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TITLE DEVELOPMENT OF A MOLYBDENUM-RHENIUM ALLOY FOR SPACE NUCLEAR REACTORS

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DEVELOPMENT OF A MOLYBDENUM-RHENIUM ALLOY FOR SPACE NUCLEAR REACTORS

Lynn B. Lundberg

1. The study of Mo-Re alloys was prompted by the revelation that earlier DBTT data indicated no major reduction in the transition temperature above about 10 wt% Re.

2. Solution softening is the mechanism that drives the reduction in DBTT. It also causes a reduction in flow stress and elastic modulus. Solution softening occurs in Group VIA metals and alloys that contain small quantities of VIIA or VIIIA metals, and it is manifested at temperatures < 0.16 T_m. It is an intrinsic effect rather than either a purity or interstitial extrinsic effect. It appears to be caused by an electronic effect.

3. The Mo-Re equilibrium phase diagram does not give any indication of solution softening as previously shown for Mo-Re in the 10-15 wt% range.

4. The Mo-Re alloy development efforts supported by Los Alamos have included: (1) studies on vacuum arc cast (VAC) alloys at the AMAX Materials Research Center, (2) production of alloy VAC products for irradiation experiments, (3) creep studies on VAC sheet, and (4) studies on powder metallurgically produced alloys.

5. The Mo-Re development effort at AMAX Materials Research Center (ARML) included the production of light alloy compositions in the form of approximately 35 lb. VAC ingots with Re contents from 5 to 17 wt% at a 2% interval. A second Mo-13 Re ingot with carbon added to about 75 wppm. All eight alloy ingots were hot extruded into rectangles, warm-rolled and finally cold-rolled into plate from which samples were machined. These specimens were evaluated by measuring their room temperature hardness, determining recrystallization temperatures, measuring low temperature tensile mechanical behavior, measuring dynamic elastic moduli, determining gas-tungsten arc (GTA) weldability, and measuring the coefficients of thermal expansion from room temperature to 1375 K.

6. The VAC Mo-Re alloys were produced by first blending the metal powders in the desired proportions and then cold-pressing and sintering the powder blend into a bar to be used in the consumable electrode vacuum arc melter. Alloy ingots about 3-1/2 inches diameter by about 18 inches long were produced in the vacuum arc melter at AMRC. These electrodes were extruded to rectangles at AMRC using an area reduction ratio of about 5.1.

7. The microstructures of Mo-7.2 (+0.06 C) and 7.4 (+0.03 C) at.% Re VAC alloys were comparable and free of second phases. Even at the higher carbon level, no carbide precipitates were found.

8. As expected from the previous work by Joe Stephens and others at NASA Lewis, the ductility of Mo-Re alloys passes through maximum at about 11 wt Re at room temperature. However, as the temperature was reduced, the ductility peak shifts to higher Re contents. The high carbon alloys had much higher ductility than the low carbon alloys.

9. The variation of room temperature yield strength with Re content is also indicative of the solution softening phenomenon in Mo-Re alloys. The ultimate strength shows a general increase as the rhenium content is increased beyond about 5 wt%.

10. The thermal expansion behavior was measured for all the VAC alloys produced by AMRC. The variation of the coefficient of thermal expansion (CTE) with rhenium content does not agree with Russian data at 873 K.

11. The work at AMRC indicated an optimum Re content of 12 ± 1 wt%, maximum carbon content of 100 wppm, maximum nitrogen content of 20 wppm, maximum oxygen content of 30 wppm, and maximum hydrogen content of 5 wppm.

12. A good variety of Mo-11 to 13 wt% Re products were made for irradiation experiments. These products included Mo-13 Re plate sheet and tubing primary for the study of the fast neutron irradiation behavior of the alloy and Mo-11 Re tubing for fuel cladding.

13. The room temperature tensile properties of PM Mo-13 Re are comparable to the high carbon VAC Mo-13 Re alloy. The ductility indicated by total elongation and reduction of area is very high.

14. The excellent room temperature ductility of the PM Mo-13 Re is indicated by the broken tensile specimens. More plastic instability is observed for the specimens with the stress-relieved condition.

15. The fractographs of Mo-13 Re indicate brittle failure as the recrystallized sample shows transgranular cleavage, and the stress-relieved samples do not exhibit classical cup and cone ductile failure. Grain boundary separation might be indicative of reduced ductility in the transverse direction.

16. Seamless tubing has been made successfully from VAC Mo-11 Re and Mo-13 Re using a process that involves warm swaging extruded bar to near the tube blank diameter, gun-drilling a hole down the center of the swaged bar, and cold-drawing the blank through a hardened steel die while using a bullet-shaped floating mandrel to maintain the internal diameter of the tubing at a value dictated by the desired wall thickness reduction. In-process stress relief of the tubing was required after about every 80% reduction of the wall cross-sectional area. These alloys were stress-relieved by heating in dry hydrogen at about 1200 K for 15 minutes.

17. A single length Mo-11 Re tubing was drawn to nearly 15 ft from a bar that was only about 2 ft long. The tubing was delivered in the stress-relieved condition. The tubing was accidentally bent during shipping which contrasts with a previous experience in which a pure molybdenum tube arrived broken into about four pieces after similar treatment during shipment.

18. A variety of weldability studies have been performed on both VAC and PM Mo-Re alloy products. Longitudinal EB welds were placed in VAC Mo-13 Re tubing, and ring tension tests were performed on several sections of this welded tubing. Mo-13 Re end caps were successfully EB welded into Mo-11 Re cladding tubes. EB weld trials were also performed on PM Mo-13 Re tubes with PM Mo-13 Re end caps. Gas-tungsten arc (GTA) welding studies were performed at AMRC on both Mo-11 Re and Mo-13 Re using Mo-20 Re filler metals. Plates 3/16" thick were successfully welded inside a high purity inert gas glove box. Room temperature tensile tests were performed on samples having the weld zone across the center of the test sections.

19. The results of the ring tests on the EB welded Mo-13 Re tubing shows strength and ductility comparable to the parent metal.

20. Microstructures of EB welds in VAC Mo-11 Re were sound and pore-free, while EB welds in PM Mo-13 Re were sound but somewhat porous. Both welds were helium leak tight.

21. Ductile GTA welds were obtained in both Mo-11 Re and Mo-13 Re using Mo-20 Re filler metal . Mo-11 Re gave the best ductility. Both the elongation and the yield stress were fairly strain-rate insensitive over four orders of magnitude. The failures were seen to occur equally in the welds or heat affected zones (HAZ).

22. The reassembled GTA welded tensile test specimens show the higher room temperature ductility of the Mo-11 Re specimen.

23. A survey of the creep behavior of three of the VAC Mo-Re alloys was performed at ORNL in high vacuum facilities. Test temperatures included 1400, 1500, and 1600 K, and stresses included 2000, 3000, and 4000 psi. Step increases in both stress and temperature to obtain a quick survey of creep behavior. Tests at 1500 K, 3000 psi were run to >4000 h.

