Survey of Irradiation Data on Molybdenum

R.E. Schmunk and G.L. Kulcinski

June 1976

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FUSION TECHNOLOGY INSTITUTE

UNIVERSITY OF WISCONSIN

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"LEGAL NOTICE"

Introduction

Molybdenum and its alloys with titanium and zirconium have been frequently mentioned as back up alloys for the LMFBR[1-3] and are seriously being considered as structural materials for fusion reactors.[4-10] The attractive properties of this element include high elevated yield and creep strength, high thermal conductivity, low thermal expansion coefficient, high Youngs Modulus, extremely good corrosion resistance in liquid metals and high temperature He, reasonably low absorption coefficient for fission spectra neutrons and high (n,2n) cross section for fusion neutrons, low solubility and permeability of tritium, and moderately low cost (compared to other high temperature metals). Its drawbacks as a nuclear material include joining and welding, volatile oxide formation at elevated temperatures in air, and high long lived radioactivity. On balance, this element and its alloys can provide some very attractive options to the use of stainless steel and will certainly be studied for many years to come.

The purpose of this study is to gather together, in one document, as much of the experience on the response of Mo to various kinds of irradiation as is possible. It is hoped that this report will not only aid researchers in pinpointing information that may be required for a definitive assessment of the Mo system in any given irradiation environment, but also to lend some perspective to investigators about the "holes" in existing data. Such a study as this literally is out of date the moment it is printed and we therefore urge the reader to update this report continuously. It is felt that the information presented here represents practically all of the work conducted on irradiated Mo (or its alloys) from 1956 to the summer of 1976. We briefly describe in Chapter II the method by which we referenced this material and Chapter III covers the chronology of data generation along with a comparison of types of testing performed at various temperatures. Chapter IV gives a perspective on where the data has been generated (i.e., laboratory and country) while Chapter V separates the data on the basis of investigative technique (i.e. electron microscopy, resistivity, mechanical property, etc.). A certain amount of cross referencing is listed in Chapter VI and conclusions are presented in Chapter VII. Finally, Chapter VIII lists the complete bibliography compiled during this study which can serve as a basis for future updating of this work.

References for Chapter I

1. L. L. Bennett, W. E. Thomas and F. J. Homan, "Nuclear and Economic Performance of Niobium and Molybdenum in LMFBR Cores," Trans. Am. Nucl. Soc. 13, 469, 1970.

2. D. T. Eggen and P. R. Huebolter, "Molybdenum to Mitigate Radiation Swelling Problems in LMFBR Fuel Assemblies," Trans. Am. Nucl Soc. 14, 169, 1971.

3. P. R. Huebotter, T. R. Bump, W. T. Sha, D. T. Eggen, and P. J. Fulford, "Design Research, and Development Implications of Metal Swelling in Fast Reactors," ANL-7786, April 1971.

4. R. Carruthers,P. A. Davenport, and J. T. D. Mitchell, "The Economic Generation of Power from Thermonuclear Fusion" CLM-R-85 (1967).

5. R. Carruthers, "Engineering Parameters of a Fusion Reactor," Proc. Conf. Nuclear Fusion Reactors, UKAEA, Brit. Nucl. Energy Soc. (Sept. 1969),p. 337.

6. R. G. Mills, "Some Engineering Problems of Thermonuclear Reactors," Nucl. Fusion, 7 (4) 232, 1967.

7. S. Forester, and K. U. Schneifer, "Design Possibilities and Consequences for the Conventional Parts of Fusion Power Plants," Proc. Symp. Fusion Technology, Aachen, Germany, Sept. 1970.

8. I. J. Spalding, "CUSP Containment and Thermonuclear Reactors," Nuclear Fusion, 8, (3), 161, (1968).

9. B. Badger et al., "UWMAK-III," UWFDM-150, 1976.

10. K. Sako et al., "Design Studies of a Tokamak Reactor," IAEA-CN-33/G 1-5 in Plasma Physics and Controlled Nuclear Fusion Research 1974, Vol. III, IAEA, Nov. 1974.

II. Description of Referencing Technique

All papers included in this bibliography are listed in the appendix, arranged alphabetically according to the author's (first author) name. Each paper has been assigned an alpha-numeric identification which consists of a letter and two numbers. The letter and first number of the identification are assigned to a given author and appear with all appers listed under his name in the appendix. The second number distinguishes between different papers belonging to a given author. As an example the paper given the code B.9.5 has as first author J. L. Brimhall, and it is the fifth paper listed under his name. All papers dealing with the molybdenum alloy TZM as well as Mo-0.5%Ti can be found easily in the appendix because the author code is underlined for these papers.

In the sections that follow the papers are categorized according to the properties being investigated, the type of irradiation, irradiation temperature and fluence. Participation of different laboratories is also identified, and the laboratories are referred to by a code number which is given in section IV.A. The data plots of section V are particularly important because they display the levels of radiation and the irradiation temperatures to which the examined materials have been exposed.

III. Overview of Investigation on Molybdenum

Tables III-1 thru 3 have been prepared to obtain a perspective on the chronology of the work on Mo and its alloys as well as to outline the general temperature regions where investigations have been carried out. The first table reveals the following information:

. 70% of the investigations of irradiated Mo have been with neutrons

. Over half of the work on irradiated Mo has been reported in the last 5 years.

. The use of electrons as an investigative tool for fundamental properties has increased in the past 10 years with over half of the reported work occuring in the last 5 years.

. Heavy ion studies have, after an initial burst of activity in 1971, begun to increase in the past two years.

. Less than 10% of the studies have used light gas ions such as the hydrogen isotopes and helium.

We further subdivided the information in Table III-2 into that associated with the specific type of investigative tool. Some pertinent points about Table III-2 are listed below.

. The early work on Mo utilized electrical resistivity, X-ray analysis and mechanical property measurements to characterize the damage state.

. The use of X-ray analysis has fallen off in the past 10 years.

. The use of transmission electron microscopy has increased from the 1960-5 period where it represented only 20% of the work to the 1970-75 period where it was used in approximately 40% of the studies.

. The electron damage work has been very strong for the past 7 years and this period has accounted for roughly 75% of the reported work in the study of electrical resistivity increases.

. Practically all of the transmission electron microscopy work has been performed on neutron and heavy ion irradiated material with approximately 60% of that work associated with neutron effects and 40% with heavy ion bombardment.

. As might be expected, most of the mechanical property measurements have been performed on neutron irradiated specimens and that work has been carried on at a fairly steady rate for over 15 years.

The temperature regimes which have been studied by the various techniques are summarized in Table III-3. A few of the observations on this data are:

. There have been a large number (43) of investigations at temperatures <100°K but very few in the 100-273°K range.

. The region from 0-200°C has been investigated most heavily while that from 200-450°C has received less attention.

