

FRACTURE TOUGHNESS OF HIGH MELTING POINT MATERIALS

D.L.Chen, B.Weiss, R. Stickler - Universität Wien
M.Witwer, G. Leichtfried, H.Hödl - Metallwerk Plansee
Austria

ABSTRACT

Little information has been published on the fracture toughness properties of high-melting point metals and alloys which are mainly produced in sheet or rod form requiring special testing procedures and sample geometries. Modifications of the recommendation according to ASTM E 399 and E-740 were required in order to determine fracture toughness data at room temperature for rods of Mo and Mo-alloys produced in various sizes by pm-techniques. For larger diameter bars, disk-shaped compact tension specimens were tested, for smaller diameter rods, surface-cracked tension specimens had to be evaluated. In the latter test, during the required fatigue precracking the crack growth can be monitored to determine threshold values for fatigue crack growth prior to the fracture toughness test. The applicability, advantages and limitations of both test procedures will be discussed.

KEYWORDS: mechanical properties, fracture toughness, molybdenum, molybdenum alloys

1. INTRODUCTION:

It is well recognized that many engineering components fail in service by fracturing due to the growth of flaws. Traditional tensile tests cannot accurately predict the fracture resistance of a material (1); a more appropriate criterion is the fracture toughness as determined by standard test procedures.

The conventional plane-strain fracture toughness test according to ASTM E 399 (2) requires a minimum specimen thickness to ensure plane-strain conditions to prevail along a major portion of the crack front during the test. This criterion makes testing of many materials and product forms impractical. In addition, this ASTM procedure requires fatigue precracking by tension cycling to achieve a sharp crack tip which for many high-strength low-ductility materials is difficult to realize.

Several experimental procedures have been described in the literature permitting the use of smaller specimens which require special evaluation methods but may not yield true plane-strain fracture toughness values. However, it has been stated that, although valid K_{Ic} values are certainly very useful one should keep in mind that for each application a toughness value should be used that is relevant to that particular geometry and thickness (3). One should not strictly adhere to a valid K_{Ic} value irrelevant to the practical application.

Technical structures often contain semi-elliptical surface cracks for which it is known that the K_{IC} value falls between the stress intensity values at the tips of the crack at the surface (crack length "2c") and at the deepest point (crack depth "a"). A test procedure using specimens with such type of surface cracks is recommended in ASTM E 740 (4). It could be demonstrated in a previous study (5) for an isotropic high-strength pm Al-alloy that both test methods yielded practically identical K_{IC} -values (K_{IC} from disk-shaped tension specimens 7.3 MPa.m^{1/2}, K_{IC} from surface-cracked tension specimens 7.8 MPa.m^{1/2}).

Little information has been published on the fracture toughness properties of high-melting point metals. Only rarely these materials are available in product forms which permit the preparation of standard fracture toughness specimens. These alloys are mostly produced in sheet or rod form which require modified testing procedures and sample geometries.

Marschall and Holden (6) determined the fracture toughness behavior of Mo and Mo-alloys, W, Ta and Nb by testing center-cracked tension specimens of sheet metals (1.25 mm thick). More recently the fracture toughness of W-2%ThO₂ has been deduced from 3-point bend tests of notched miniature specimens (7).

The present investigation was initiated to determine fracture toughness data of bars and rods of Mo and Mo-alloys by test procedures similar to those recommended by ASTM E 399-83 (2) and ASTM E 740-88 (4), modified to permit testing of these materials in product forms of large diameter bars and thin rods.

2. SPECIMEN MATERIALS:

As specimen materials Mo and a Mo-Ti-Zr alloy (TZM) produced by conventional powder metallurgical processing routes were used. To prepare disk-shaped compact tension specimens (according to Fig. A6.1 in ASTM E-399-83) hot-forged and subsequently air-cooled bars of Mo and TZM with diameters of about 60mm were selected. The specimens were machined out of circular sections cut normal to the axis of the bars with deformed microstructure of grains elongated in the forging direction.

To evaluate the properties of swaged-rod product with diameters of less than 14mm, dumbbell-shaped specimens with a rectangular gauge section were machined of Mo and TZM either in the as-swaged (deformed microstructure) or in a recrystallized condition (equiaxed fine-grain microstructure). Composition and properties of these materials are listed in Table 1.