24. The creep strengths of the VAC Mo-Re alloys are somewhat lower than predicted from published data for electron beam melted alloys. This difference is more likely due to a difference in creep mechanism at the lower stress rather than the difference in melting process.

25. The producibility of low Re content Mo-Re alloys is crucial to their useage. The AMRC study and the production of test samples for the irradiation experiments demonstrated their optimum composition and their ability to be formed into useful products. Their joinability by EB and GTA welding methods has also been demonstrated.

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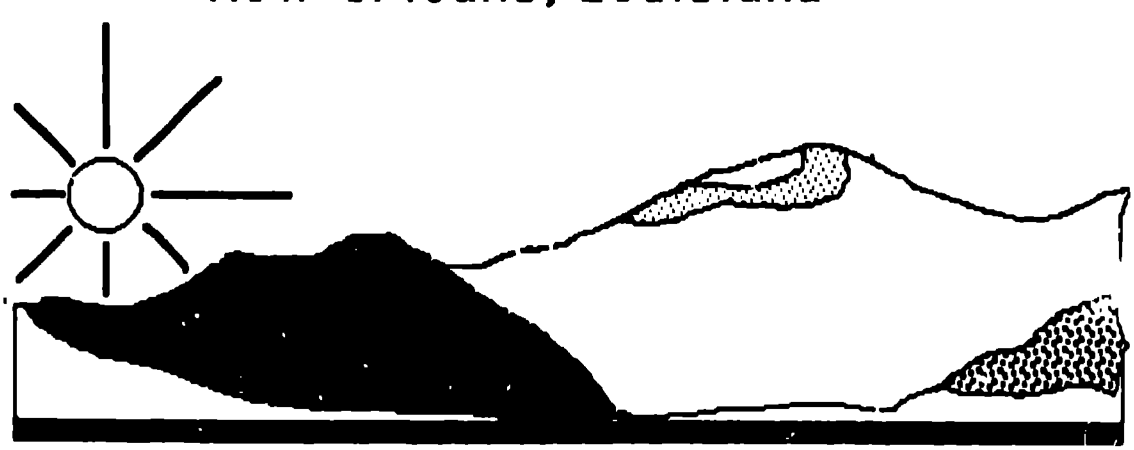
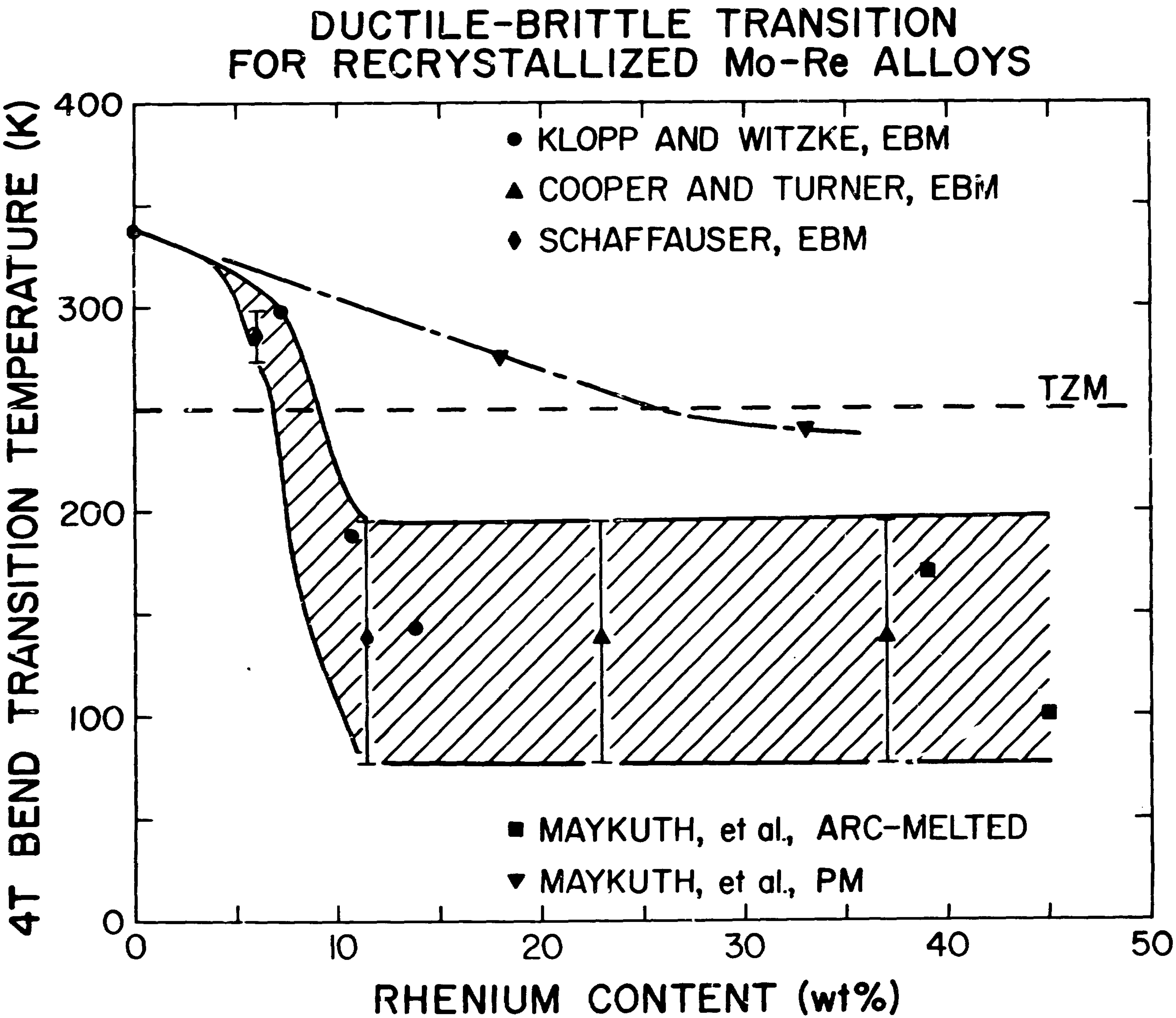


ADVANCED ENGINEERING TECHNOLOGY
Q-13


DUCTILE-BRITTLE TRANSITION
FOR RECRYSTALLIZED Mo-Re ALLOYS
4T BEND TRANSITION TEMPERATURE (K)
RHENIUM CONTENT (wt%)
KLOPP AND WITZKE, EBM
COOPER AND TURNER, EBM
SCHAFFAUSER, EBM
MAYKUTH, et al., ARC-MELTED
MAYKUTH, et al., PM
TZM
0
100
200
300
400
0
10
20
30
40
50

SOLUTION SOFTENING IN Mo-Re ALLOYS

- Flow stress, elastic modulus, and DBTT reduced.
- Occurs in Group VIA metal alloys containing small quantities of VIIA or VIIIA metals <0.16Tm.
- Intrinsic effect.