. A fair number of studies have been conducted in the 450-1000°C range with some 16 reports of Mo irradiated above 1000°C.

. As might be expected, roughly 70% of the low temperature work has been characterized by electrical resisitivity but very few measurements of irradiation induced resisitivity increase have been made above 200°C.

. All of the X-ray, stored energy and positron annihilation experiments have been performed on samples irradiated above 0°C.

. The region from room temperature to 1000°C has been fairly well covered by TEM and scanning electron microscopy studies. The region from 450-1000°C has been particularly emphasized because of the void studies.

. Over half of the mechanical property measurements have been made on Mo irradiated below 200°C and no substantial in-reactor measurements of mechanical properties have been made.

The tables given here do not completely illustrate the breadth and depth of the data so we will attempt in sections V and VI to provide even more perspective.

Table III-1

III-1 Chronology of Experiments by Type of Irradiation

	n	p	α	e^-	HI	d	TOTAL
1955							
56	1						1
57	1						1
58	2						2
59	1						1
1960	8						8
61	3						3
62	6	1	1	2			10
63	4						4
64	4				1		5
65	12			1	1		14
66	2			1			3
67	10				1		11
68	7				1		8
69	16			2			18
1970	9			3			12
71	19	1		1	7		27
72	13		2	8	3	1	27
73	16		2	3	1	1	23
74	12	2	3	4	1		22
75*	12			2	4		18
76*	12			3	7		25
	170	4	8	30	27	2	243

n = Neutron

p = Proton

α = Alpha Particle

e^- = Electron

HI = Heavy Ion

d = Deuteron

*Numbers should be considered incomplete

Table III-2

Chronology of Investigations by Physical Property vs. Type of Irradiation

YEAR	Resistivity			TEM					Mech. Properties				X Ray			Other			
	n	e^-	Other	n	p	α	HI	Other	n	e^-	α	Other	n	α	Other	n	α/p	HI	Other
1956	1																		
57			(1)						1										
58	1		(1)										1						
59													1						
1960	3								4				2						
61	1								1				1						
62	1	2		1	1	1			4										
63	1			1					1				1						
64	2						1	(1)	2										(1)
65	1	1		6			1		3				1			1			
66		1		1					1			(1)	1						
67	1		(2)	5					3			(1)			(1)			1	
68	2		(1)	4				(2)	1			(1)	2			1		1	(1)
69	3	2	(1)	7			1		7			(1)			(2)				
1970	4	3	(3)	3				(2)	3				2						2
71		1	(2)	15			7	(2)	1										4
72	5	7	(3)	6			4		3			(2)		1					2 (1)
73	5	3	(1)	9			1	(1)	6	1	1	(3)					1/		1
74	1	2		8			1	(1), 1-e^-	5	1	2	(3)			1		2/ 3		7(1)
75 *	2	4	2	7		1	6	(1)	4							1			2(2)
76*		14	1	2			3	1	2							1			1(1)
TOTAL	32	40	3(15)	75	1	2	25	2(10)	52	2	3	(12)	12	1	1 (3)	4	3/3	2	19 (7)

( ) = NO Irradiation

* Numbers should be considered incomplete

Table III-3 - Number of Papers (Property Vs. Irradiation Temperature)

		0-100°K	100-273K	0-50C	50-200C	200-450C	450-700C	700-1000C	>1000C
Mechanical Properties	TOTAL	4	0	13	26	8	12	14	3
	CREEP	-	-	1	6	1	2	5	
	TENSILE	1	-	8	12	4	6	6	3
	HARDNESS	-	-	4	5	2	4	3	
	OTHER	3	-		3				
RESISTIVITY		30	0	13	14	1	1	1	0
TEM		5	0	22	24	14	42	38	8
X-RAY		0	0	13	3	1			
SURFACE		2	1	6	1	3	4	3	3
STORED ENERGY		0	0	2					
POSITION				3	1	1			
TOTAL		45	1	85	95	35	71	70	17

IV. Summary of Literature by Working Groups

Table IV-1 lists all authors, primary (marked by an asterisk) and secondary, who have contributed to the work on molybdenum over the years. A total of sixty-four working groups are included in the listing. In the case of General Electric Company and Westinghouse, several groups of authors from within each company are listed together.

Table IV-2 shows the distribution of reported work by chronology and laboratory. The total number of investigations per year shows a steady increase from the mid-1950's up to the present time, which at least partially reflects the interest in molybdenum as a potential material for constructing fusion reactors. Most of the investigations have been conducted by a select few laboratories. Battelle's Pacific Northwest Laboratory (PNL) has published the largest number of papers on molybdenum followed by the Harwell group. Nearly equalling the efforts of these two groups are the combined efforts of General Electric (Cincinnati) and the University of Cincinnati which are primarily due to John Moteff. The ten most productive laboratories have produced two thirds of the papers, and the remaining papers are scattered evenly among the other contributors.

Looked at another way, Table IV-3 indicates that TEM as an investigative technique has been used twice as much as the other methods. Those laboratories which have been most productive in terms of publications (PNL and Harwell) have concentrated their efforts in the use of TEM, reporting nearly half the work published for that technique. Resistivity work has been conducted primarily at Harwell and d'Orsay, France, although the publications reflect a slow steady effort. Mechanical property data have been generated principally by Moteff and co-workers followed by Oak Ridge National Laboratory and the USSR Academy of Science.

Turning to Table IV-4, neutron irradiations have been involved in approximately three-fourths of all irradiation experiments conducted on molybdenum. Of that effort, only one paper has been published to data for which irradiations were made with 14 MeV neutrons. All other neutron irradiations utilized either thermal or fast fission reactor facilities. The electron irradiations have been used for resisitivty studies while the proton, deuteron and alpha particle irradiations were conducted mainly for blistering or gas re-emission studies. The more recent heavy ion irradiations have been used for damage simulation studies.

Nearly half of the published irradiation effects work on molybdenum has been done in the United States. Principal U. S. research groups include PNL, Moteff and co-workers (both at G. E. and Univ. of Cincinnati) and ORNL. About twenty percent of the publications are from the U. K. and these are primarily from Harwell as cited earlier. A note of caution is in order with regard to drawing close comparison between different tables. Some publications include the use of more than one investigative approach and a number of papers are included, such as theory, which did not involve any irradiations. Hence, one cannot be too definitive about the comparison of the number of papers from table to table.

