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TITLE: SOLUTION SOFTENING IN MOLYBDENUM-RHENIUM ALLOYS

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SOLUTION SOFTENING IN Mo-Re ALLOYS

by

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S. M. Tuominen, and E. P. Whelan

I. INTRODUCTION

One of the major deterrents to the use of molybdenum in many applications is its tendency to be brittle in the recrystallized condition near room temperature and below, at typical engineering strain rates. However, considerable work¹⁻⁶ has indicated that good low temperature ductility can be achieved in molybdenum-rich Mo-Re alloys containing as little as 5 at.% rhenium. Rhenium in this region of composition produces "solution softening" in molybdenum alloys that is manifested by a reduction in the hardness² and flow stress⁶, relative to pure molybdenum, in addition to a reduction in the ductile-brittle transition temperature (DBTT). This phenomenon differs from that seen at higher rhenium contents in Mo-Re alloys where low temperature ductility is improved but the flow stress is increased.

As pointed out by several authors^{2,7,8}, solid solution softening is not unique to the Mo-Re system but is found generically when the Group VIA metals - Cr, Mo, W - are alloyed with small quantities of Group VIIA and VIIIA metals. The mechanisms that cause this phenomenon are not well understood, but two related mechanisms have been postulated. Stephens and Witzke² and Kurdyumova, et al.⁹ propose solution softening results from electron concentration effects, while other authors^{5,6,10} propose a reduction of the Peierls stress with increasing rhenium content.

This paper discusses the current state of knowledge of the behavior of Mo-Re alloys containing 2.5 to 9.6 at.% Re relative to the question of solution softening. Emphasis will be given to the results of a current, on-going systematic study of Mo-Re alloys in this composition range that is aimed toward establishing the range of rhenium content over which solution softening is effective and to better define the potential of these alloys for engineering applications. Results from this study that will be discussed include recrystallization behavior, mechanical behavior, and fracture behavior of eight vacuum arc cast (VAC) alloys prepared by AMAX Materials Research Center.

II. EXPERIMENTAL

A. Alloy Preparation

Alloy ingots were prepared by consumable vacuum arc melting electrodes produced from cold-pressed and sintered bars derived from mechanically blended powders. Carbon was used as the deoxidant. All process heating was performed in dry hydrogen. The chemical composition of the nine ingots that were prepared for this study is listed in Table I. As will be noted, two alloys near the 7.2 at.% level were prepared, one with high carbon and one with low carbon content comparable to the rest of the alloys in the series. All the alloys were also found to contain both nitrogen and silicon at levels around 0.02 at.%. Electron microprobe scans of cast structures indicated very uniform rhenium concentration across grains demonstrating that coring during alloy solidification was not a problem.

The cast ingots were hot upset into the extrusion liner and extruded to rectangular bar stock after preheating to temperatures up to 1775 K. The bars were recrystallized and then rolled to 73% reduction in thickness, following a 1225 K preheat and stress relief.

B. Alloy Behavior

The alloy plates were evaluated by determining the temperatures for complete recrystallization in one hour, determining the room temperature hardness, determining tensile mechanical behavior from 77 to 295 K, measuring elastic wave velocities at room temperature, measuring lattice constants, and performing Auger electron analysis of fracture surfaces prepared under ultra-high vacuum.

1. Recrystallization Behavior

Stress relieved coupons of cold rolled plates of each alloy were annealed for one hour in dry hydrogen at temperatures ranging from 1073 to 1873 K at 100 K intervals. Metallographically prepared coupons were attack polished in aqueous $K_3Fe(CN)_6$ and etched in an aqueous solution containing 2.08% $K_3Fe(CN)_6$ and 0.65% NaOH. The volume fraction of recrystallized material was determined by manual point counting. The temperatures at which 100% recrystallization was achieved as a result of a 1 h anneal are listed in Table II. The grain size of each of the alloys was found to be in the range ASTM No. 6 to 7 after 1 h at 1673 or 1773 K. Typical microstructures of the 7.2 and 7.4 at.% Re

alloys can be seen in the photomicrographs in Fig. 1. The angular precipitates seen in both microstructures have been determined, using electron microprobe techniques, to contain Mo, Re and C. The spherical inclusions were determined to contain mostly O, La, and Ce along with minor amounts of Cu, Fe, Ca, Si and Al. Because only Fe, Si and Ca were ever detected in the alloys by chemical analysis, it is suspected that the rest of the elements came from the final polishing compound.

2. Mechanical Behavior

Room temperature hardness, HV10, was determined as a function of annealing temperature for each alloy, and the data are plotted in Fig. 2. The hardness values for the recrystallized samples and the stress-relieved, cold-rolled materials are plotted as a function of rhenium content in Fig. 3. This plot indicates that a hardness minimum occurs at approximately 3 at.% Re.

Tensile tests of both stress relieved and recrystallized materials were conducted at 77, 197, and 295 K at a strain rate of 0.001 s^{-1} using specimens with a gauge section of 3 mm by 4.7 mm and gauge length of 25.4 mm. The specimens also had a central reduced gauge section measuring 2.85 by 4.55 mm by 8 mm long. An extensometer with the capability of measuring strains up to 15% and a gauge length of 7.6 mm was attached to each specimen during testing. Total elongation beyond 15% was measured by fitting the specimen back together and measuring the distance between two hardness indentations that had been placed 25.4 mm apart on the gauge section prior to testing.

The tensile properties for the recrystallized materials at 295 K are presented graphically in Fig. 4. The tensile elongation and reduction of area at 295K are shown in Fig. 5. The alloy series exhibits a maximum in fracture stress and ductility, as measured by both elongation and reduction of area, and a minimum in 0.2% yield strength (0.2 YS) in the range 4.6 to 7.4 at.% Re. All of the recrystallized Mo-Re alloys exhibit an upper/lower yield point phenomenon at this temperature that is illustrated in Fig. 6. The magnitude of the effect does not appear to vary in any systematic way with either rhenium or carbon content in the alloys.

No yield point phenomenon is observed for the cold-rolled, stress-relieved alloys at room temperature. Both the yield and ultimate strengths of the alloys in this metallurgical condition were observed to increase steadily with

rhenium content. The ductility of the alloys in the stress-relieved condition showed a slight tendency to peak around 7 at.% Re.

The tensile mechanical properties measured at 197 K for recrystallized alloys are listed in Table III. These data indicate that ductile behavior is not observed at this temperatures in alloys with less than 5.8 at.% Re. All indicators of ductility tend to exhibit peaks at both 5.8 and 8.2 at.% Re.

