7 34.5 48 3	24.1 34.5 41.4	42 7 55 2 62 1
23	51		24	:	13	22	!	25	1
100	100	0z	100	1	100	100		100	1
	t	t 1	!		1 24	0.25			i i i
0.56	м Ф	• 2Z	0.11	0.46		-	60 60 60	0 20	0 35 35 35
0. 29	0 37	0 44	0.49	0.51	0.56	0.58	о м 1	0 %2	0.65
86	26	98	74	101	75	73	104	102	103

TABLE VII - CREEP PROPERTIES OF RECRYSTALLIZED

Mo-2Re-Hf-C ALLOYS STEP LOADED AT 1315⁰ C

[Heat-treatment time, 1hr.]

Alloy	Heat-treatment	Calculated	Hf	С	Average grain	Estimated	
	temperature,	HfC con-	Excess solute,		diameter in	stress for	
	°C	tent (after			unstressed	$10^{-7} - \sec^{-1}$	
		test),			Dutton-nead		creep rate
		mol%			at % after test,		MN/m^2
					$\mu { m m}$		
88	1760	0 11	0 65		54	74	
97	1980	14	91		39	95	
87	1760	15	74		56	77	
95	1930	18	64		32	86	
100		19	02		34	142	
94		19	40		26	113	
98	₩	34	51		23	90	
86	1870	34	61		27	93	
96	1930	42	24		21	103	
75		56		1 70	13	62	
73		57	01		34	110	
101	*	68	29		34	113	

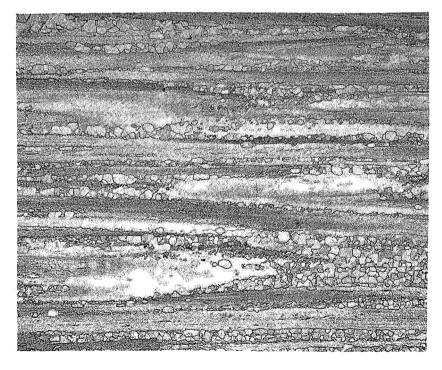


Figure 1 - Microstructure of as-extruded Mo-1 93Re-0.60Hf-0.49C alloy $\times 50$.



(a) Annealing temperature 1370°C

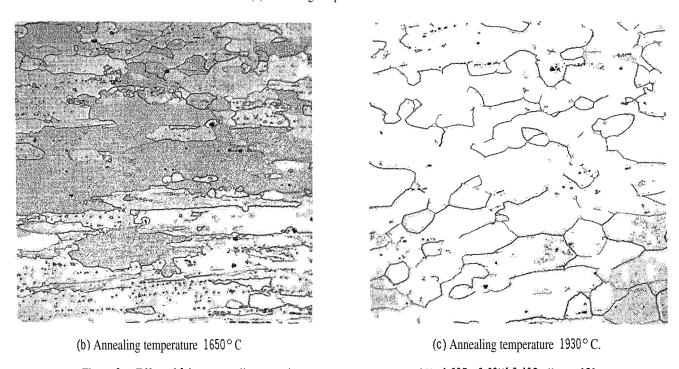


Figure 2 Effect of 1-hour annealing at various temperatures on swaged Mo-1 93Re-0.60Hf-0.49C alloy ×250

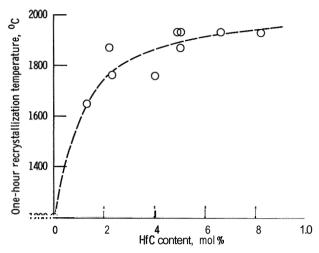
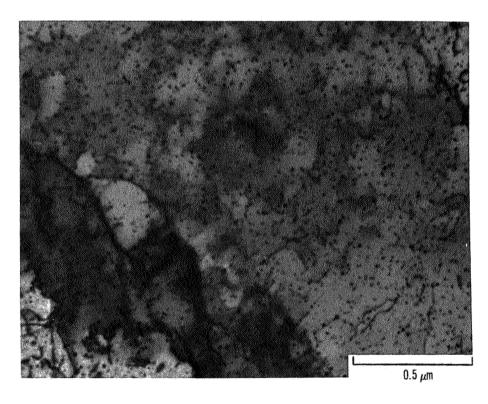
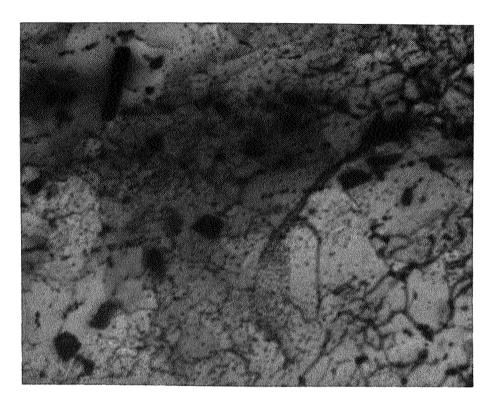