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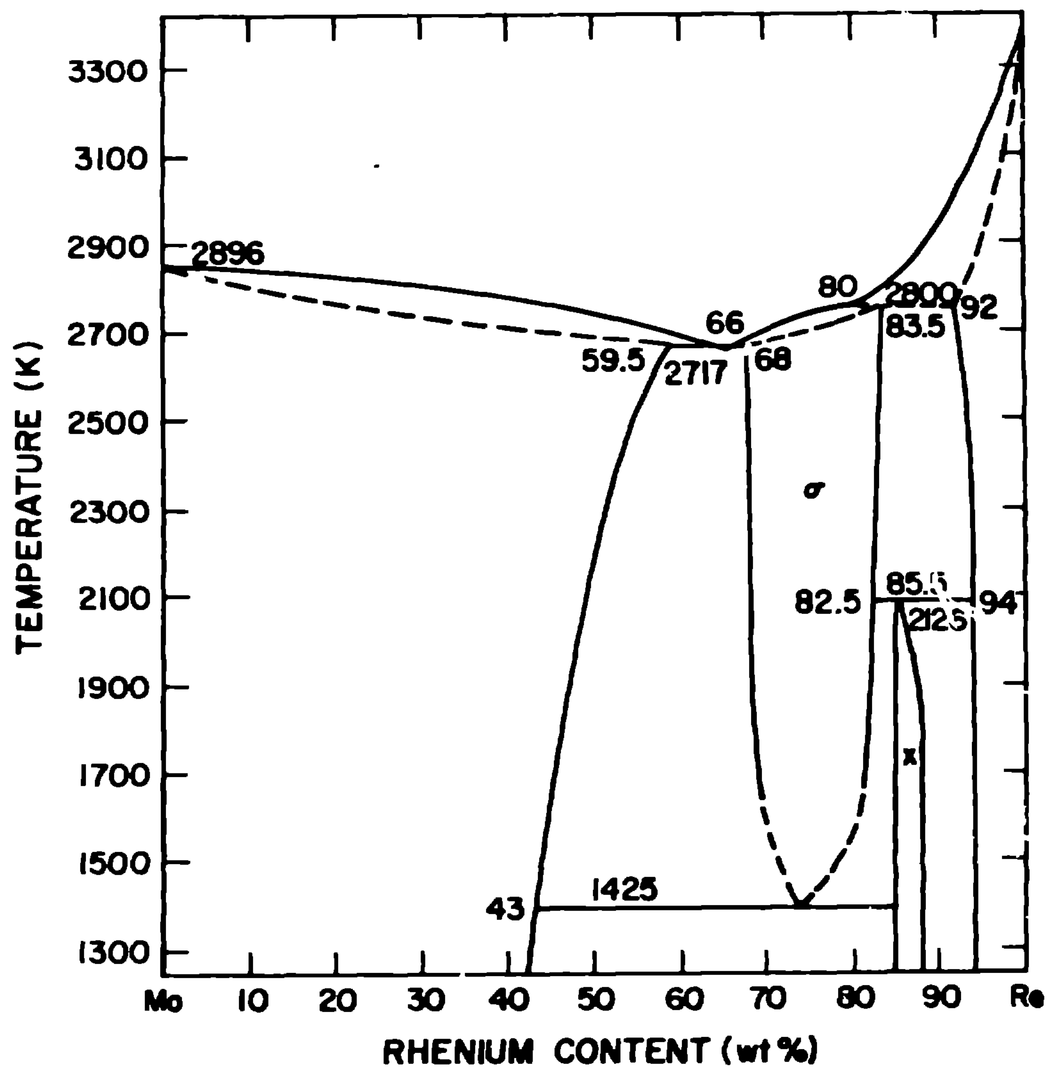
MOLYBDENUM-RHENIUM EQUILIBRIUM PHASE DIAGRAM



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Mo-Re ALLOY DEVELOPMENT PROGRAM

- AMAX VAC ALLOY STUDIES
- IRRADIATION EXPERIMENT MATERIAL
- CREEP STUDIES
- PM ALLOY STUDIES

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Mo-Re DEVELOPMENT STUDY AT AMRC

- VAC Alloys 5, 7, 9, 11, 13, 13+C, 15, and 17 wt% Re.

- All alloys hot extruded to rectangles, warm-rolled, and cold-rolled to plate.

- Evaluations included hardness, recrystallization, low-temperature tensile behavior, elastic moduli, GTA weldability, and CTE.

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PRODUCTION OF VAC Mo-Re ALLOYS

BLEND PURE METAL POWDERS

COLD-PRESS AND SINTER ELECTRODE

VAC ELECTRODE

EXTRUDE BILLET, 1700 K, 5:1

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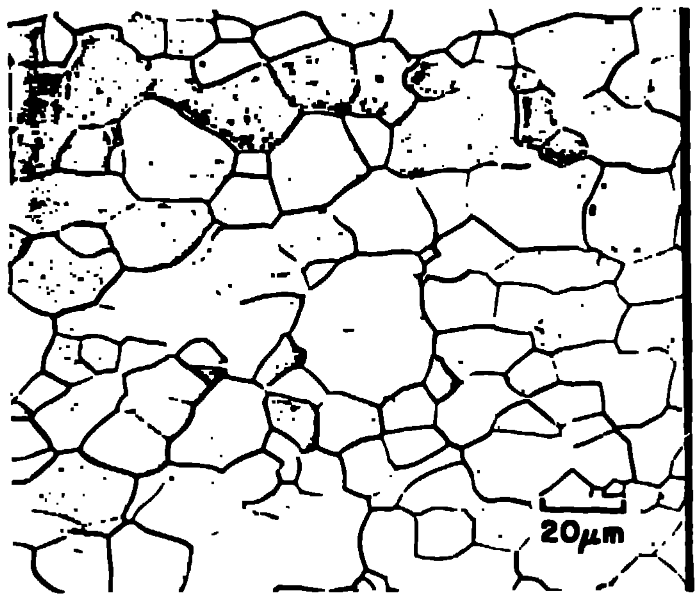
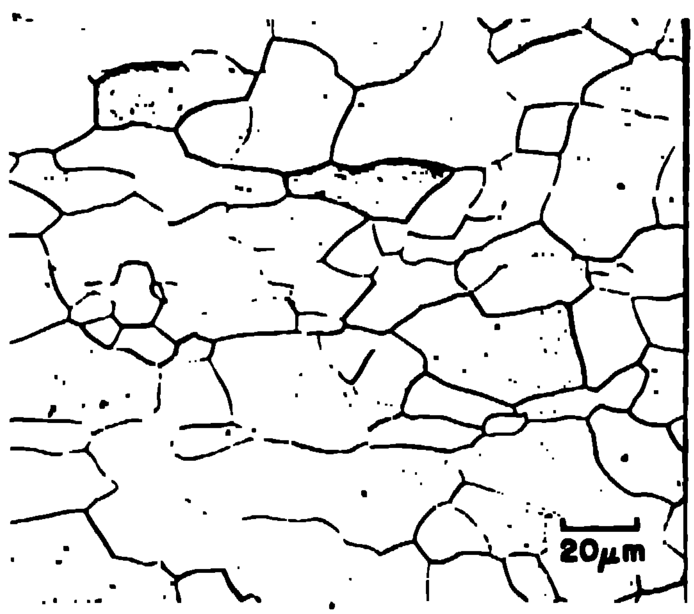