3. Lattice Constants

The lattice constants of all nine materials were measured from unannealed filings using standard x-ray diffraction techniques. Only diffraction lines from the bcc solid solution were observed. The lattice constants which are listed in Table IV were computed from measured d-spacings using a computer program based on the computational methods described by Vogel and Kemper¹¹. These data compare closely with earlier data¹² that indicated a linear relation between lattice constant and rhenium content in solid solution Mo-Re alloys.

4. Elastic Wave Velocities

The longitudinal or compression wave velocity (V_p) and the shear wave velocity (V_s) were determined at room temperature for eight different alloy plates. The longitudinal and shear waves were generated by a 250V pulsed, 10 MHz, X-cut quartz transducer and 5 MHz, Y-cut quartz transducer, respectively. The initial stress pulse is transmitted through a 3-mm-thick aluminum plate and into a similar quartz transducer whose output is amplified and displayed on an oscilloscope. The sample is sandwiched with the aluminum plate between the two transducers, and the additional signal delay due to the sample is measured using the scope delayed time base and a time-interval meter. Light machine oil is used for the longitudinal coupling medium and Dow Resin 276-VR is the shear coupling medium.

From the measured elastic wave velocities and assuming the alloys are elastically isotropic and homogeneous in density (ρ), the elastic moduli can be determined. The shear modulus, $G = \rho \cdot V_s^2$, the bulk modulus, $K = \rho \cdot (V_p^2 - 4/3 V_s^2)$, and the Young's modulus, $E = 3K / [(V_p/V_s)^2 - 1]$. The Young's shear and bulk moduli of the alloys at 295 K are listed in Table V.

5. Fracture Behavior

Fracture surfaces of vacuum annealed, rolled plate specimens of the eight alloys along with the companion pure molybdenum and six as-cast Mo-7.2Re specimens were examined using Auger electron analysis techniques. The vacuum annealing was performed for 1 h at 1650 K at about 10^{-7} Torr. All specimens were fractured by bending, in a vacuum of about 10^{-10} Torr, and the analysis was begun immediately before surface contamination could develop to a significant degree. All of the alloy specimens were difficult to break at room temperature, and the specimens containing 5.8, 7.4 (low carbon), and 8.2 at.% rhenium had to be cooled down to near liquid nitrogen temperature to break them.

The fracture surfaces of all the rolled-plate specimens were very clean, and the fractures tended to be mostly transgranular. Carbon and oxygen were the only impurities detected on these fracture surfaces, and as the rhenium content increased, the levels of these impurities decreased on the fracture surfaces. This is illustrated by comparing a typical Auger survey spectra taken from the fracture surface of the pure molybdenum presented in Fig. 7 with a typical spectra from the surface of the Mo-7.2 at.% Re (high C) specimen in Fig. 8. The Auger survey spectra fracture surfaces of the Mo-8.2 at.% Re and Mo-9.6 at.% Re specimens were very similar to that for the Mo-7.2 at.% Re+C seen in Fig. 8.

All of the as-cast Mo-7.2 at.% Re specimens broke at room temperature in a predominantly intergranular mode. The Auger survey spectra from the fracture surfaces of all the samples indicated the presence of significant levels of carbon, nitrogen, and oxygen. As can be seen in the Auger electron dot maps in Fig. 9, the oxygen seemed to be concentrated in particles or bubbles on the fracture surface, while the carbon and nitrogen seemed to be more uniformly distributed. In addition to carbon, nitrogen and oxygen, calcium was found on the fracture surface of one specimen and sulfur on another.

An Auger depth profile was performed on the sample that contained calcium in an area of high oxygen contamination using 2kV argon ions to sputter away the surface, and all impurities except carbon were removed by this process at a depth of about 15 nm. Another curious observation on the fracture surfaces of all of these cast specimens was the low to nonexistent rhenium concentrations which could be brought up to bulk levels after sputtering to depths of

the order of 100 nm. This is in contrast to the wrought alloy specimens for which the Auger electron analyses tended to indicate higher than nominal rhenium contents on their fracture surfaces.

III. DISCUSSION

The room temperature mechanical properties of the 73% rolled, stress relieved VAC alloys are comparable to those reported by Klopp and Witzke¹ for as-rolled plates and as-swaged bars, with comparable rhenium contents and impurity levels, that were originally prepared by electron beam melting (EBM). This is also true for recrystallized VAC Mo-Re alloys. The VAC alloys have slightly higher ultimate and yield strengths and slightly lower ductilities than the EB-melted alloys. The room temperature tensile strengths reported for as-drawn Mo-5.4 at.% Re wire⁵ are almost twice that of the previously mentioned products. One of the more interesting aspects of the mechanical behavior of these alloys in general is that recrystallized samples have better ductility than those that have only been stress-relieved after working which is opposite behavior from that observed for pure molybdenum and most Mo-base alloys.

The ductility peak that was predicted by Lundberg³ to occur around 7 at.% Re for recrystallized alloys at room temperature is confirmed in Fig. 5 except that the observed ductility rise with rhenium content is more gradual than previously reported. The abrupt drop in room temperature ductility around 8 at.% was also predicted³. This narrow ductility peak as a function of rhenium content might explain why solution softening in Mo-Re was generally overlooked in previous studies^{1,14}. The ductility peak seems to shift to higher rhenium content as the test temperature is reduced which is similar to the hardness minimum shift reported previously.² This would suggest that the lowest DBTT in this composition range would be obtained for the 8.2 at.% Re alloy. The hardness minimum seen in Fig. 2 for recrystallized alloys occurs at a lower rhenium content than does the ductility maximum seen in Fig. 5.

The addition of carbon to the 7.2 at.% Re alloy appears to have made it slightly more ductile. At room temperature the high carbon alloy had almost twice the RA of the low carbon 7.4 at.% Re alloy, while at 155 K it showed some ductility as opposed to none for the low carbon alloy.

The yield point phenomena displayed by the recrystallized alloys in this study (See Fig. 6) has been previously seen in both single¹⁵ and polycrystalline⁴ alloys in this composition range. Lawley and Maddin¹⁵ suggest that this behavior is due to dislocation locking by interstitial impurities. The effect of carbon on the yield point phenomenon in the alloys in this study was not clear as there was little difference in the behavior of the comparable high and low carbon alloys.