Table 1: Properties of specimen materials:

Mo: Chemical Composition (ppm): 13 O₂, 10C

Mechanical Properties	R _m [MPa]	R _{eL} [MPa]	R _{eH} [MPa]	R _{n0.2} [MPa]	A [%]	HV10
Swaged rod, 12 mm Ø, as-swaged (1070 °C/air cool)	659	655	749	-	40	234
Swaged rod, 12 mm Ø, recrystallized (1400 °C/2 h)	400	-	-	361	50	183
Forged bar, 54 mm Ø, properties in axial direction	480	-	-	420	10	247

TZM: Chemical Composition (ppm): 170 O₂, 305 C, 4700 Ti, 820 Zr

Mechanical Properties	R _m [MPa]	R _{eL} [MPa]	R _{eH} [MPa]	R _{n0.2} [MPa]	A [%]	HV10
Rod, 12 mm Ø, recrystallized (1500 °C/1 h)	581	443	536	-	30	210
Bar, 54 mm Ø, as-forged, properties in axial direction	720	-	-	700	2	270

3. EXPERIMENTAL DETAILS:

3.1 Plane strain fracture toughness testing of disk-shaped compact tension specimens (DCT-specimens similar to ASTM E 399-83)

According to ASTM E 399 the recording of P-COD curves is required. The size of the specimens depends on the mechanical properties of the specimen material, i.e., fracture toughness and yield strength (K_{IC} , S_{YS}), the specimen thickness ("B") and the starting notch plus precrack ("a") must obey the relation

$$(B,a) > 2.5 \times (K_{IC}/S_{YS})^2,$$

with the width W of the specimen $2 < W/B < 4$.

According to this ASTM recommendation, a starter notch with the length of the fatigue crack for a straight-through notch being 2.5 % of W or at least 1.3 mm was used. The tensile test to obtain the maximum load should be carried out under a loading rate of 0.34-1.7 kN/s. The ASTM standard defines loads of P_Q (load at intersection with 95 %-elastic slope secant) and P_{max} (maximum load during test) which must obey the relation $P_{max}/P_Q < 1.10$ (first instability close enough to the final catastrophic instability) in order to yield valid K_{IC} values.

Specimen geometry and dimensions

The geometry and dimensions of the DCT-specimens is shown in Fig.1a. Preliminary tests revealed that Chevron-notches do not facilitate the initiation of a fatigue crack; therefore, specimens were prepared with a straight-through notch. In these specimens the crack plane was oriented in the radial direction parallel to the forging direction.

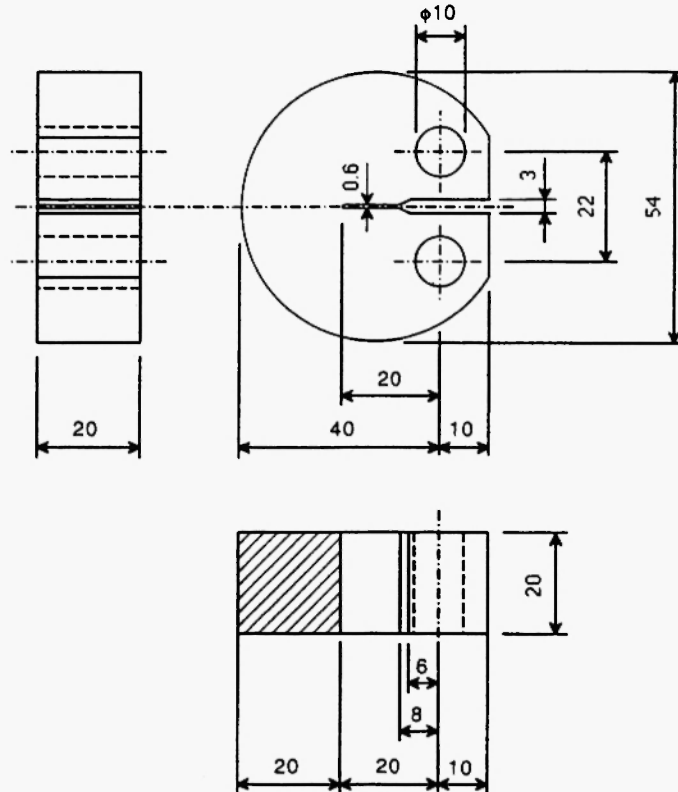


Fig. 1a: Dimensions of disk shaped compact tension specimen (ASTM E-399)