Table IV-1

Listing of Organizations and Authors Who Have Contributed to the Literature on Irradiated Mo and Mo Alloys

1. Battelle Northwest Laboratories (Formerly General Electric Company, Hanford)

   J. L. Brimhall*
   G. L. Kulcinski*
   H. E. Kissinger*
   B. Mastel*
   E. P. Simonen*
   E. R. Bradley*
   F. A. Smidt
   J. J. Laidler*
   T. K. Bierlein

2. General Electric Company, Vallecitos & Cincinnati

   R. C. Rau*
   R. L. Ladd
   J. Moteff*
   F. Secco D'Aragona
   J. P. Smith
   P. G. Luccasson*
   R. M. Walker
   C. A. Bruch*
   W. E. McHugh
   R. W. Hockenbury
   F. D. Kingsbury*
   R. L. Treinen
   D. L. Gray*
   W. V. Cummings*
   J. L. Kamphouse*
   D. A. Myers

3. Westinghouse, Bloomfield, N. J., Pittsburgh, Pa. and HEDL

   Bevil J. Shaw*
   R. H. Schnitzel
   J. M. Steichen*

4. General Atomic, Inc.

   R. H. Chambers*
   T. A. Trozera
   J. L. White

5. Ford-Dearborn

   P. Beardmore*
   P. H. Thornton

6. U.S. Steel

   S. S. Brenner*
   D. N. Seidman

7. RIAS

   H. R. Peiffer*

* = Primary authors

8. NASA-Cleveland

H. R. Grimes*
E. R. Gotsky
J. A. Dicarlo

9. Atomics International

A. G. Pard*
K. R. Garr
D. Kramers*
C. G. Rhodes

10. Oak Ridge National Lab. (Hollifield)

J. O. Stiegler*
F. W. Wiffen*
R. R. Coltman*
T. H. Blewitt
C. E. Klabunde
J. K. Redman
D. L. McDonald
C. T. Lui*
H. Inouye
J. R. DiStefano*
J. H. DeVan
J. Bentley*

11. Naval Research Laboratory

C. Z. Serpan, Jr.*
H. E. Watson
J. R. Hawthorne
D. J. Michel*
H. H. Smith
A. G. Pieper
F. A. Smidt, Jr.*
J. A. Sprague

12. Argonne National Laboratory

J. A. Horak*
T. H. Blewitt

13. Univ. of Wisconsin

K. Y. Liou*
H. V. Smith, Jr.
G. L. Kulcinski
P. W. Wilkes

14. Lawrence Radiation Laboratory

S. S. Lau*
J. E. Dorn
D. R. Olander*
J. L. Schofill, Jr.

15. E. I. duPont, Savannah River

G. R. Caskey, Jr.*
M. R. Louthan, Jr.
R. G. Derrick

16. Sandia

W. Bauer*
G. J. Thomas*

17. Michigan Tech.

R. A. Ayres*
D. F. Stun

18. Monsanto-Mound

R. E. Zielinski

19. State University of New York, Stonybrook

J. C. Bilello*
G. Cagliotti
S. C. Srivastava
L. L. Seigle

20. Naval Academy

D. F. Hasson*
R. J. Arsenault
Y. Huang
E. Pink

21. University of Maryland

R. J. Arsenault*

22. University of Cincinnati

V. K. Sikka*
J. Moteff*
D. J. Michel
R. C. Rau*
R. L. Ladd
C. G. Schmidt*
H. I. Jang*
J. F. Stubbins*

23. Franklin Institute

J. D. Meakin*
I. G. Greenfield
A. Lawley
R. C. Koo

24. Univ. of Calif., Berkeley

P. Rao*
G. Thomas

25. State University of New York, Albany

L. J. Cheng*
P. Sen

26. University of Minnesota

D. F. Stein*

27. Rice University

L. D. Whitemire*
F. R. Brotzen

28. Washington State University

A. A. Johnson*

29. Brown University

H. E. Farnsworth*
K. Hayek

30. UK-Harwell

J. H. Evans*
D. M. Maher*
B. L. Eyre*
S. Mahajan
R. Bullough*
M. R. Hayns
R. C. Perrin
K. Malen*
A. M. Stoneham
A. C. Roberts
P. R. B. Higgins*
A. F. Bartlett
M. H. Loretto
S. L. Sass*
M. E. Downey*
D. G. Martin*
H. K. Birnbaum *
G. H. Kinchin*
M. F. Makin*
E. Gillies
J. Adam*
D. J. Mazey

31. Culham Lab. (United Kingdom)

S. K. Erents*
G. M. McCracken*

32. Berkeley Nuclear Lab.

C. J. Beevers*

33. Queen Mary College, London

B. Etemad*
F. Guiu

34. Imperial College, London

J. F. Kerridge*
S. S. Sheinen
A. A. Johnson*
H. I. Matthews
D. E. Peacock*
A. S. Wronski*
N. Milasin
F. N. Zein
G. J. Irwin*
F. Guiu
P. L. Pratt

35. Royal School of Mines, London

D. N. Sethna*
A. A. Johnson
K. J. Proud
S. S. Sheinin

36. University of Surrey

P. N. Kenny*
P. T. Heald

37. J. J. Thomson Phys. Lab., Univ. of Reading

J. N. Lomer*
R. J. Taylor

38. University of Bradford

J. C. Thornley*
A. S. Wronski

39. University of Birmingham

J. Bentley*
M. H. Loretto

40. Adacemy of Science of U.S.S.R.

E. D. Martynov*
Sh. Sh. Ibragimov*
N. F. Pravdyuk*
S. Veljkovic
A. I. Zav'Yalov
V. A. Kasakov*
S. K. Mikhaylov*
I. M. Voronin*
V. A. Nikolaev
A. N. Vorob'ev
B. A. Vandyshev*
A. S. Panov
F. P. Burta
V. S. Lyashenko

41. Central Research Inst. of Ferrous Metallurgy

V. A. Ilyina*
V. K. Kritskaya*
B. V. Aharov
A. A. Vasil'Yev
A. P. Kuznetsova
B. V. Sharov

42. Inst. Metallurgy of Acad. Science

S. T. Borimskaya*

44. Petrozavodsk State University

I. A. Malinenko*

45. Moscow Engineering Physics Institute

V. N. Bykov*

50. J.A.E.R.I. (Japanese Atomic Energy Research Inst.)

S. Takamura*
H. Maeta
S. Okuda*
H. Mizubayashi

51. Tohoku University

T. Igarashi*
H. Oikawa
H. Matsui*
H. Saka
K. Noda
H. Kimura
T. Imura
S. Karashima*
M. Suezawa*
R. Hanada*
S. Takamura*
S. Okuda