Reported recrystallization temperatures of the comparable EBM alloys¹ are somewhat higher than were found in this study. The reported 1 hour recrystallization temperature for 81% cold rolled EBM Mo-3.9 at.% Re plate is 1590 K as compared to 1473 K for Mo-3.7 at.% Re in this study. The reported 1 hour recrystallization temperature for EBM Mo-5.9 and 7.7 at.% Re alloys, swaged 50%, is 1700 K, as compared to 1573 for 7.4 at.% Re and 1673 K for 8.2 at.% Re in this study.

The lattice constants were measured to confirm the linear relationship with rhenium content that had previously been proposed based on much more widely spaced rhenium concentrations¹². Our data do not indicate any anomalies in the average atomic spacing nor previously undetected secondary phases that might explain the observed solution softening.

The discontinuities in the elastic moduli centering around 6 at.% Re are a further manifestation of solid solution softening in this alloy system. This behavior is an indication of the intrinsic nature of this phenomenon, and it is consistent with at least one theory of solid solution softening.¹⁷ The texturing in the plate specimens may have influenced the absolute values of the moduli, but it is highly unlikely that the relative changes with composition are strongly influenced by this effect. It is interesting to note the good agreement between the data obtained from these polycrystalline specimens and that previously reported by Davidson and Brotzen¹³ for single crystals. The reduction in the bulk modulus, especially, in the 6 to 7 at.% Re range is believed to be an indication of a change in the bond character in this composition range.

The fracture studies on the worked alloys indicated that rhenium additions to molybdenum causes the role of interstitials in the fracture process to be reduced as the rhenium content increases. In contrast, the role of the interstitial elements precipitated out of the melt during solidification appeared to be predominant in the fracture of as-cast materials. Our observations of

impurity concentration on grain boundaries are very similar to those of Mil'man, et al.¹⁶ in a molybdenum alloy containing 0.004 at.% C, 0.000^{at.3%} N, 0.02 at.% O and 0.5 at.% Ta. The increased rhenium content on the fracture surfaces of the mechanically worked specimens might indicate a micro-ordering like that proposed by Arsenault¹⁷ as a prerequisite for small quantities of solute to cause a reduction in the Peirels stress and a resultant solid solution softening.

IV. CONCLUSIONS

This study has clearly demonstrated the solution softening phenomenon in Mo-Re alloys that contain 5.8 to 8.2 at.% Re. The intrinsic nature of the phenomenon tends to be supported by this work; however, the addition of rhenium to molybdenum appears to cause a redistribution of interstitials that affects the fracture behavior in worked alloys. Much more fundamental research is needed to clearly establish the mechanisms that cause this phenomenon.

The fabrication of these alloys seems to be no more difficult than any molybdenum alloy produced commercially today. Even the control of rhenium content in the maximum solution softening range does not appear to be difficult. More research will be required to obtain a better picture of the effects of interstitials of both fabricability and properties.

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TABLE I.
Alloy Compositions

at. %			wt. %		
Re	C	O	Re	C	O
0	0.014	0.007	0	0.0017	0.0011
2.5	0.018	0.012	4.7	0.0022	0.0020
3.7	0.006	0.011	6.9	0.0007	0.0018
4.6	0.021	0.009	8.5	0.0025	0.0015
5.8	0.027	0.010	10.7	0.0032	0.0016
7.2	0.062	0.004	13.1	0.0073	0.0006
7.4	0.020	0.006	13.4	0.0023	0.0010
8.2	0.011	0.015	14.8	0.0013	0.0024
9.6	0.014	0.022	17.2	0.0016	0.0034

TABLE II.
Recrystallization Temperatures for 3.2 mm Thick,
73% Cold-Rolled Sheet

Re Content (at.%)	Temperature for 100% Recrystallization in $^{\circ}$ C (K)
0	1373
2.5	1473
3.7	1473
4.6	1573
5.8	1573
7.2+0.06C	1573
7.4	1573
8.2	1673
9.6	1773

Table III.
Low Temperature Tensile Properties of Recrystallized Alloys

Temperature K	Re Content (at.%)	0.2% YS (MPa)	Upper Yield Strength (MPa)	UTS (MPa)
197	0	-*	-	558
	2.5	-	-	531
	3.7	-	-	434
	4.6	-	-	572
	5.8	503	545	600
	7.2+.06C	490	579	524
	7.4	490	552	517
	8.2	510	545	662
	9.6	490	531	531
155	0	-	-	635
	2.5	-	-	730
	3.7	-	-	635
	4.6	-	-	710
	5.8	-	-	745
	7.2+.06C	640	685	630
	7.4	-	-	650
	8.2	575	630	620
	9.6	-	-	665

*Dash indicates brittle failure, no significant yielding.

Table. IV.
Room Temperature Lattice Constants of Mo-Re Alloys

Re Content (at.%)	Lattice Constant (nm)
0	0.31475(3)
2.5	0.31459(3)
3.7	0.31460(5)
4.6	0.31445(2)
5.8	0.31444(5)
7.2+0.06 C	0.31437(3)
7.4	0.31438(5)
8.2	0.31429(9)
9.6	0.31425(4)

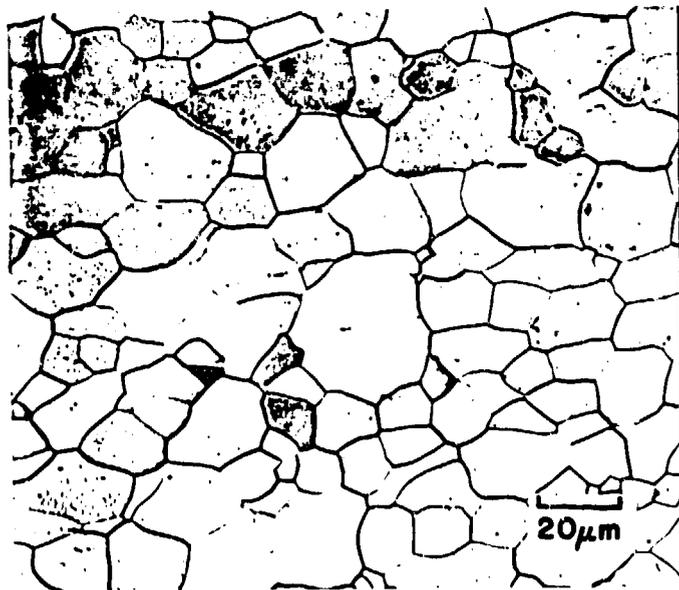
Note: Numbers in parentheses are standard deviation values for the last digit.