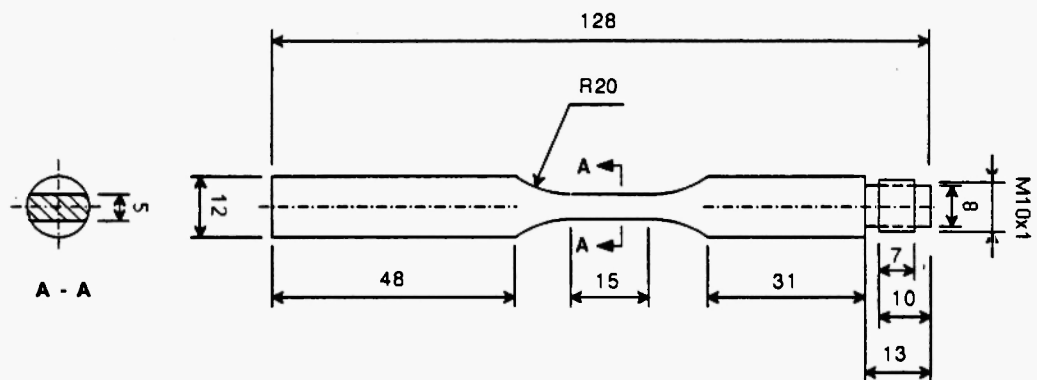


Fig. 1b: Dimensions of surface crack tension specimen (similar to ASTM E-740)

In comparison with the ASTM standard the specimen dimensions all comply with the requested values:

Parameter:	Required according to ASTM E-399		Present investigation	
	Mo	TZM	Mo	TZM
$(B, a) > 2.5 / (K_{IC} / S_{YS})^2$	0.9 mm	1.9 mm	20 mm	20 mm
$2 < W/B < 4$	2-4	2-4	2	2

Test procedure:

Attempts to initiate fatigue cracks under cyclic loading at stress ratio $R = 0.1$ resulted in a spontaneous failure of the specimen without an indication of the formation of a fatigue crack. Following the experience with other brittle materials (8, 9), experiments were carried out to initiate fatigue cracks under compressive cycling ($R = 0.1$ in compression). A fatigue crack of approx. 0.1 mm length could easily be produced at relatively low loading amplitudes (less than 8 kN compressive mean load and 6 kN cyclic load for 10^4 loading cycles). After fatigue precracking the specimens were fractured in tension at the recommended loading rate and the load/crack-opening-displacement (P-COD) curves were recorded (the COD signal was obtained from a strain gauge applied across the notch near the fatigue precrack). The stress intensity factor was calculated according to the following equation,

$$K_I = P / (t \cdot W^{1/2}) \times f_I$$

with f_I taken from published tables (10) for the observed ratios of crack length to specimen width.

3.2 Fracture toughness testing with surface-crack tension specimens (SCT-specimens similar to ASTM E-740-88)

The test procedure according to ASTM E 740 recommends the use of flat specimens with a surface starter notch introduced by electro discharge machining and a fatigue crack of surface length $2c$ introduced by fatigue loading. The specimen dimensions should obey $W > 10c$, $L > 2W$, $c > 1.3$ mm, with K_{max}/E during fatigue cracking not to exceed $0.0003 \text{ m}^{1/2}$ at a stress ratio $R < 0.1$. The subsequent fracture tests should be carried out under a loading rate of less than 690 MPa/min. For the calculation of the fracture toughness the fatigue crack size and shape has to be taken into account.

As recommended by the ASTM standard, the equation derived by Newman & Raju (12) should be used for the calculation of the fracture toughness K_{IC} or an equivalent (nominal) fracture toughness K_{Ie} for this type of crack,

$$K_{IC}, K_{Ie} = S_{max} (\pi \times a/q)^{1/2} \times F$$

The parameters q and F are two geometric shape factors depending on surface crack shape (a/c), crack length ($2c$), specimen thickness (t), and specimen width ($2b$), (12).

These shape factors must be determined by fractographic evaluation of the fracture surface after breaking the specimen.

For low-toughness materials where stable crack growth prior to failure is absent, the fracture mechanics characterization based on the maximum load in the P-COD curve is appropriate (4). For tough materials such a characterization has been described as questionable if significant stable crack growth occurs prior to failure. Limited experiments have indicated (4) that the surface-crack-fracture toughness K_{Ic} will be reasonably constant, provided that stable subcritical crack growth is not significant and that the ligament depth (B-a) is greater than $0.5(K_{Ic}/S_{YS})^2$.

