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The role of stress state on the fracture toughness and toughening mechanisms of wrought molybdenum and molybdenum alloys

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ABSTRACT

Rolling unalloyed molybdenum, molybdenum alloys, and Oxide Dispersion Strengthened (ODS) molybdenum into sheet produces microstructures with elongated, pancake shaped grains that can result in anisotropic mechanical properties. In this work, unalloyed molybdenum, molybdenum alloys, and ODS molybdenum are rolled to thinner sheet and then subjected to tensile and fracture toughness testing and examination of the toughening mechanism. The ductile laminate toughening mechanism observed for wrought molybdenum results from a lower toughness in the short-transverse orientation that leads to separation of the layers of sheet-like grains of the microstructure along the grain boundaries in the regions of stress concentration. This splitting of the microstructure results in the formation of ligaments of grains, or non-constrained laminates, that are stretched to failure under a plane stress-state with large amounts of plastic deformation. The thinner specimens exhibit higher fracture toughness values and lower Ductile to Brittle Transition Temperature (DBTT) values than for thicker specimens machined from thicker starting material from the same alloy. The lower constraint of the thinner specimens tested in this work results in higher toughness and lower DBTT values. The finer grain size, finer precipitate size, and state of plane stress achieved for the thinner sheet specimens appears to enhance the ductile laminate toughening to result in higher fracture toughness and lower DBTT values. The detrimental effect of crack initiation from brittle carbides, oxides, and second phases is also observed to be diminished under a stress-state of plane stress.

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1. Introduction

Molybdenum is a refractory metal with a relatively high melting temperature of 2610 °C that is used in applications where high-temperature strength and creep resistance are needed and its poor oxidation resistance may not degrade performance [1–6]. Molybdenum has a body-centered cubic (bcc) structure and, like many bcc metals, exhibits a Ductile to Brittle Transition Temperature (DBTT) that is sensitive to interstitial content, impurities and alloying content, grain size, grain boundary character, microstructure, and stress-state [7–29]. Large amounts of ductility are generally measured for molybdenum and its alloys near a homologous temperature of 0.1 (≈17 °C) or lower to define the DBTT using smooth tensile or bend specimens. Use of fracture toughness testing results in plane strain conditions and higher DBTT values of 50 °C to 200 °C have been measured [16–19] for commercially available wrought unalloyed Low Carbon Arc Cast (LCAC) molybdenum and a wrought molybdenum alloy with 0.5% Ti and 0.1% Zr additions by weight (TZM). The finer grain size of wrought Oxide Dispersion Strengthen (ODS) molybdenum results in higher fracture toughness values at lower temperatures and a lower DBTT [16–19].

Wrought molybdenum and molybdenum alloys generally have microstructures consisting of elongated, pancake shaped grains and a general anisotropy in fracture toughness exists [19]. The lower fracture toughness in the short-transverse direction for wrought molybdenum alloys results in splitting along the grain boundaries and interfaces that reduces the constraint on the grain interiors so they can deform more freely and absorb plastic energy as the crack propagates across the sample [16–19,23]. This ductile laminate form of thin sheet toughening observed for wrought molybdenum alloys [16–19] has also been observed for Al-based alloys [30–32], steels [33], lamellar TiAl-based alloys [34], and metal-matrix composites [35], and molybdenum produced by sintering [23]. The efforts to measure the fracture toughness of molybdenum-base alloys are relatively recent [14–23,36–50], and the role of microstructural variables and stress state on the toughening mechanisms have not been well studied.

Since the size of the ligaments resulting in toughening are related to the thickness of the sheet-like grains, there is generally a strong correlation between grain size and fracture toughness DBTT with higher toughness values at lower temperatures and a lower DBTT observed for materials with a finer grain size [16–19]. The

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Table 1

Summary of previously reported chemical analysis data for LCAC, TZM, and ODS molybdenum alloys as obtained from H.C. Starck [16–19], HP-LCAC [52], and Mo–Al–B (Alloy 6) and Mo–Zr–B (Alloy 5) [53].

Material/Lot #	C	O	N	Ti	Zr	Fe	Ni	Si	La	Al	Ca	Cr	Cu	Other
ODS molybdenum/ingot #382 Heat # LA22963	10	NA	NA	<10	NA	74	12	24	1.6 wt%	21	320	24	10	<10 Mg<10 Mn<10 Pb<10 Sn
TZM molybdenum/ingot 61722B Lot # TZM24080	223	17	9	5000	1140	<10	<10	<10	NA	NA	NA	NA	NA	NA
LCAC molybdenum/ingot 50823 Lot # C24728	50	11	4	NA	NA	<10	<10	<10	NA	NA	NA	NA	NA	NA
High-purity LCAC (HP-LCAC) Molybdenum/heat # M1529	20	4	3	5	1	20	3	3	0.6	1	0.07	3	0.4	120 W 14 Nb
Alloy 6 (Mo–Al–B)	73	14	<1	NA	31	3	<1	2	NA	6	NA	NA	<1	21 W 4 Nb 2 Ir 2 Re
Alloy 5 (Mo–Zr–B)	81	9	<1	NA	1700	3	<1	6	NA	<1	NA	NA	<1	32 W 6 Nb 4 Ir 2 Re 13 Hf

Note: The compositions for LCAC and TZM are within the ASTM B386-91 specification for these alloys. ASTM B386 – 365 for arc-cast LCAC molybdenum and ASTM B386 – 363 for TZM molybdenum [54]. NA = not available. Values of 1 ppm or less are not listed.