TABLE V

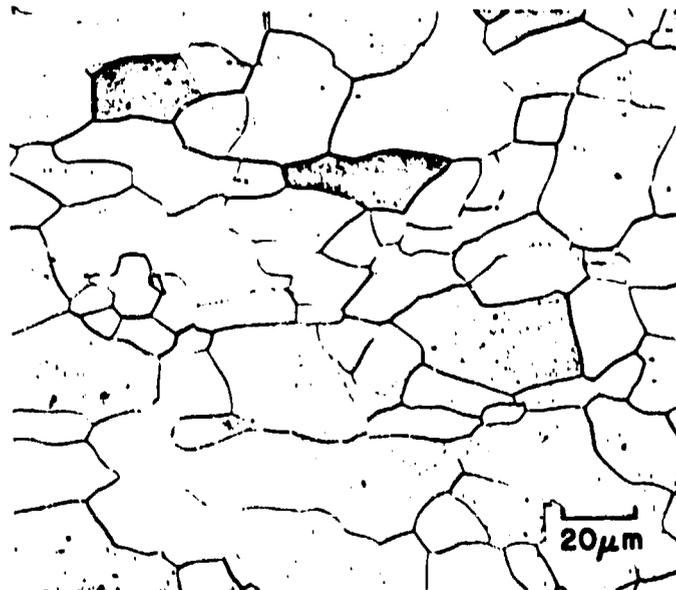
Elastic Moduli of Molybdenum-Rhenium Alloys

At. % Re	Density g/cc	Modulus, GPa		
		Bulk	Young's	Shear
0	10.222	241	289	111
2.5	10.495	260	304	116
3.7	10.611	285	277	103
4.5	10.725	291	285	106
5.8	10.843	271	324	123
7.2	10.959	276	319	122
7.4	10.979	281	315	120
8.2	11.084	297	299	113
9.6	11.211	273	328	126
0*		265	323	124
7.0*		271	329	128
16.6*		279	337	130
26.9*		284	345	133

*Data for single crystals from Ref. 13. Isotropic Young's modulus and shear modulus computed from elastic constants of single crystals by Hill's method.



(a)



(b)

Figure 1 - Microstructure of Recrystallized Mo-Re Alloys.
(a) Mo-7.2Re-0.06C, (b) Mo-7.4Re-0.02C

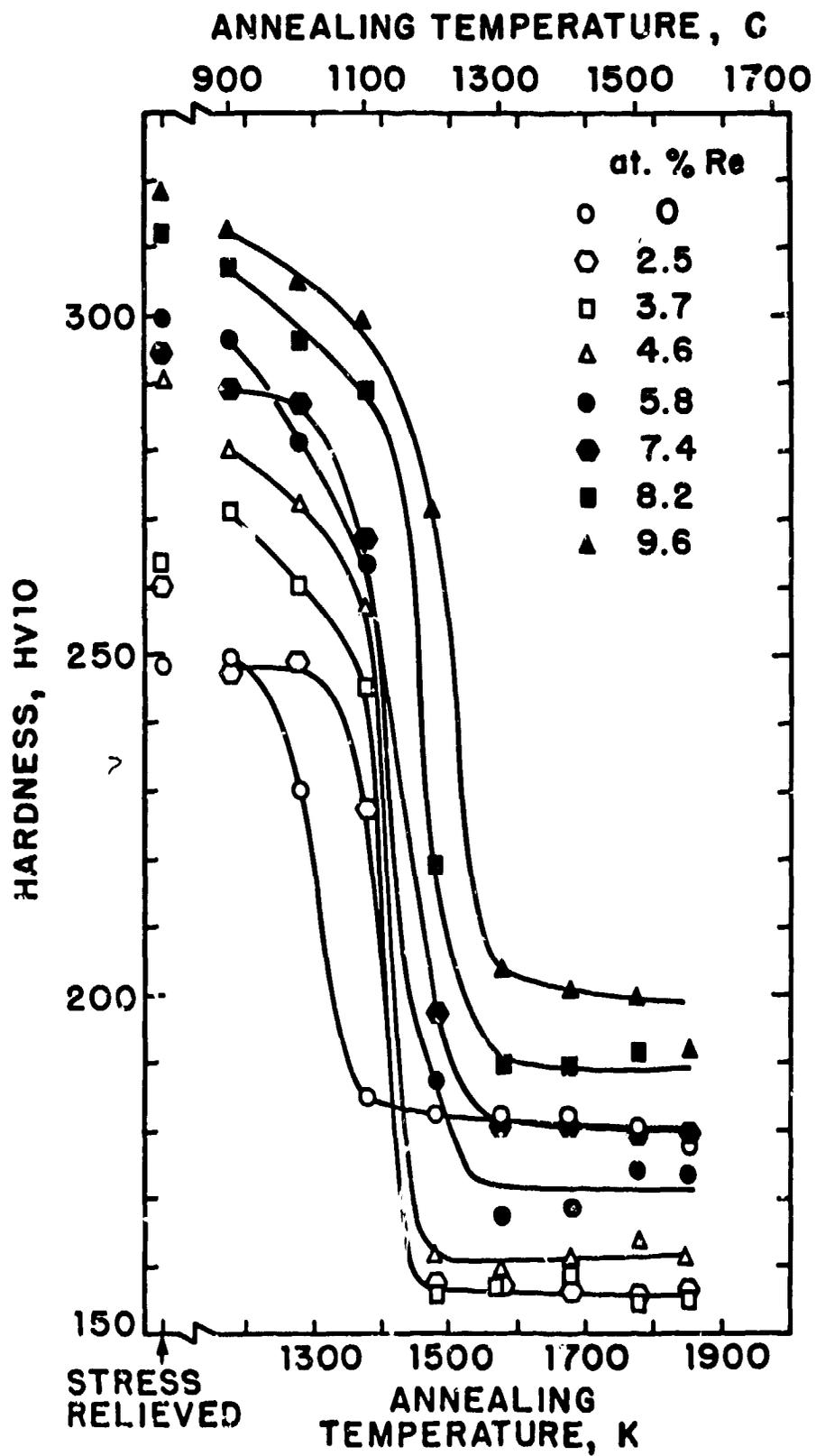


Figure 2 - Dependence of Room Temperature Hardness on Annealing Temperature of Molybdenum-Rhenium Alloys.