Figure 3. Temperature for 100-percent recrystallization in 1 hour as function of HfC content in Mo-2Re-Hf-C alloys containing excess IE

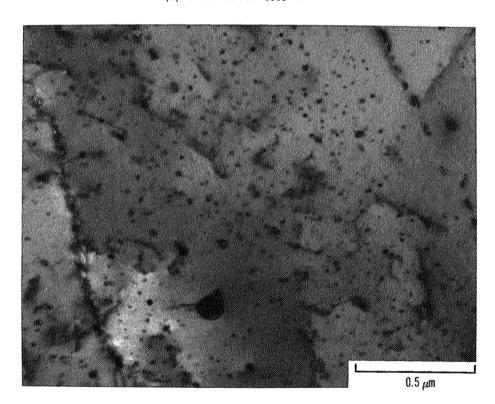


(a) As swaged.

Figure 4 Transmission electron micrographs of swaged Mo-1 93Re-0.60Hf-0 49C alloy before and after testing



(b) Tensile tested at 1315°C

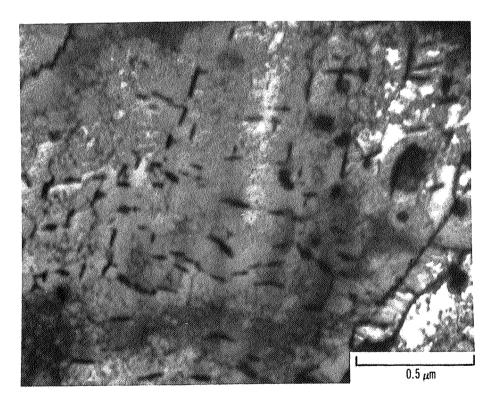


(c) Creep rupture tested at 1315°C and 241 MN $\rm m^2$; life 72 4 hours

Figure 4 Concluded

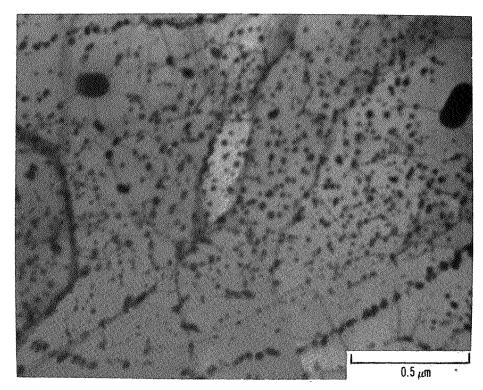


(a) As swaged



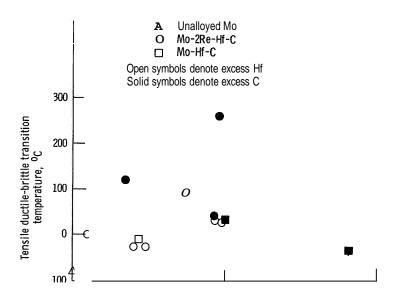
(b) Tensile tested at 1315° C

Figure 5 Transmission electron micrographs of swaped Mo-1 97Re-0 58Hf-0 70C alloy before and after testing



(c) Tensile tested at 1650° C

Figure 5 Concluded



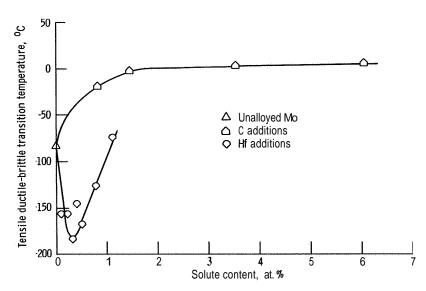


Figure 7 - Tensile ductility as function ${\bf d}$ solute content of swaged Mo rod at 5 percent reduction in area.