60. University of Stuttgart, Stuttgart
Federal Republic of Germany

K. Niebel*
M. Wilkins

61. Academy of Science of the German Democratic Republic, Dresden

A. Köthe*
P. Burck
A. Luft*
L. Kaun

62. Julich, Federal Republic of Germany

G. Antesberger*
K. Sonneberg
H. Schroedor
U. Dudek

64. Belgium - MOL

L. Stals*
J. Nihoul
G. Goedeme*
B. M. Pande'*
J. Cornelis*
P. De Meester

67. Netherlands - Petten (RCN)

J. D. Elen*
G. Hamburg
A. Mastenbrook

68. University of Amsterdam

M. De Jong*
B. L. Wensink
H. B. Afman*
J. H. Mooy
H. Rademaker

70. Canada - AECL (Chalk River)

G. R. Piercy*
R. H. Tuxworth

71. AECL - Whiteshell

A. Semeniuk

74. The Technical Univ. of Denmark, Lyngby, Denmark

Kurt Petersen*
N. Thrane
R. M. J. Cotterill

75. AEK, Riso, Denmark

M. Eldrup*
P. E. Mogensen

77. Univ. Newcastle, N.S. W., Australia

W. A. Oates*
R. B. McLellan

80. France d'Orsay

P. Lucasson*
R. M. Walker
A. Lucasson*
R. Rizk*
P. Vajda
M. Biget*
J. P. Touboul*

81. Grenoble, France

P. Moser*
R. Pichon*
E. A. Bisogni

82. Saclay

Y. Adda*

85. Indian Inst. of Tech., New Delhi

O. P. Agnihotri*

90. Academy of Science, Leoben, Austria

E. Pink*

94. AEB, Pretoria, South Africa

C. M. Van der Walt*

95. Roanoke College, Salem, Virginia

C. A. Finfgeld

Table IV-2

Chronology of Work by Each Laboratory

Laboratory	56	7	8	59	60	1	2	3	4	5	6	7	8	69	70	1	2	3	4	75	76
1								1	1	4	2	3	3	8	3	3	4	4	1	4	5
2	1			1	1		1	1		3		1		7	1		1				
3								1						1							1
4										1											
5															1						
6																				1	
7			1														1				
8																		2			
9																	1			1	
10						1			2			1			1	3		3	1		2
11																		1			1
12																	1				
13																					1
14													1		1						
15																				1	
16																			2		
17																	1				
18																			1		
19																	1		1		
20																			1		
21															1		1		1		
22													1		1	1		2	3	1	4
23									1	1											
24													1								
25																				1	
26													1								
27													1								
28																		1			
29													1								
30		2	2						1	2			1	2 1	4	(13)	4	3	2	3	3
31																		2	1		1
32							1														
33																			1		
34					2		3	2							1						
35					1																
36													1						1		
37														1							
38														1							
39																				1	1
40					4		3		1					3					1	1	1
41										1									1		
42											1										
43											1			1							
44														1							
45																	1				
50														1				2			2
51															3	2	1	1	1		
52																					1
60																	1		2		
61																	1	1		1	2
62																				1	

Table IV-2 (cont.)

Year

Laboratory	56	7	8	59	60	1	2	3	4	5	6	7	8	69	70	1	2	3	4	75	76
64														1	3		1	3	2	1	2
67												1						2			
68										1					1	1	3				
70						1															
71																				1	
74																	2			3	1
74																		1			
80					1		1	1				1					1	2	2	2	
81																		3			
82																	1				
85																	1				
90																	1				
94																			1		
95																1					
TOTAL	1	2	3	1	9	2	9	6	3	13	4	12	3	20	21	25	31	32	26	23	25

Table IV-3

Distribution of Work for Laboratory by Type of Investigation

Laboratory	TEM	Resistivity	Type of Investigation Mech. Prop.	X-Ray & Other
1	34	4	4	8
2	4	5	7	3
3			2	1
4				1
5			1	
6	1			
7		1		
8		2		
9	1		1	
10	6	3	7	1
11	1			
12		1		
13	1			
14			1	1
15				1
16				2
17			1	
18			1	
19			1	
20			1	
21	1		3	
22	9	2	6	2
23	2			1
24	1			
25				1
26	1			
27		1		
28		1		
29				1
30	30	10	2	2
31				4
32	1			
33			1	
34	1	1	6	
35			1	
36	1		1	
37		1		
38			1	
39	2			
40	2	4	6	2
41			1	2
42				1
43			1	1
44				1
45				1
50		1	5	1
51	4	4	2	
52				1

Table IV-3

Distribution or Work for Laboratory by Type of Investigation

Laboratory	TEM	Resistivity	Type of Investigation Mech. Prop.	X-Ray & Other
60	3			
61	1	1	1	
62		3		
64	10			
67	3			
68	6			
70				1
71			1	
74	2			2
75	1			
77				1
80		10	1	1
81		2		1
82	1			
85		1		1
90			1	
94	1		1	
95				1
Total	131	58	68	48

Table IV-4. Distribution of Work for Each Laboratory by Type of Irradiation

	No. of Investigations by Type Irradiation					
Laboratory	n	p	α	e^-	d	HI
1	33			1		9
2	15					
3	2					
4	1					
5						
6	1					
7						
8						
9	1		1			
10	12					
11			1			1
12	1					
13						1
14						
16		2	2			
17						
18	1					
19						
20	1					
21	3					
22	12					1
23	1					
24	1					
25	1					
26						
27						
28	1					
29						1
30	25			4		9
31			2		2	
32		1	1			
33						
34	7					
35	1					
36	1					
37				1		
38						
39	2					
40	12					
41	1			2		
42	1					1
43	1					
44						
45			1			
50	7					
51	2			1	1	
60	2					1
61						

Laboratory	n	p	α	e^-	d	HI
62				3		
64	7			2		
67	3					
68				6		
70	1					
71	1					
74	6					
75				1		
77						
80	2			7		
81	2			2		
82	1					
85						
90						
94	1					
95		1			1	
Total	173	4	8	30	3	24

Table IV-5

Distribution of Papers on Molybdenum
by Country and Type of Investigation

National Group	TEM	RESISTIVITY	MECH. PROP.	X RAY & OTHER	TOTAL
U.S.	62	20	37	25	144
U.K.	35	12	12	6	65
USSR	1	4	8	10	23
JAPAN	4	5	7	1	17
GERMANY	3	4	1	-	8
BELGIUM	-	10	-	-	10
NETHERLANDS	3	6	-	-	9
CANADA	-	-	-	2	2
DENMARK	4	-	-	3	7
AUSTRALIA	-	-	-	1	1
FRANCE	1	12	1	2	16
INDIA	-	1	-	1	2
AUSTRIA	-	-	1	-	1
SOUTH AFRICA	1	-	-	-	1
TOTAL	114	74	67	51	306

V. Perspective on the Level of Irradiation Damage as a Function of Temperature

We have only considered the mechanical properties, transmission electron microscopy and heavy ion bombardment in this section. Figure 1 displays the neutron fluence versus temperature map for Mo and for reference, a zone of typical CTR condition is outlined. It is also worth noting that 10^{22} fission neutrons per cm^2 correspond to approximately 4 dpa.