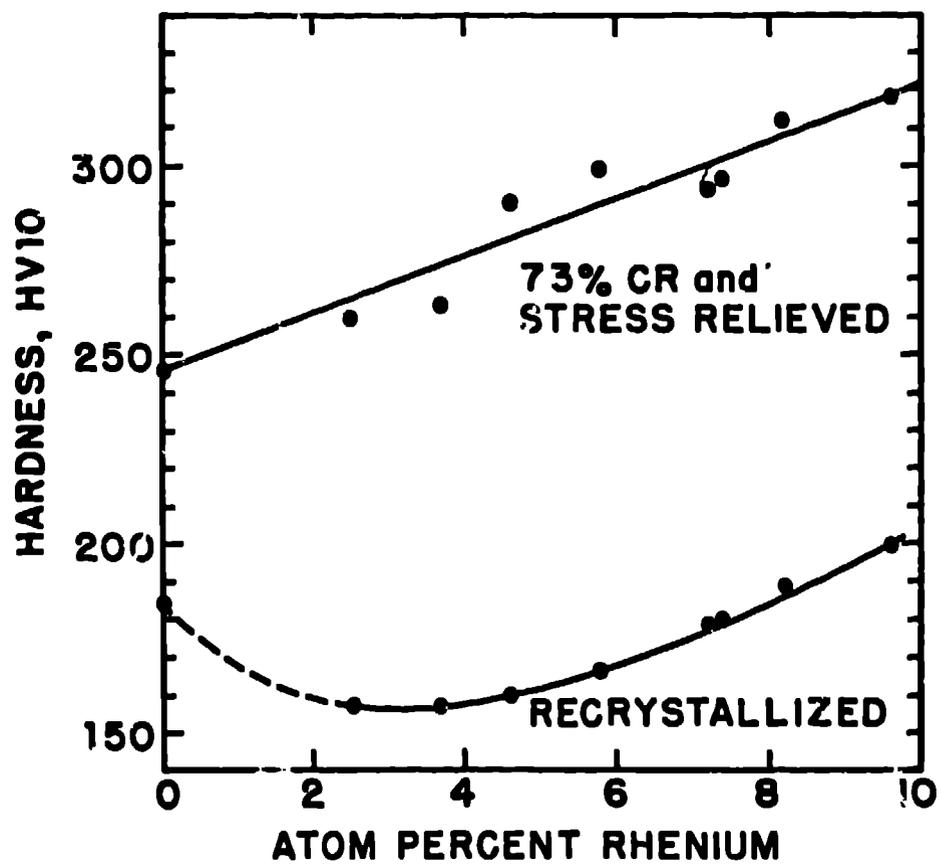


Figure 3 - Room Temperature Hardness as a Function of Rhenium Content of Molybdenum-Rhenium Alloys.

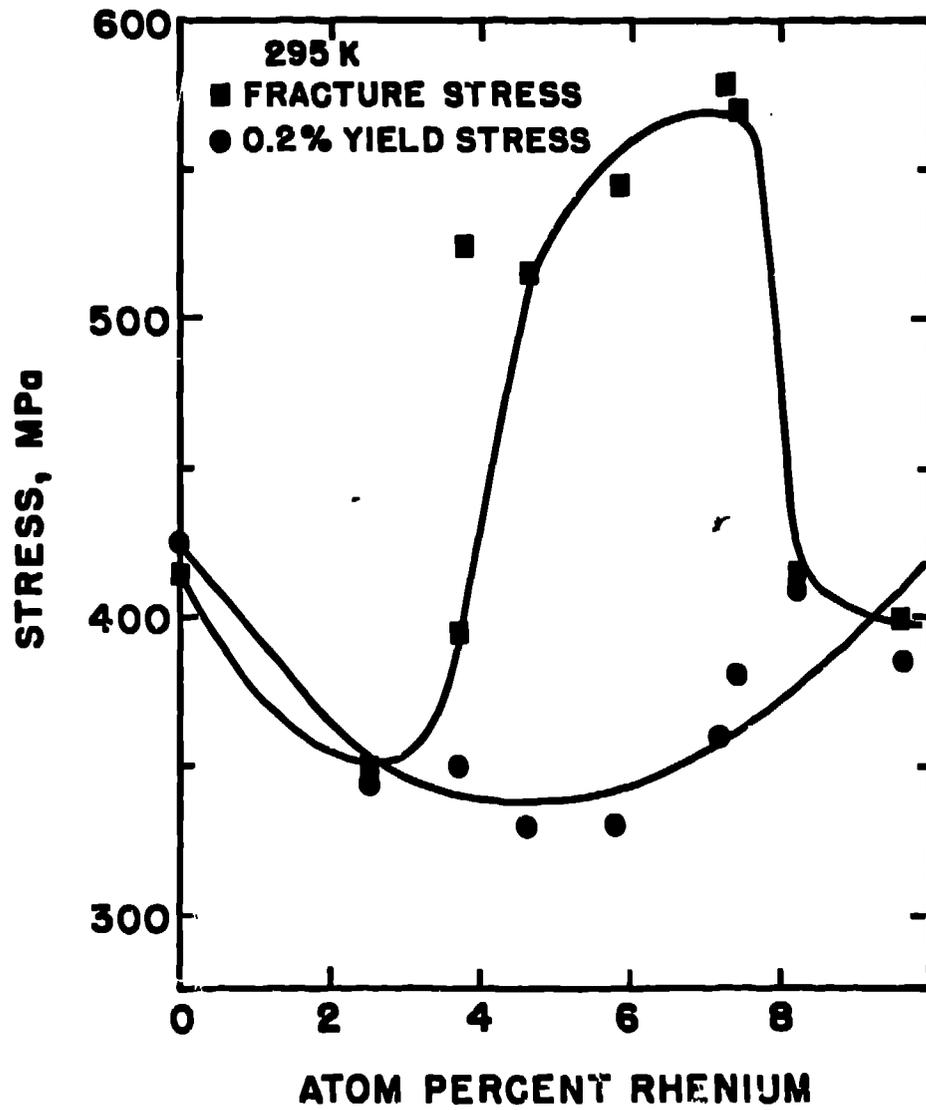


Figure 4 - Room Temperature Tensile Stress and Yield Stress of Molybdenum-Rhenium Alloys. Recrystallized.