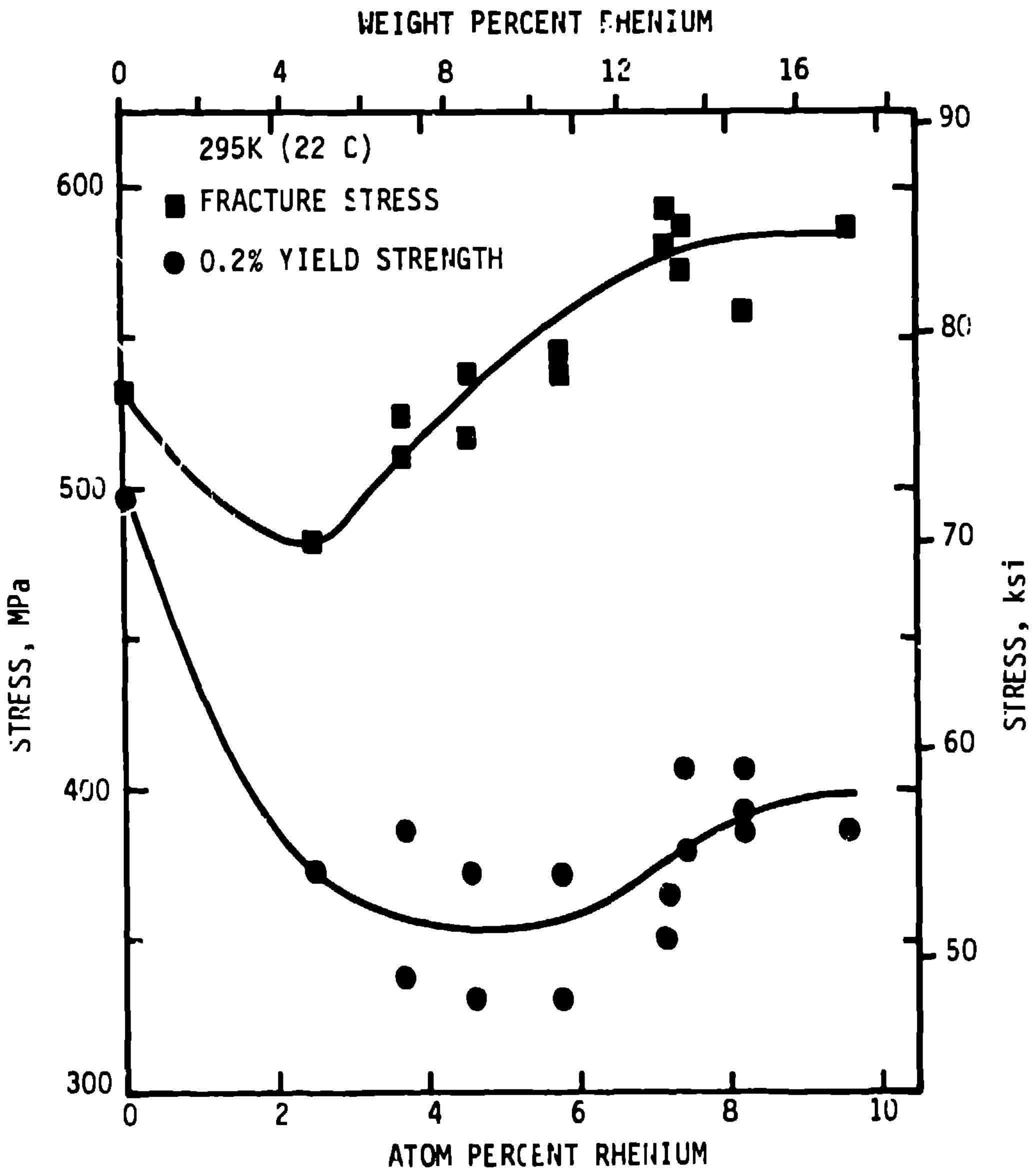
(a)



(b)