One of the first observations that can be made is that there is very little information which has been obtained from specimens irradiated at even 1 $MW\text{-}yr/m^2$ (~8 dpa) damage levels for fusion reactor application. Post-irradiation tensile and creep tests have been performed in this region but there is no information on in-reactor fatigue, creep, or tensile strength. Data point number 15 comes from a specimen which experienced a large temperature excursion during irradiation and therefore the data is subject to some uncertainty. Once one drops below 10^{22} n cm^{-2} there is very little information at $T_{irr} > 400°C$. Obviously, much more work is required in this region if one wants to understand the behavior of Mo or its alloys in a fusion (or fission) environment.*

The situation is even worse with respect to fission reactor conditions ($T \cong 400\text{-}700°C$ and $\phi t > 10^{23}$ n cm^{-2}). Here there is no known data, as of late 1976, that could be used to predict the behaviour under typical test reactor conditions.

A large number of samples have been irradiated at room temperature at fluences from 10^{18} n cm^{-2} to ~10^{21} n cm^{-2}. Such studies, while valuable from a fundamental standpoint, shed little light on the high temperature, high fluence behavior.

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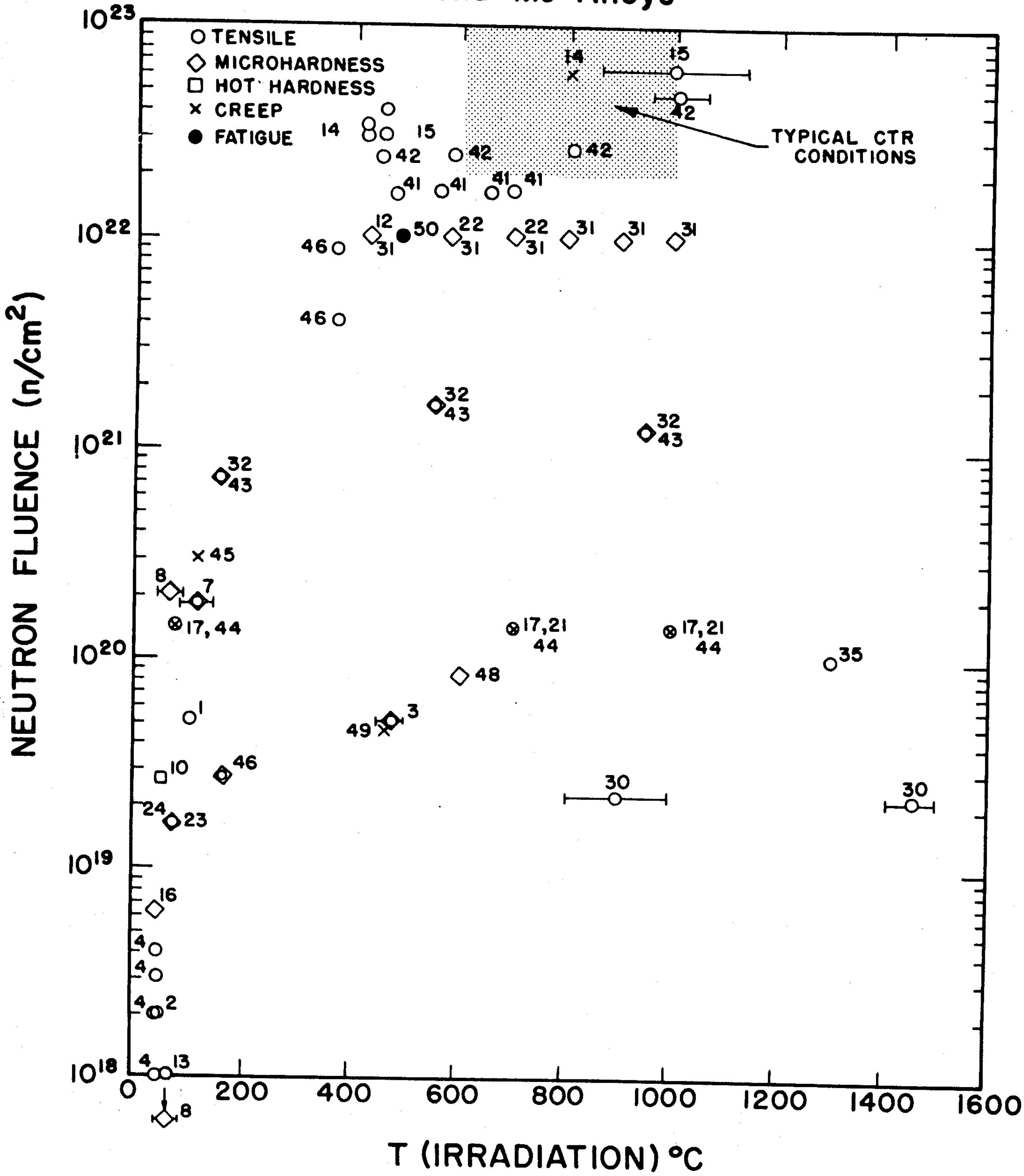
*An extra caution here should be made because even those samples which have suffered adequate displacement damage, have an inadequate amount of helium gas generated internally.

Figure 1
Mechanical Properties of Neutron Irradiated Mo and Mo Alloys



Key to numbering of mechanical property papers in Fig. 1, plotted by neutron fluence vs irradiation temperature. The number assigned to a point in the figure appears to the left and to its right is the author code given in the appendix for that paper.

1 - M.2.1	21 - M.11.4	41 - W.4.1
2 - J.2.1	22 - M.11.5	42 - B.7.2
3 - J.2.2	23 - H.2.2	43 - K.2.2
4 - V.4.1	24 - H.2.1	44 - M.11.7
5 - S.4.1	25 - O.2.1	45 - L.8.1
6 - W.2.1	26 - O.2.2	46 - O.4.1
7 - J.2.3	27 - P.6.1	47 - S.16.1
8 - I.2.4	28 - S.5.1	48 - S.17.1
9 - I.2.3	29 - T.3.1	49 - Z.1.1
10 - W.2.2	30 - N.3.1	50 - S.20.1
11 - M.11.2	31 - S.6.1	
12 - T.4.1	32 - K.2.1	
13 - M.11.3	33 - L.2.1	
14 - W.1.2	34 - L.7.1	
15 - W.1.3	35 - M.10.1	
16 - K.9.4	36 - B.2.1	
17 - K.6.2	37 - E.3.1	
18 - K.6.1	38 - I.4.1	
19 - L.3.1	39 - B.5.1	
20 - M.9.1	40 - A.4.1	

Much of the same observation can be made for Figure 2 as was made for Figure 1, that is, there is very little data available on the microstructure of Mo which has suffered sufficiently high displacement damage* at high temperature. There are a large number of data points at room temperature from 10^{19} to over 10^{22} n cm^{-2}. However, such irradiations occur below the vacancy migration temperature (~150-200°C) and therefore would give rather atypical microstructures. The large number of data points in the 400-1000°C and 10^{20} to 2×10^{21} n cm^{-2} range (~0.04 to 0.8 dpa) represent only 3 days to 2 months of exposure in a modestly irradiated (~1 MW/m^2) CTR first wall. It should be noted that the highest 14 MeV neutron irradiated specimen was bombarded at room temperature and at a fluence two orders of magnitude below the bottom of Figure 2!