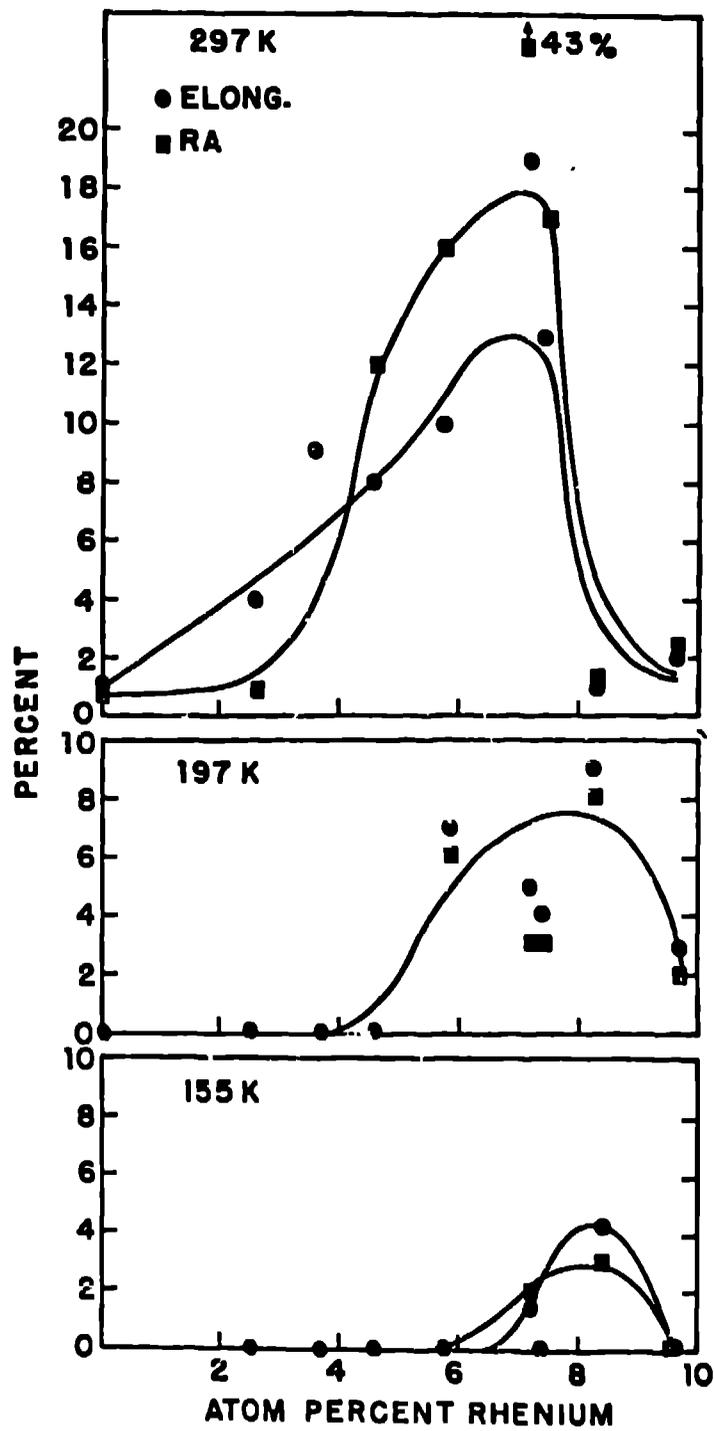


Figure 5 - Tensile Elongation and Reduction of Area of Recrystallized Molybdenum-Rhenium Alloys at Three Test Temperatures.

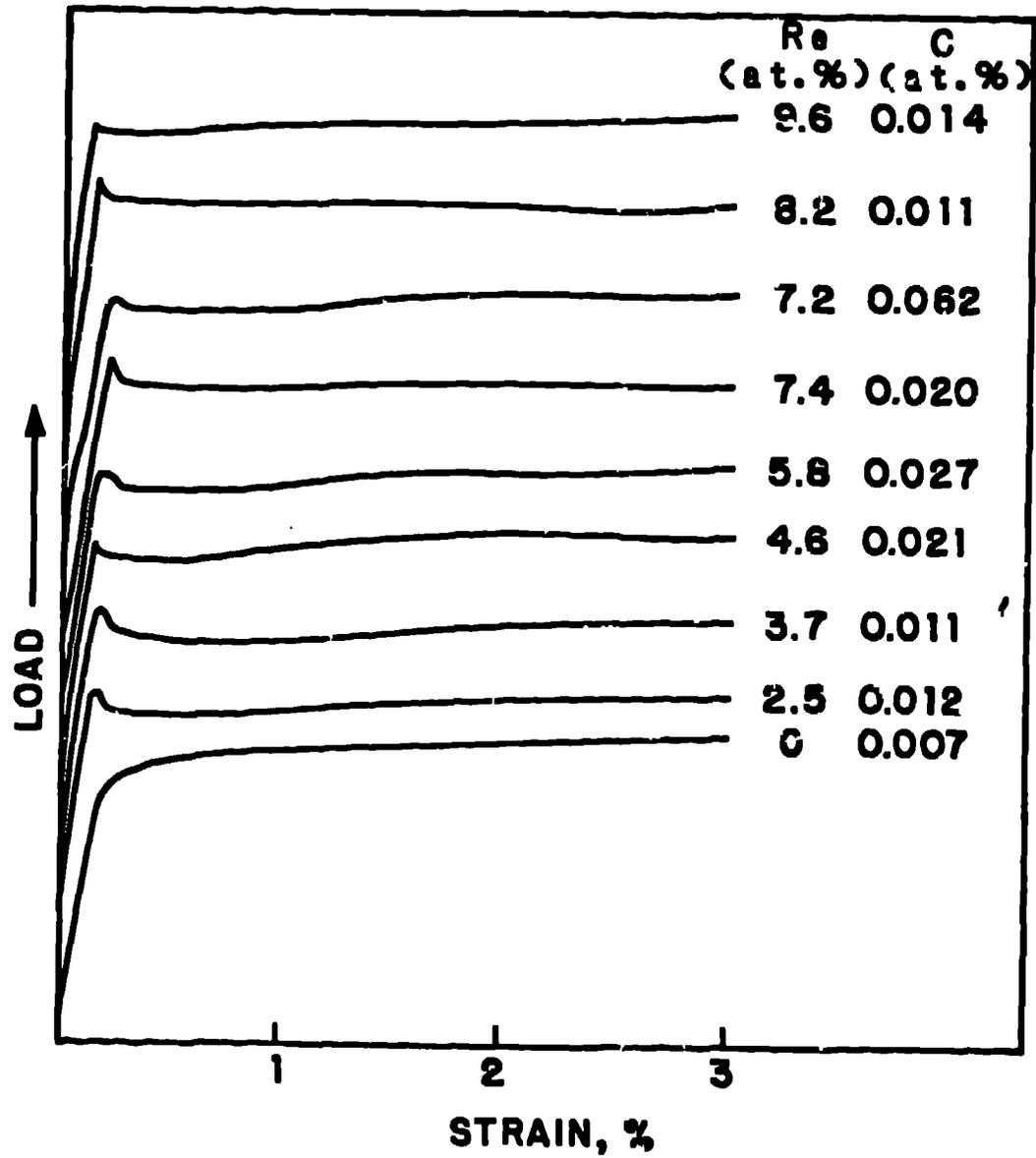


Figure 6 - Tensile Test Curves of Recrystallized Alloys at 295 K.