Specimen geometry and dimension

The specimen dimensions used in these tests are shown in Fig. 1b. Since precracking was performed under resonance loading, the specimen length is dictated by the resonance conditions of the specimen. In these specimens the crack plane was oriented in the radial direction normal to the forging direction.

In addition to the specimens prepared from the swaged 12mm diam. rods, identical specimens were also machined from the 54mm diam. forged bars with the longitudinal axis parallel to the forging direction.

The specimens obey the specified requirements:

Parameter	Required according to (12)		Actual	
	Mo	TZM	Mo	TZM
W/2c	2	2	2-2.5	2-2.5
K_{max}/E	3×10^{-4}	3×10^{-4}	$< 4 \times 10^{-5}$	$< 4 \times 10^{-5}$
L/W	2	2	2	2
R	<0.1		-1	

Test procedure:

To introduce the required fatigue precrack, the specimen was subjected to cyclic loading at $R = -1$ in a resonance test equipment operated at 20 kHz (11). A traveling light microscope permitted the continuous monitoring of the starter notch to reveal fatigue crack initiation and to follow with high resolution the crack growth. This method permits continuous monitoring of the fatigue crack growth and the determination of fatigue threshold values (Fig.2) prior to the fracture toughness test. For the latter test a strain gauge to monitor COD was applied across the fatigue crack. The specimen was mounted in a tensile test machine and fractured in tension at the recommended loading rate (650 MPa/min) with the P-COD curve recorded. After fracture of the specimen, the fracture surface was examined by light or scanning electron microscopy to determine the crack size and shape required for the K_{Ic} computation.

Test results from specimens which showed subcritical crack growth were discarded.

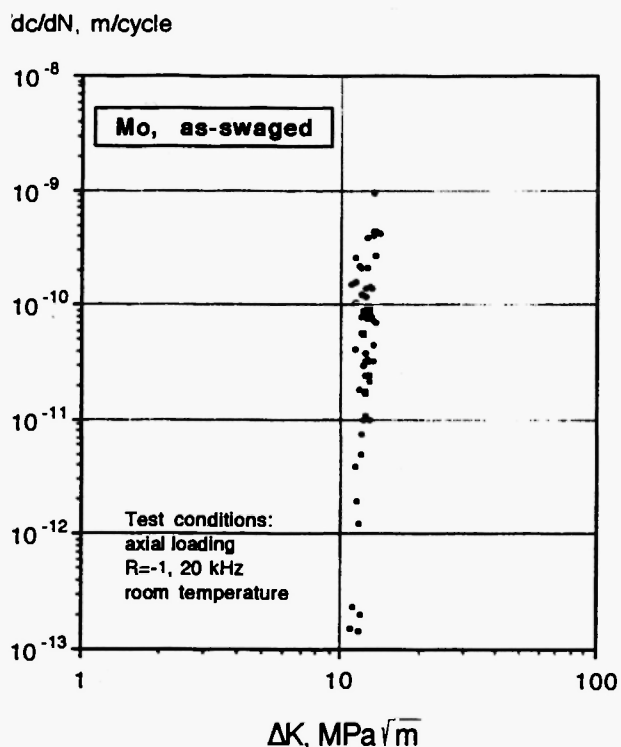


Fig. 2: Fatigue crack growth curve and threshold value for swaged Mo rods (12 mm \varnothing)

4. SUMMARY OF TEST RESULTS AND COMMENTS

The test results, averaged from 2-8 duplicate tests, are listed in the following.

- (i) Fracture toughness of forged bars of Mo and TZM (54 mm \varnothing), disk-shaped compact tension specimens, room temperature

Mo: $K_{IC} = 8 \pm 5\% \text{ MPa}\cdot\text{m}^{1/2}$

TZM: $K_{IC} = 19 \pm 5\% \text{ MPa}\cdot\text{m}^{1/2}$
- (ii) Fracture toughness of surface crack tension specimens (12 mm \varnothing) prepared from forged bars of Mo and TZM (54 mm \varnothing) and tested at room temperature; the values have been calculated for the deepest point of the semielliptical crack (at 90° to the surface, (12))

Mo: $K_{II} = 17 \text{ MPa}\cdot\text{m}^{1/2}$

$K_{Ie} = 40 \pm 10\% \text{ MPa}\cdot\text{m}^{1/2}$

TZM: $K_{IC} = 17 \pm 10\% \text{ MPa}\cdot\text{m}^{1/2}$

- (iii) Fracture toughness of swaged rods of Mo and TZM (diameter 12mm) tested at surface-crack tension specimens at room temperature, the values have been calculated for the deepest point of the semielliptical crack (at 90° to the surface, (12))