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

ROOM TEMPERATURE STRENGTH OF Mo-Re



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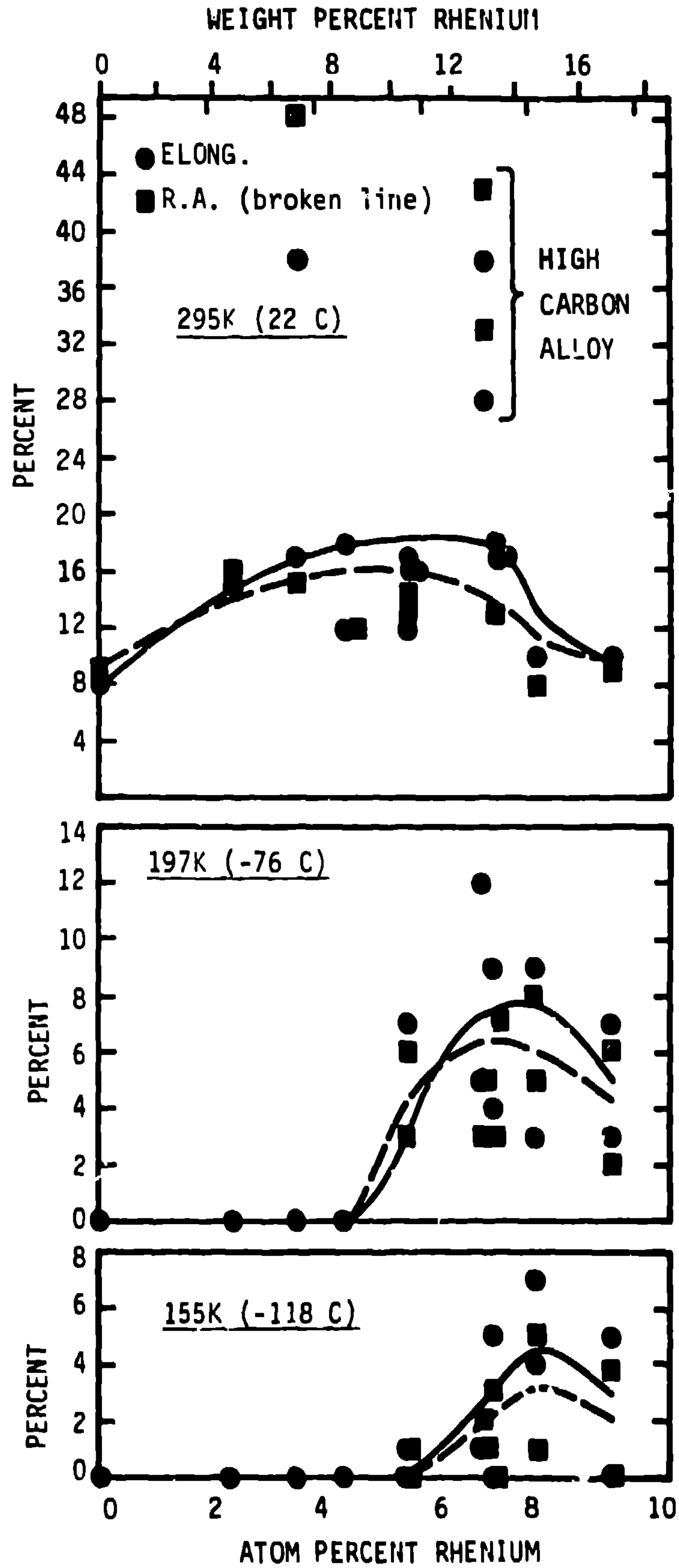
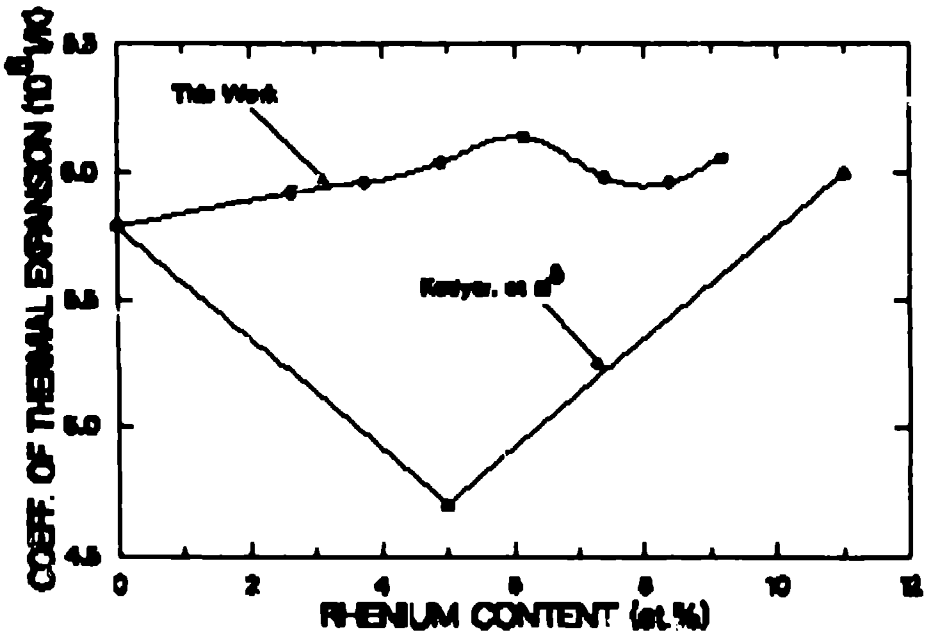
Mo-Re DUCTILITY PEAK IS TEMPERATURE DEPENDENT



Mo-Re CTE at 873 K



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OPTIMUM VAC Mo-Re ALLOY COMPOSITION

- Rhenium Content = 12±1wt%
- Carbon Content = 100 wppm Max.
- Nitrogen Content = 20 wppm Max.
- Oxygen Content = 30 wppm Max.
- Hydrogen Content = 5 wppm Max.

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Mo-Re PRODUCTS FOR IRRADIATION EXPERIMENTS

Mo-13Re

- Biaxial Creep Tubing (with bar for end caps) - 0.230" o.d. x 0.015" thick wall.
- Plate for End-Caps - 0.5" thick.
- Plate for Charpy Specimens - 0.25" thick.
- Sheet for Fuel Pin Spacers - 0.625" thick.
- Sheet for Tensile and Bend Specimens - 0.030" thick.

Mo-11Re

- Fuel Clad Tubing - 0.300" o.d. x 0.025" thick wall (with bar).

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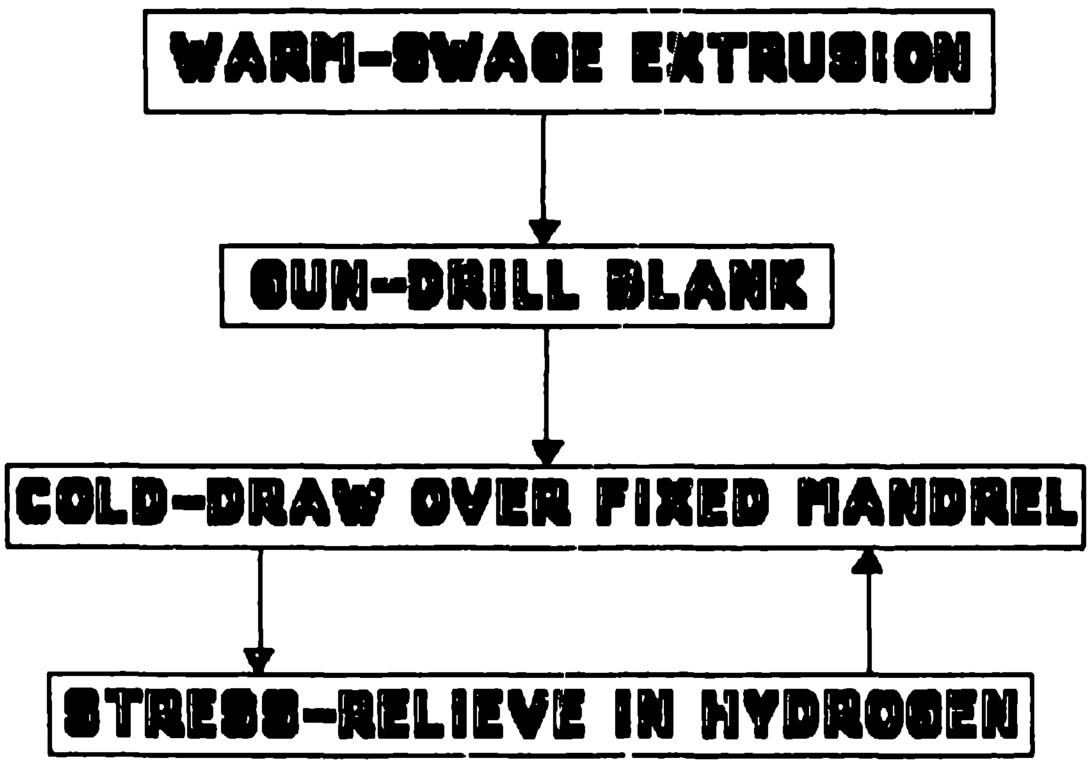
Mo-Re SEAMLESS TUBE FABRICATION PROCESS