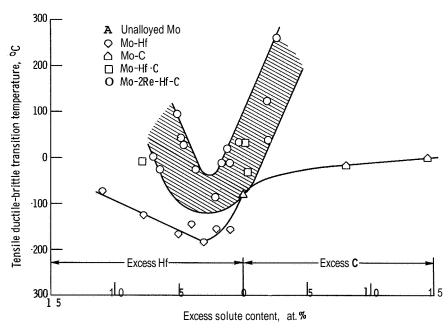
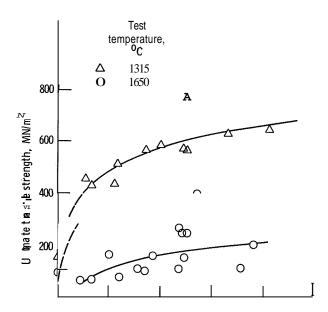
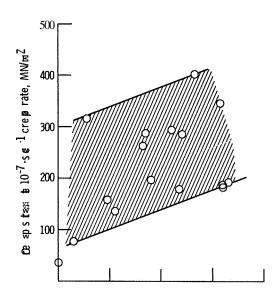


Figure 8. Tensile ductility as function of excess **H** and C content in swaged Mo alloy rod at 5 percent reduction in area.





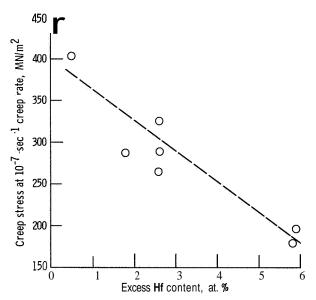


Figure 11 Creep strength at 1315⁰ C as function of excess Hf content of swaged Mo-2Re HE·C alloys con taining 0. 33· to 0.53-mol % HfC

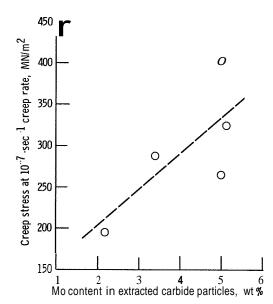


Figure 12. Creep strength at 1315^o C as function of Mo content in carbide particles extracted from swaged Mo-2Re-Hf-C alloys containing 0. 33- to 0. 53-mol % HfC,

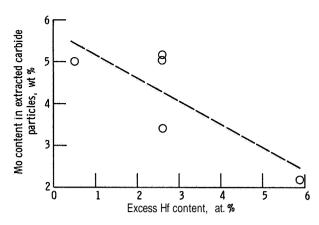
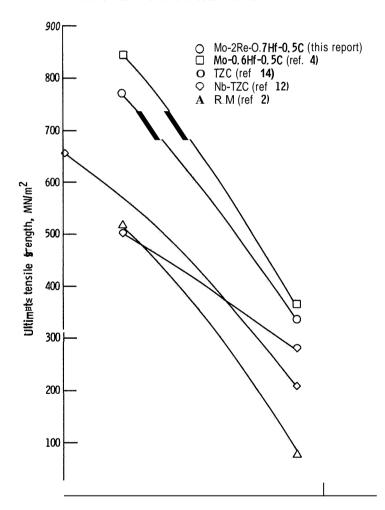


Figure 13. Molybdenum content of carbide particles extracted from swaged Mo-2Re-Hf-C creep specimens tested at 1315⁰ C and having 0.33- to 0.53-mol % HfC content as function of excess Hf content.



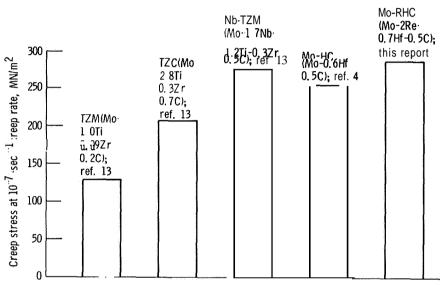


Figure 15 Comparison of 1315^0 C creep strength on **Mo Hf**·C and Mo-2Re·Hf C alloys with other in process solution treated **Mo** alloys. (All compositions are in at %)

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