Table 2

Summary of grain size measurements for wrought LCAC, TZM, and ODS molybdenum in the starting plate and sheet form, HP-LCAC sheet, and Mo–Al–B (Alloy 6) and Mo–Zr–B (Alloy 5) sheet [16–19,52,53].

Alloy	Grain diameter [μm]				Grain length [μm]			
	Average	Standard deviation	Maximum	Minimum	Average	Standard deviation	Maximum	Minimum
LCAC plate – LSR	14.0	10.4	70	1	340	138	820	13
LCAC plate – TSR	15.2	10.5	50	2	255	113	700	70
LCAC 0.86–0.89 mm sheet – LAW	9.8	6.4	30	2	452	219	1050	160
LCAC 0.86–0.89 mm sheet – LSR	8.9	6.5	34	2	412	191	820	85
LCAC 0.61–0.74 mm sheet – LAW	6.8	4.0	20	1	423	249	1000	50
LCAC 0.61–0.74 mm sheet – LSR	7.9	5.1	23	1	411	245	1050	60
ODS plate – LSR	1.4	0.7	4	0.5	29.0	16.2	75	4
ODS plate – TSR	2.0	1.1	7	0.5	13.6	6.6	30	2
ODS 0.86–0.89 mm sheet – LSR	1.1	0.6	2.1	0.5	91	47	185	30
ODS 0.61–0.74 mm sheet – LAW	0.9	0.4	2.5	0.4	221	159	550	25
ODS 0.61–0.74 mm sheet – LSR	0.8	0.4	2.5	0.3	285	159	600	30
TZM Plate – LSR	3.9	2.5	15	1	273	105	680	70
TZM plate – TSR	6.1	3.8	22	2	132	69	580	20
TZM 0.86–0.89 mm sheet – LSR	1.8	1.0	4.0	0.5	236	107	430	40
TZM 0.61–0.74 mm sheet – LAW	1.5	0.8	3.5	0.4	318	149	570	35
TZM 0.61–0.74 mm sheet – LSR	1.4	0.7	4.0	0.4	322	155	650	70
HP-LCAC-1 sheet – LAW	1.3	0.9	5.0	0.5	465	231	880	40
HP-LCAC-1 sheet – LSR	1.2	0.7	3.1	0.5	494	237	900	80
HP-LCAC-1 sheet – LR	26	13	60	3	44	35	160	8
HP-LCAC-2 sheet – LAW	1.3	0.9	5.0	0.4	452	317	1100	45
HP-LCAC-2 sheet – LSR	1.3	0.8	4.0	0.4	383	181	730	140
Mo–Al–B (Alloy 6) sheet – LAW	1.2	0.6	3.0	0.4	262	109	550	105
Mo–Al–B (Alloy 6) sheet – LSR	1.1	0.6	3.5	0.3	252	123	550	20
Mo–Zr–B (Alloy 5) sheet – LAW	1.7	0.9	5.0	0.5	215	101	430	65

Table 3

Summary of loads and conditions used for the pre-cracking of the sheet specimens used for toughness testing. All pre-cracking was performed at a load ratio (*R*) of 0.1.

Alloy/orientation/condition	Nominal base area [mm^2]	Range of max stress used [MPa]	Stress range to initiate [MPa]	Total pre-crack cycles [thousands of cycles]	Crack length [mm]
LCAC/longitudinal/LSR	13.89	270–230	190–210	100–250	4.38–4.73
LCAC/transverse/TSR	16.18	210–220	120–140	210–300	4.86–4.98
ODS/longitudinal/LSR	15.24	260–330	230–250	170–921	3.78–6.36
ODS/transverse/TSR	16.71	160–168	80–100	61–230	5.48–7.36
TZM/longitudinal/LSR	17.18	250–300	240–260	80–270	4.22–5.14
HP-LCAC-1/LONGITUDINAL/LSR	15.98	228–250	190–210	120–160	4.40–4.78
HP-LCAC-2/longitudinal/LAW	15.86	240–280	200–220	120–200	4.24–5.84
Mo–Al–B (Alloy 6)/longitudinal/LAW	21.44	250–370	230–250	73.7–250	4.42–5.92
Mo–Al–B (Alloy 6)/transverse/TAW	22.07	200–210	170–190	80–220	5.06–6.04
Mo–Zr–B (Alloy 5)/longitudinal/LAW	27.87	220–240	190–210	125–180	4.44–5.36