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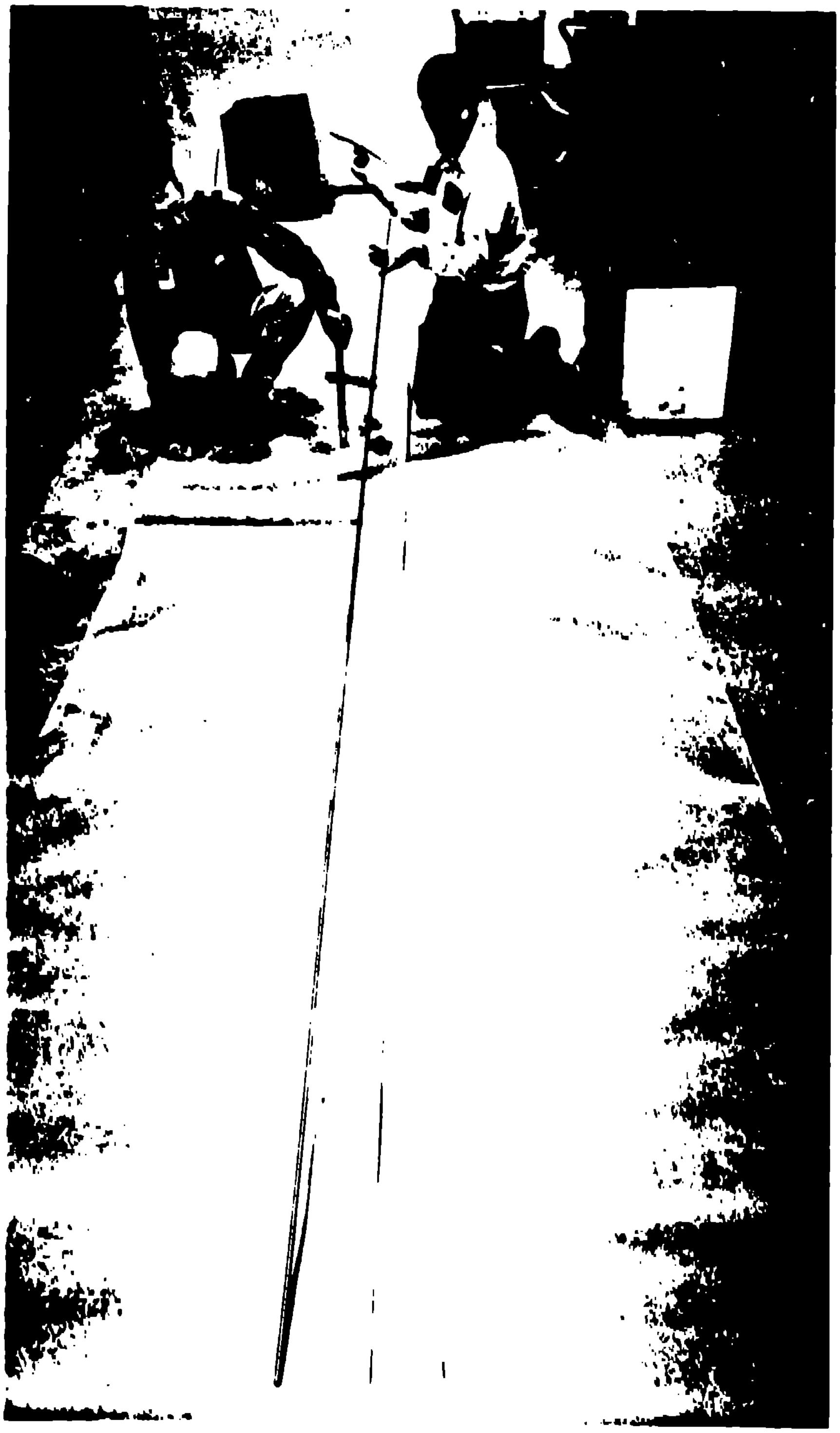
Mo-11Re CLAD TUBE



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WELDABILITY STUDIES HAVE BEEN PERFORMED

- **ELECTRON BEAM WELDING STUDIES**
  - **Longitudinal Welds in Mo-13Re Tubes have been Evaluated.**
  - **Cladding Tube Closure Welds were Qualified in Mo-11Re.**
- **GTA WELD STUDIES**
  - **Mo-11Re and Mo-13Re were Welded with Mo-20 Re Filler.**
  - **Room Temperature Tensile Tests Performed on Welds.**

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Mo-13Re EB-WELD STRENGTH AND DUCTILITY INDICATED BY RING TENSION TESTS

	UTS (ksi)	RA (%)
PARENT METAL	75	11
WELDED TUBE	81	14*

*Parent metal fracture, weld also failed with about 3% RA.

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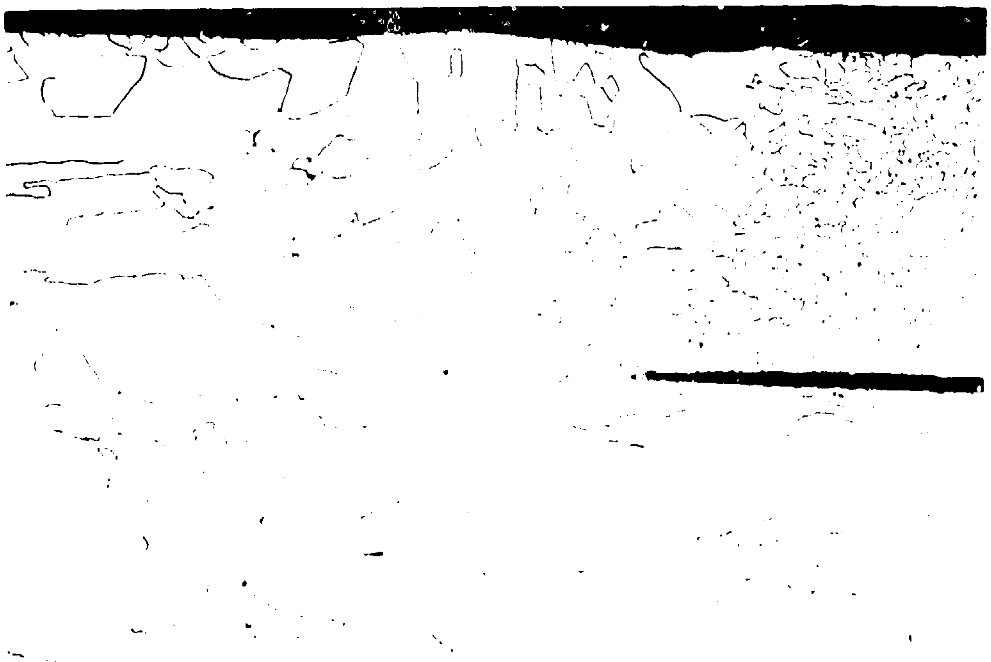
EB-WELDS IN Mo-Re