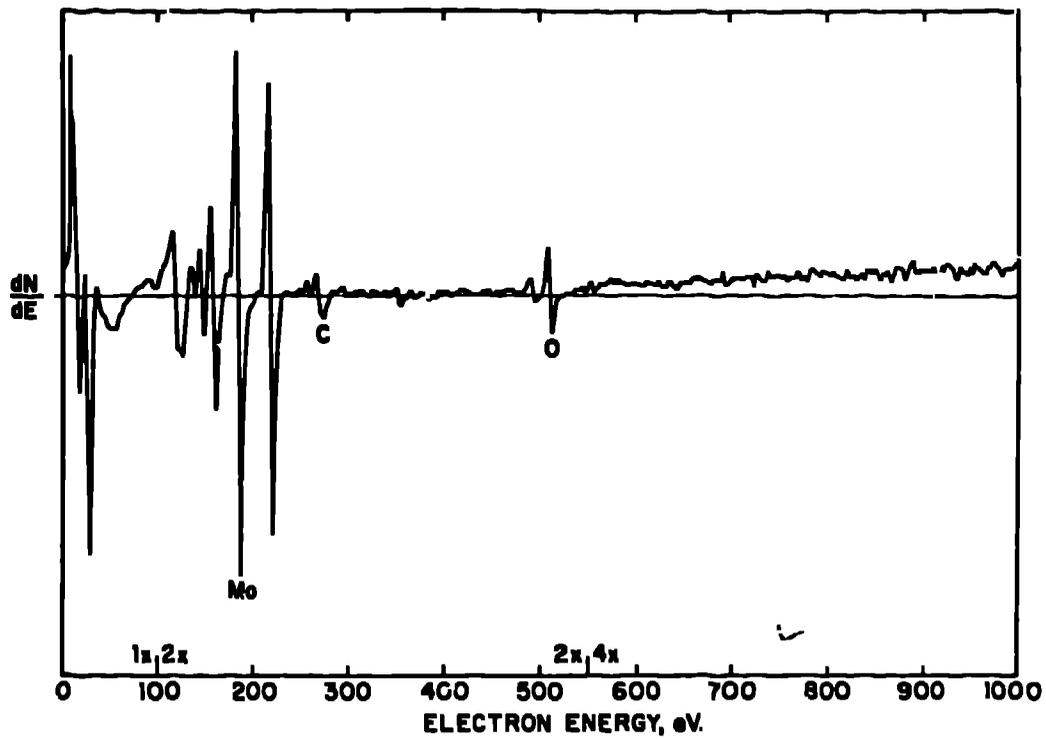


Figure 7 - Auger Spectra of Recrystallized Mo Fracture Surface

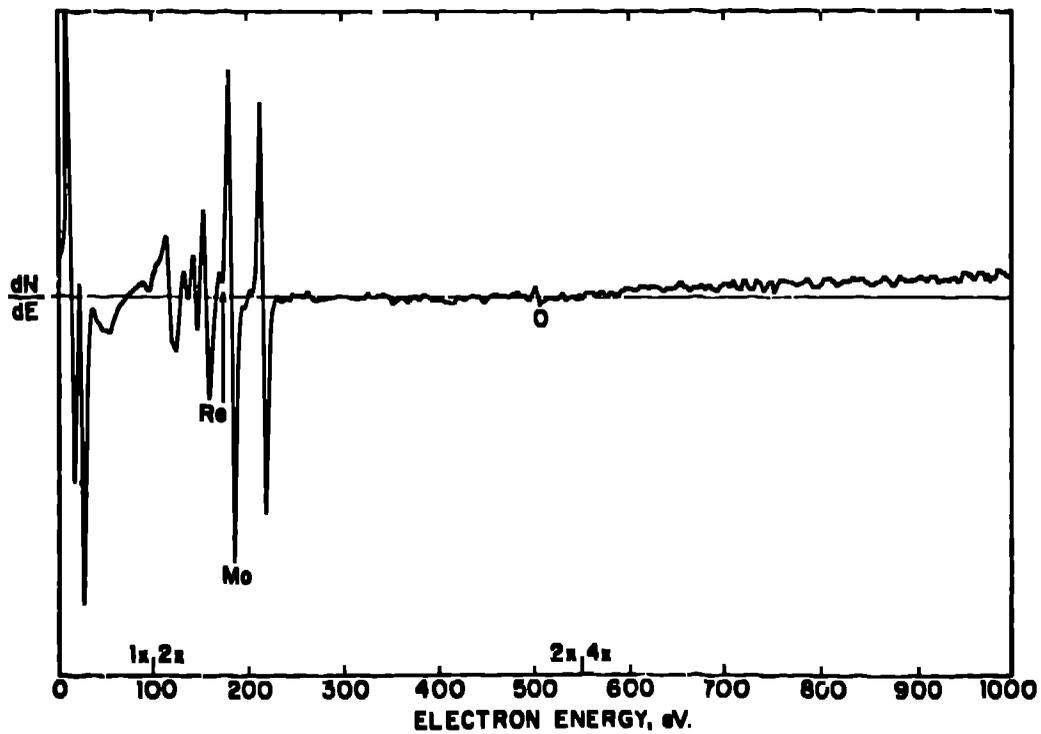
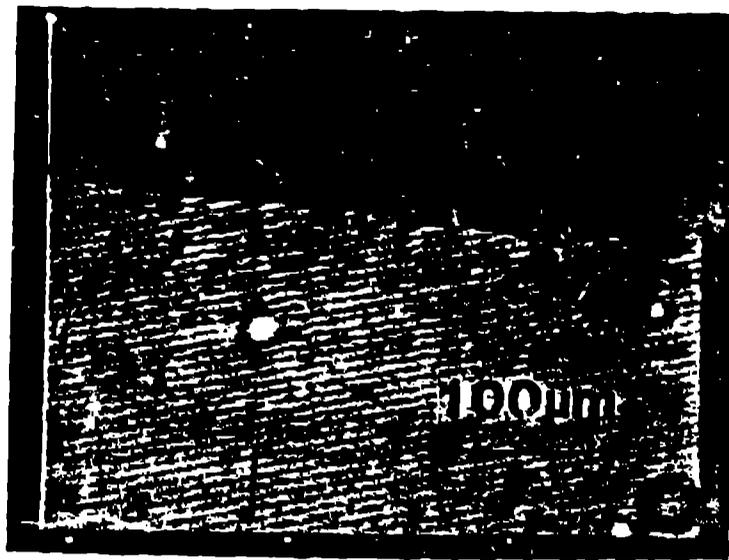
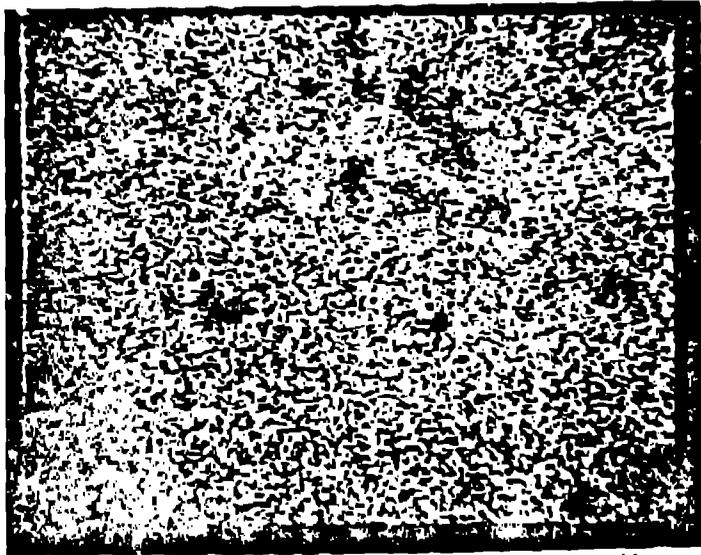