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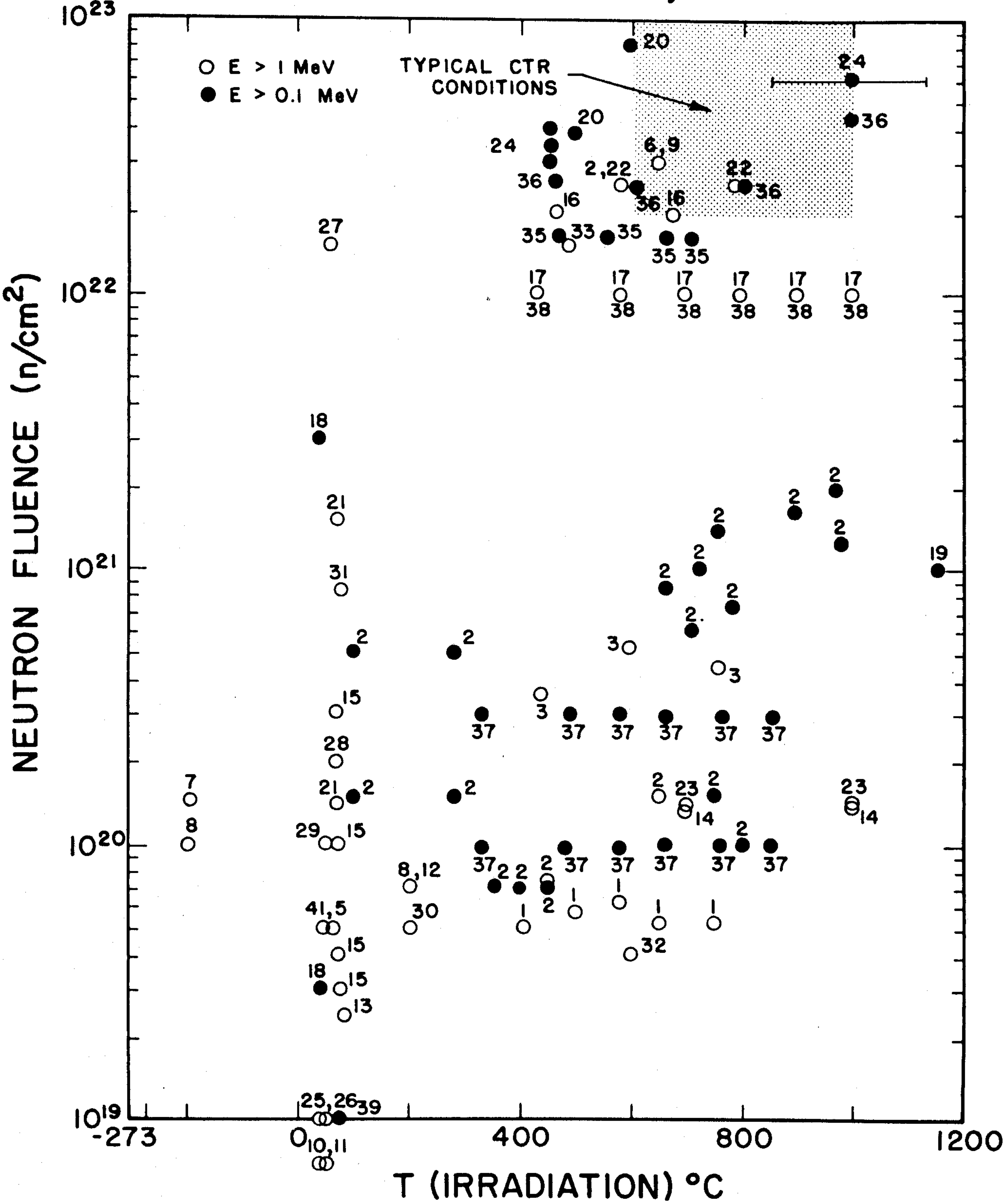
*Remember that such specimens contain approximately 1% of the helium that would be present after an equivalent amount of displacement damage.

Figure 2
Microstructure and TEM of Neutron Irradiated Mo and Mo Alloys



Key to numbering of microstructure and TEM papers in Fig. 2, plotted by neutron fluence vs. irradiation temperature. The number assigned to a point in the figure appears to the left and to its right is the author code given in the appendix for that paper.

1 - B.9.6
2 - B.9.8
3 - E.1.2
4 - E.4.10
5 - E.4.9

6 - E.5.6
7 - E.5.5
8 - E.5.4
9 - E.5.3
10 - K.9.2

11 - K.9.11
12 - M.1.5
13 - N.1.1
14 - R.2.3
15 - R.2.4

16 - S.1.1
17 - S.6.1
18 - K.7.3
19 - K.9.6
20 - P.9.1

21 - R.2.2
22 - W.1.1
23 - M.11.4
24 - W.1.3
25 - K.10.1

26 - M.5.1
27 - D.3.1
28 - E.5.2
29 - M.5.2
30 - E.5.1

31 - E.1.1
32 - B.9.4
33 - W.1.2
34 - B.17.1

35 - W.4.1
36 - B.17.2
37 - B.17.3
38 - L.9.1, M.11.8, S.6.4
39 - P.5.3

It is also worthwhile to examine the level of effort on ion bombarded specimens because that has been a recent and important aspect of radiation damage studies. Table V-1 lists the various studies which have been reported in the literature with respect to type and energy of bombarding ion, damage level, irradiation temperature, and type of analytical technique used. A few interesting observations about Table V-1 are:

. Almost two-thirds of the studies have been conducted with gaseous ions. The main objective here was to elucidate the surface blistering and gas re-emission problem. However, nitrogen ions have also been used to study void formation.

. The remaining heavy ion irradiations have been conducted with ions of energy ranging from approximately 3 to 19 MeV to a maximum dpa value of 53 dpa. The temperature region from 20°C to 1000°C has been covered but the major emphasis has been in the 900-1000°C region.

. Only one experiment has been conducted with Mo containing helium, either pre-injected or simultaneously injected. More work is needed to corraborate this result.

The general impression one gets from Table V-1 is that the investigation of Mo and its alloys has been spoty at best, with few in depth studies outside that of Stubbins (S.18.1).