VAC Mo-11Re

PM Mo-13Re

152 µm

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DUCTILE GTA WELDS WITH Mo-20Re FILLER

Mo-11Re

STRAIN RATE (1/s)	ELONG. (%)	0.2 YIELD STRESS (MPa)	ULTIMATE STRESS (MPa)	FRACTURE BEHAVIOR
10^{-4}	16	250	511	IN-WELD
10^{-3}	12	281	500	2-HAZ, 1 IN-WELD
10^{-2}	14	315	548	HAZ
10^{-1}	11	364	537	IN-WELD

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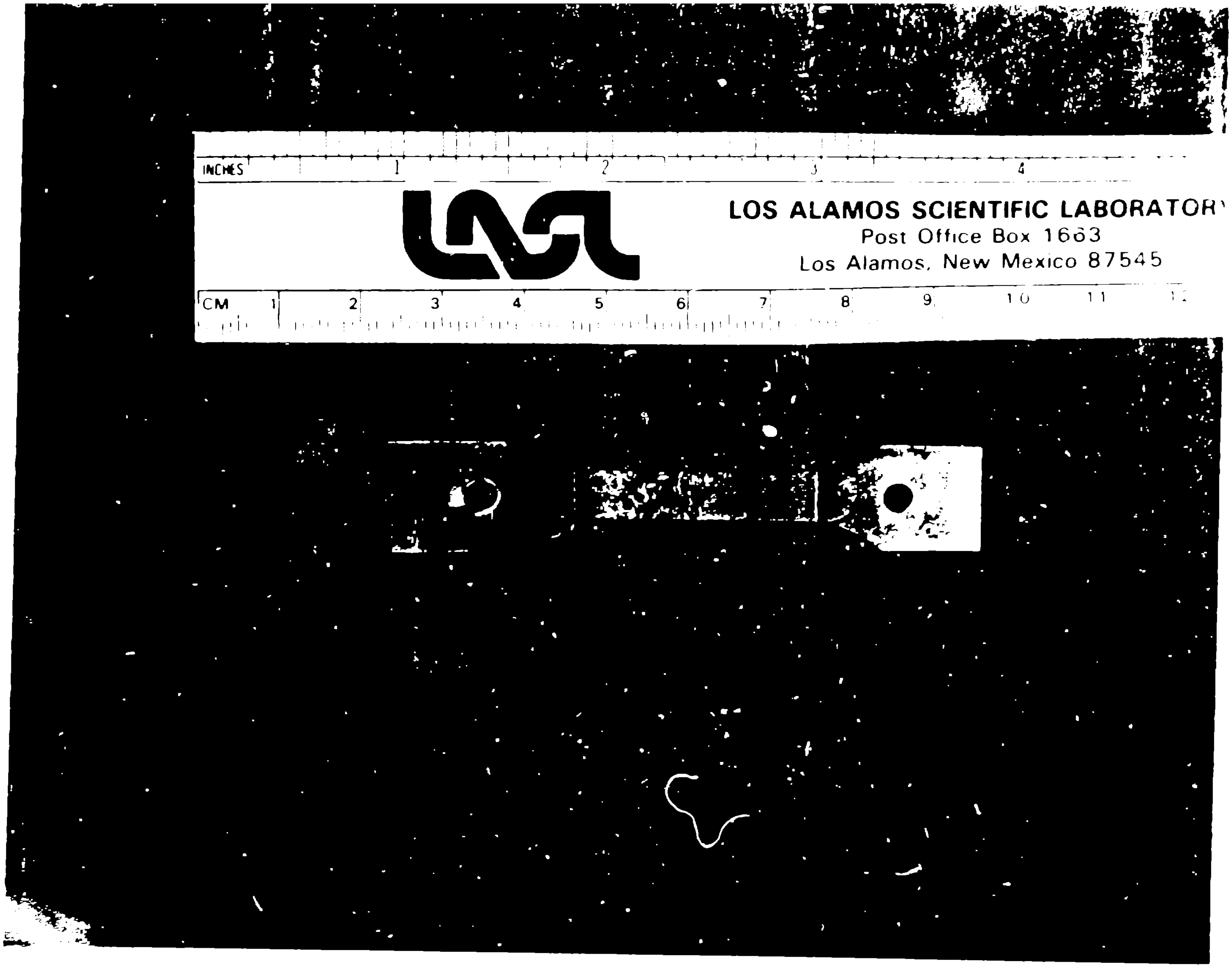

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ROOM TEMPERATURE TENSILE BEHAVIOR OF PM Mo-13Re

CONDITION	UPPER YIELD STRENGTH (MPa)	LOWER YIELD STRENGTH (MPa)	UTLIMATE STRENGTH (MPa)	ELONGATION (%)	REDUCTION OF AREA (%)
RECRYST.	295	270	560	43	43
	305	270	560	50	51
STRESS-RELIEVED	670	---	670	32	61
	480	---	660	42	62

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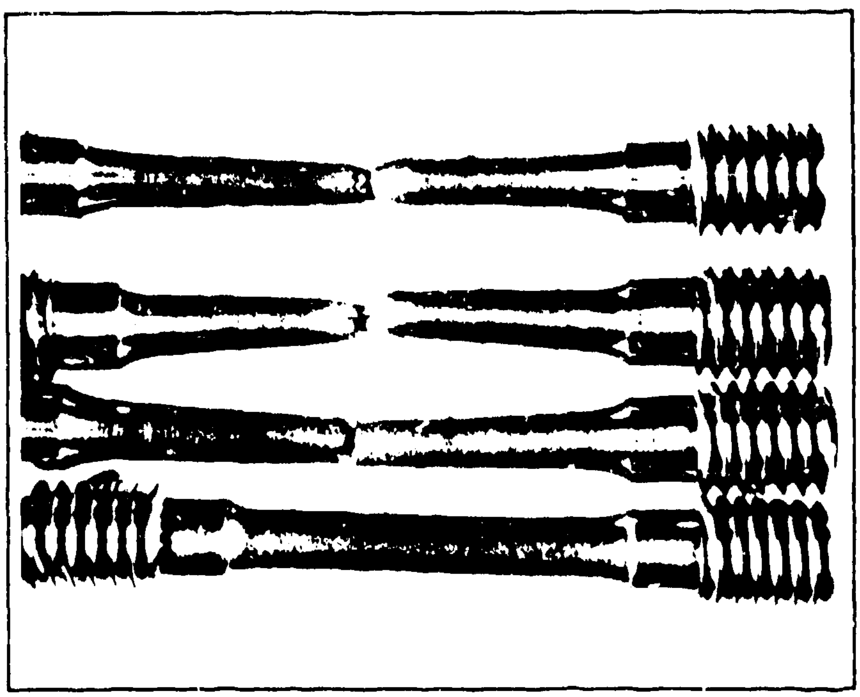
Mo-13Re EXHIBITS EXCELLENT ROOM TEMPERATURE DUCTILITY