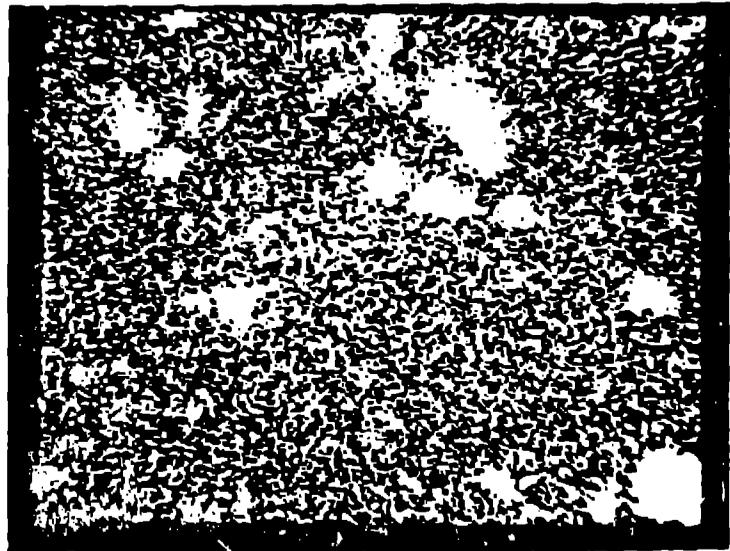
Figure 8 - Auger Spectra of Recrystallized Mo-7.2 at.% Re-0.062 at.% C Fracture Surface.



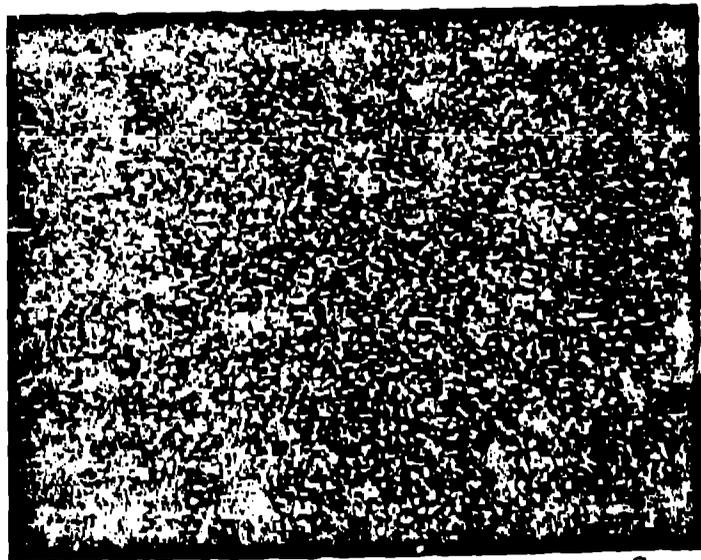
absorbed current image



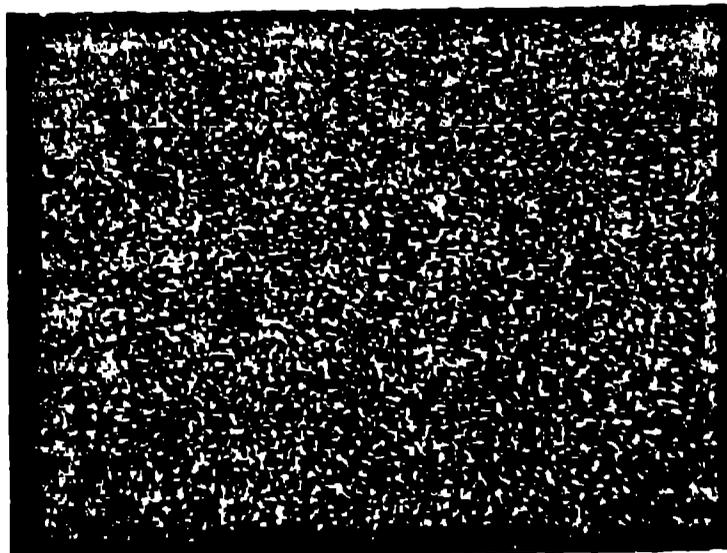
Mo



O



C



N

Figure 9 - Absorbed Current Image and Auger Electron Dot Maps Obtained from the Fracture Surface of Mo-7.2 at.% Re Alloy.