Table V-1 - Summary of Ion Bombardment Experiments on Mo and Mo Alloys

Ion	Energy (MeV)	Dpa	Fluence	$T_{irradiation}$ (C)	Type of Study	Reference
H^+	38		$5\times10^{18}/cm^2$	80	TEM	B.14.1
H^+	.150			-90 to +115	Re-emission	B.1.1
H^+			$2\times10^{19} H/cm^2$	-170 to 1200	Surface Deformation	T.2.1
d^+	0.020		$\leq 1.9\times10^{17}/cm^2$	250		M.7.1
D^+	0.007-0.02		$1.8\text{-}2.4\times10^{17}$	-196	Re-emission	E.2.2
D^+	0.02		2×10^{17}	-196+27	Re-emission	E.2.3
He^{++}	.085		$1.5\times10^{7}/cm^2$	400-500	TEM	B.14.1
He^{++}	-	3×10^{-3}	10 appm	≤ 200	Fatigue	M.9.1
He^{++}	.30			400-1200	Re-emission	B.1.1
He^{++}			$4\times10^{18} He/cm^2$	-170 to 1200	Surface Deformation	T.2.1
He^{++}	.036		$1\times10^{17}/cm^2$	20	TEM	S.1.1
He^{++}			1.3 appm			W.1.2
He^{++}	0.007-0.080		$\leq 7\times10^{17}/cm^2$	50, and to 1500	Surface-SEM	E.2.1
He^+	0.055		$1\times10^{18}/cm^2$	-196	X-Ray	B.16.1
He^{++}	0.018		5×10^{17}	RT	TEM	E.4.17
N^+	2				TEM	E.4.12
N^+	2		$1\times10^{18}/cm^2$	950	TEM	E.4.7
N^+	2		$1\times10^{18}/cm^2$	600-800	TEM	E.4.6
N^+	2		$7\times10^{17}/cm^2$	870	TEM	E.4.8
N^+	2		$1\times10^{18}/cm^2$	60	TEM	E.4.9
N^+	2		$7\times10^{17}/cm^2$	870	TEM	E.5.3
Ne^+, Ar^+, Kr^+, Xe^+	25-1000(eV)		$\leq 3\times10^{17}/cm^2$		Surface-LEED	F.1.1
V^+	3.1	3-54	-	750-1500	TEM	S.18.1
Ni^{++}	5	-	-	1000	TEM	B.18.1
Ni, Se	5-8	0.47-400		525	TEM	K.9.9
Ni^{++}	5		$7\times10^{15}/cm^2$	650-1000	TEM	K.9.8
Ni^{++}	5	5		650-1000	TEM	K.9.7
Ni^{++}/He	5/0.2	2-53	$3\text{-}75\times10^{15}$	900	TEM	B.9.15
Ni^{++}	5	2-20	$5\text{-}50\times10^{15}$	900-1000	TEM	B.18.2

Ion	Energy (MeV)	Dpa	Fluence	$T_{irradiation}^{(c)}$	Type of Study	Reference
Cu^{++}	17-19	1-19		700-1000	TEM	L.10.1
Mo^{+}	0.06		$7.4x10^{11}$	20-535	TEM	E.6.1, E.6.
Mo^{+}	2.8	2-20	$10^{15}-10^{16}$	800-900	TEM	S.21.1
Ta^{+++}	7.5		$3x10^{16}/cm^2$	800-1000	TEM	B.9.9
Ta^{+++}	7.5	≤ 150	$3x10^{16}/cm^2$	900	TEM	B.9.7
Ta^{+++}	7.5	30		650-1000	TEM	K.9.7
Fission Fragments			$1.6x10^{10}$	80	TEM	H.3.1
Fission Fragments			$1x10^{15}/cm^2$	60-80	TEM	E.5.2

VI. Cross Reference

From Table VI-1, several points can be distinguished. Mechanical property data and TEM work have utilized irradiations primarily at or above room temperature whereas resisitivity measurements involved irradiations at or below room temperature. In the temperature range of interest for fusion reactors (600-1000°C) TEM work is divided nearly equally between neutron and heavy ion irradiations while mechanical property measurements have been made only for neutron irradiated materials. In the slightly lower temperature range of interest for fission reactors (400-700°C) most irradiations related to both TEM and mechanical property data generation have been made with neutrons although a few TEM studies have included heavy ion damage. The greater amount of heavy ion irradiations at the higher temperatures for TEM reflects the interest in molybdenum as a CTR material.

Of all neutron irradiations conducted, only one utilized a 14 MeV neutron source and that study documented microstructure observed in TEM. A limited number of surface sutdies have been conducted utilizing both hydrogen and helium over an extended range of temperatures.

Table VI-1

Cross Reference, Irradiation Temperature vs. Type of Irradiation for Each Property Investigated. Numbers in Chart Indicate Number of Investigations for the Given Set of Conditions.

		0-100K	100-273K	0-50C	50-200C	200-450C	450-700C	700-1000C	>1000°C
TEM	TOTAL	5	-	23	26	16	42	36	7
	n	5	-	18	23	12	36	22	5
	P			e-1	1	1	1		
	α			1	1	1	1	1	
	HI	-	-	3	5	2	7	16	2
RESISTIVITY	TOTAL	29	-	14	14	1	1	1	-
	n	11	-	9	13	1	1	1	-
	e^-	20	-	4α-1	1	1	-	-	-
MECH. PROP.	TOTAL	2	-	14	21	4	14	10	2
	n	2	-	13	20	4	13	10	2
	α	-	-	e-1	1	-	2	1	-
X RAY	TOTAL	-	-	11	3	1	-	-	-
	n	-	-	10	3	1	-	-	-
	α	-	-	1	-	-	-	-	-
SURFACE	TOTAL		1		1	2	3	3	3
	P	-	1	1	1	1	2	2	2
	α		1	1	1	2	3	3	3
	HI								
STORED ENERGY	TOTAL	-	-	2	-	-	-	-	-
	n	-	-	2	-	-	-	-	-
OVERALL TOTAL		37	1	67	66	24	60	50	12

VII. Conclusions

Reviewing the information in sections III thru IV, we come to the following general conclusions:

- Molybdenum and a few of its alloys have been studied for the past 20 years with various bombarding particles, but there has been very little in the way of a coordinated effort either by national or international organizations.

- The emphasis has shifted from one of studying low temperature mechanical properties to the present high temperature heavy ion and neutron effects. However, there are no in-situ mechanical property measurements taking place at any temperature and what post irradiation work that is being conducted, is usually at temperatures and fluences far below those characteristic of fission or fusion reactors. The understanding of the damage caused at high temperature by neutrons is particularly lacking at this time.

- There is essentially no 14 MeV data available (except for one 10^{17} n cm^{-2}, room temperature study) and no basis on which to predict fusion reactor behavior from fission reactor data.

- Essentially only one alloy, TZM, has been investigated with respect to both neutron and heavy ion induced damage. The data are sparse and there is no basis for extrapolation between the two types of irradiation.

- Very little work has been conducted on the effects of the environment (i.e. Li,Na, He with oxygen impurities, etc.) on the behavior of neutron irradiated Mo at high temperatures.