Stress-Relieved

Recrystallized

Original Sample

1 cm

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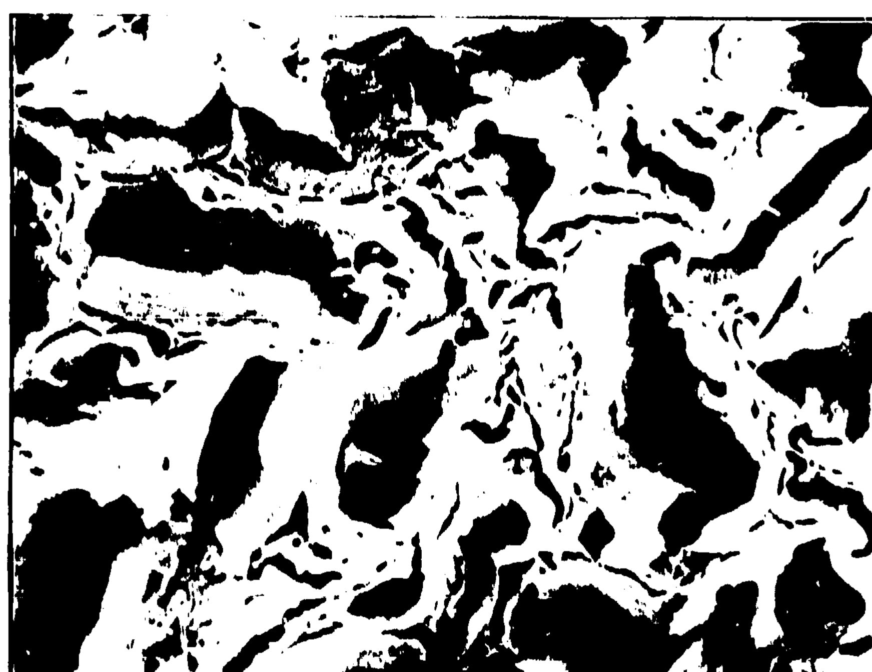
FRACTOGRAPHS OF Mo-13Re ARE MISLEADING

Recrystallized PM Alloy

20 μm



Stress-Relieved PM Alloy

10 μm

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VAC Mo-Re CREEP SURVEY

STRESS (MPa)	TEST TEMPERATURE (K)		
	1400	1500	1600
13.8		Mo-9Re Mo-11Re Mo-13Re+C	
20.7	Mo-11Re Mo-13Re+C	Mo-9Re Mo-11Re Mo-13Re+C	Mo-11Re Mo-13Re+C
27.6		Mo-9Re Mo-11Re Mo-13Re+C	

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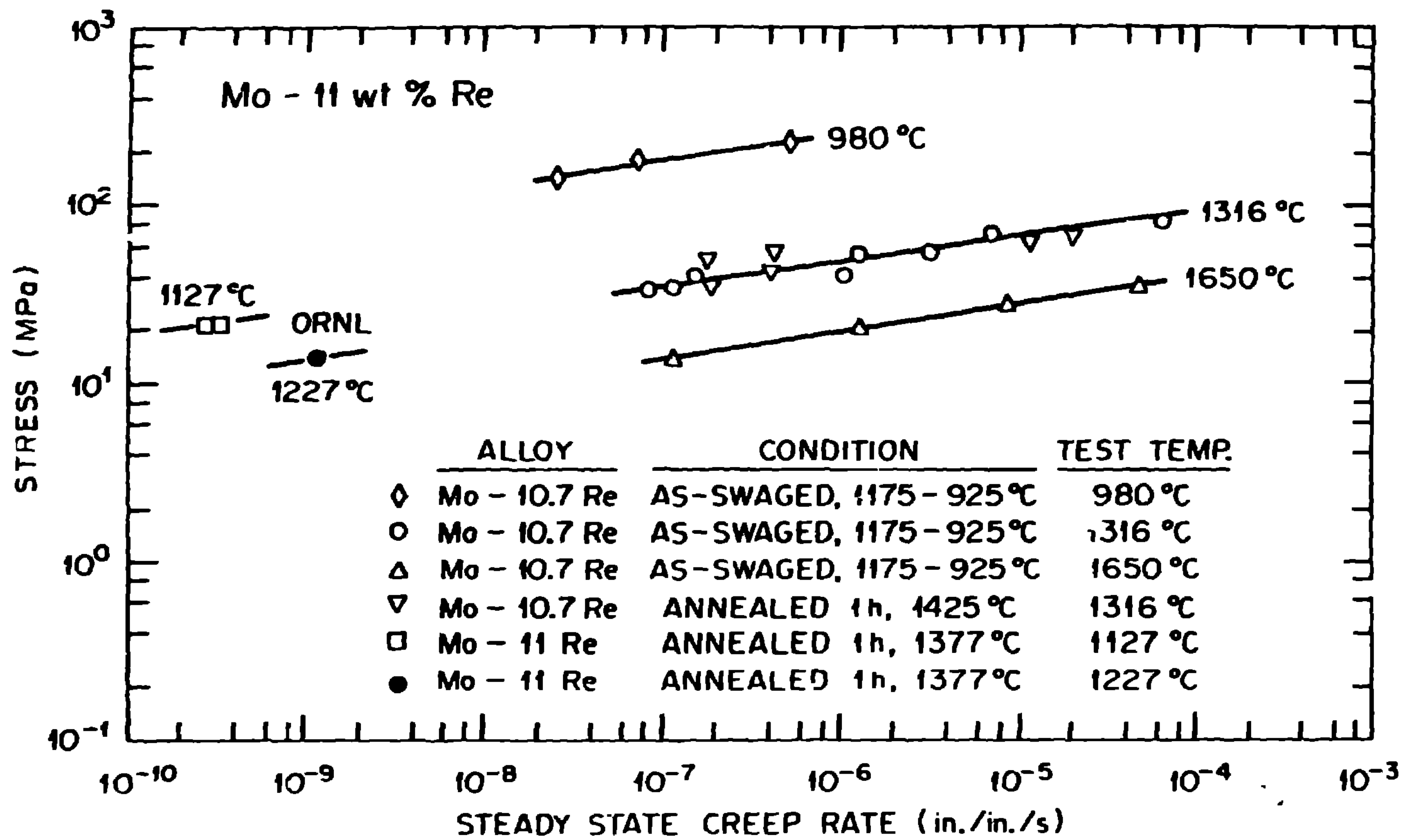
VAC ALLOYS APPEAR TO HAVE LOWER CREEP STRENGTH



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ALLOY PRODUCIBILITY IS CRUCIAL

- Optimum Alloy Composition was Determined from AMAX Work.
- Fabricability found to be Good.
- Joinability Studies Included EB and GTA Welding.

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