- The low temperature behavior of pure Mo appears to be fairly well understood but very little is known about the effect of high temperature irradiation (i.e. voids, loops) on the electrical resistivity of Mo. Such information may be needed for fusion reactors, especially if Mo is used for wave guides in RF heaters.

. About half of the reported work on irradiated Mo and its alloys comes from the U.S. with the U.K. and U.S.S.R. dominating the remaining half. The Soviet Union has concentrated mainly on neutron and electron damage while workers in the U.K. have focused most of their attention on neutron and light ion irradiated Mo. The U.S. has reported the bulk of the Heavy Ion Work and concentrated on TEM.

On the basis of our review, we would estimate that roughly a ~100 man year effort has been devoted to the study of irradiated Mo over the past 20 years. The lack of any coordination of the direction, materials, or irradiation conditions has resulted in a very uncertain picture of just what does happen to Mo during bombardment. It would be very difficult to extrapolate what is now known about Mo and its alloys to anticipated fission or fusion conditions. It is hoped that the readers of this document can use it to their advantage in focusing on those irradiation conditions which have not been examined to the present time.

Appendix - A

Bibliography of Radiation Damage in Molybdenum and its Alloys*

*See Chapter II for explanation of use

CODE	AUTHOR(S)	TITLE	REFERENCE
A.1.1	J. Adam and D. G. Martin	MEASUREMENTS OF UNIT CELL AND PHYSICAL DIMENSION CHANGES IN IRRADIATED MOLYBDENUM	Phil. Mag., 3, 1329 (1958)
A.2.1	H. B. Afman, J. H. Mooy and H. Rademaker	RADIATION DAMAGE AND STAGE III ANNEALING OF MOLYBDENUM	Scripta Met. 4, 545 (1970)
A.2.2	H. B. Afman	A METHOD FOR THE DETERMINATION OF THE ACTIVATION ENERGY OF RECOVERY PROCESSES AS APPLIED TO ELECTRON-IRRADIATED MOLYBDENUM	phys. stat. sol. (a) 4, 427 (1971)
A.2.3	H. B. Afman	STAGE I RECOVERY OF MOLYBDENUM IRRADIATED AT 4.2°K WITH ELECTRONS OF DIFFERENT ENERGIES	phys. stat. sol. (a) 11, 705 (1972)
A.2.4	H. B. Afman	THE MECHANISM OF STAGE III RECOVERY OF ELECTRON IRRADIATED MOLYBDENUM	phys. stat. sol. (a) 13, 623 (1972)
A.3.1	O. P. Agnihotri	ACTIVATION ENERGY FOR THE MOTION OF DISLOCATION IN MOLYBDENUM	phys. stat. sol. (a) 7, K33 (1971)
A.4.1	R. A. Ayres and D. F. Stein	REPLY TO 'ON THE FRACTURE ENERGY OF MOLYBDENUM'	Scripta Met. 6, 1043 (1972)
A.2.5	H. B. Afman	THE RECOVERY OF MOLYBDENUM IRRADIATED AT LOW TEMPERATURE WITH ELECTRONS OF DIFFERENT ENERGIES	pp. 19-21 in Proceedings of the Mol Conference on Defects in Refractory Metals, edited by R. De Batist, J. Nihoul, and L. Stals (1972).
A.5.1	Y. Adda	REPORT ON THE CEA PROGRAM OF INVESTIGATIONS OF RADIATION-INDUCED CAVITIES IN METALS: PRESENTATION OF SOME RESULTS	pp 31-83, Albany Conference June 9-1[illegible] 1971 on Radiation-Induced Voids in Metals.
A.6.1	R. J. Arsenault	INVESTIGATION OF IRRADIATION STRENGTHENING OF B.C.C. METLAS AND SOLID SOLUTIONS	Progress Report for January 1972 Report ORO-3612-9.

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A.7.1	G. Antesberger and K. Sonneberg	DOSE DEPENDENCE AND INFLUENCE OF RADIATION DOPING-DEFECTS ON THE ANNEALING OF MO IN STAGE I	Intl. Conf. on the Properties of Atomic Defects in Metals, October 18-22, 1976 Argonne National Laboratory
A.7.2.	G. Antesberger, H. Schroeder, K. Sonneberg and U. Dedek	DETERMINATION OF SINGLE INTERSTITIAL AND SINGLE VACANCY MIGRATION TEMPERATURE BY ELECTRON DAMAGE RATE EXPERIMENTS AT DIFFERENT TEMPERATURES	Proceedings of the Intl. Conf. on Fundamental Aspects of Radiation Damage in Metals, p. 575-581, Gatlinburg, Tenn., October 6-10, 1975.

CODE	AUTHOR(S)	TITLE	REFERENCE
B.1.1	W. Bauer and G. J. Thomas	HELLIUM AND HYDROGEN RE-EMISSION DURING IMPLANTATION OF MOLYBDENUM, VANADIUM, AND STAINLESS STEEL	J. Nucl. Mat. 53, 127 (1974)
B.1.2	W. Bauer and G. Thomas	HELIUM RE-EMISSION AND SURFACE DEFORMATION IN NIOBIUM, VANADIUM AND MOLYBDENUM	Defect and Defect Clusters in B.C.C. Metals and Their Alloys, Nuclear Metallurgy 18, 255 (1973), R. J. Arsenault (Ed.)
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B.4.1	M. Biget, P. Najda, A. Lucasson and P. Lucasson	A STUDY OF ELECTRON IRRADIATION DAMAGE IN SINGLE CRYSTALS OF MOLYBDENUM	Rad. Effects 21, 229 (1974)
B.5.1	J. C. Bilello and G. Caglioti	ON THE FRACTURE ENERGY OF MOLYBDENUM	Scripta Met. 6, 1041 (1972)
B.6.1	S. T. Borimskaya, L. N. Larikov, and N. P. Plotnikov	SOFTENING OF NEUTRON BOMBARDED Al, Cu, AND Mo SINGLE CRYSTALS BY ANNEALING	Fiz. Metal Metalloved, 21, 797 (1966)
B.7.1	S. Blow	SOME FEATURES OF THE BEHAVIOR OF STRUCTURAL MATERIALS IN A POSSIBLE FUSION REACTOR BLANKET	Brit. Nucl. Energy Soc. 11, 371-376 (1972)
B.8.1	S. S. Brenner and D. N. Seidman	FIELD ION MICROSCOPE OBSERVATION OF VOIDS IN NEUTRON IRRADIATED MOLYBDENUM	Rad. Effects 24, 73 (1975)
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CODE	AUTHOR(S)	TITLE	REFERENCE
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B.9.8	J. L. Brimhall, E. P. Simonen, and H. E. Kissinger	FLUENCE AND TEMPERATURE DEPENDENCE OF SWELLING IN IRRADIATED MOLYBDENUM	J. Nucl. Mat. 48, 339 (1973)
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