Mo: as swaged:

$$dK_{th,eff} = 11 \pm 10 \% \text{ MPa.m}^{1/2}$$

$$K_{Ie} = 17 \pm 11 \% \text{ MPa.m}^{1/2} \text{ (Fig.3a)}$$

recrystallized:

$$dK_{th,eff} = 11 \pm 10 \% \text{ MPa.m}^{1/2}$$

$$K_{Ie} = 18 \pm 10 \% \text{ MPa.m}^{1/2}$$

TZM: recrystallized:

$$K_{Ie} = 31 \pm 7 \% \text{ MPa.m}^{1/2} \text{ (Fig.3b)}$$

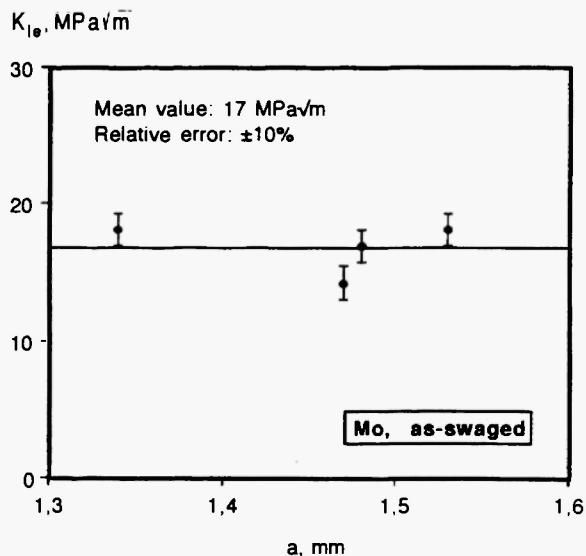


Fig. 3a: Influence of crack depth on K_{Ie} values for surface crack tension specimens, Mo as-swaged

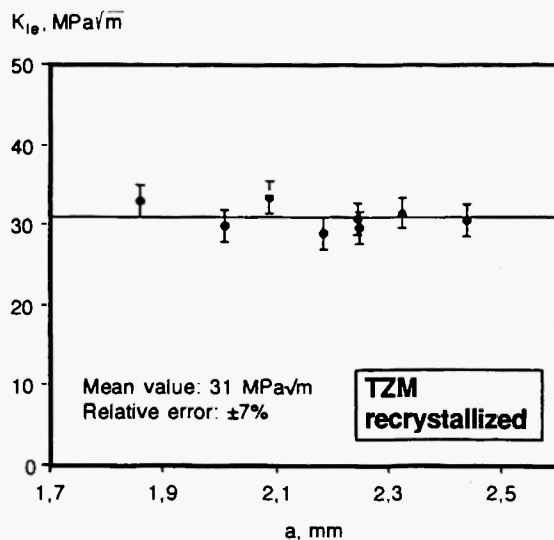


Fig. 3b: Influence of crack depth on K_{Ie} values for surface crack tension specimens, TZM recrystallized

The experimental results show that the evaluation of fracture toughness properties of high-melting point metals and alloys can be realized by means of the described test methods for low-toughness materials. If the material to be evaluated is available in large dimensions, disk-shaped compact tension specimens can be tested, provided the fatigue precracking is carried out by compressive cycling. The surface-crack tension test procedure offers the advantages of simple specimen preparation, short experimental times for fatigue precracking at low amplitudes even of brittle materials and its applicability to test rod-shaped specimen of diameters less than 12mm. Testing of sheet metals appears feasible. Previous experiments with a pm-Al alloy have shown that both test methods yield identical fracture toughness data for a homogeneous material.

The fracture toughness tests on the forged TZM materials indicated that no or only little stable crack growth occurred during fracturing. Therefore, the K_{IC} values are considered as valid. The influence of the orientation of the crack plane relative to the rolling direction appears negligible. The recrystallized TZM exhibited a small amount of stable crack growth prior to fracturing, therefore, in agreement with the standard recommendation (4) the data are given as "equivalent fracture toughness", K_{Ie} . The higher ductility of this material is expressed also in the higher fracture toughness value.

For swaged rods and recrystallized rods (12 mm Ø), tested as surface crack tension specimens, as well as for the forged bar, tested as disk compact tension specimen, negligible subcritical crack growth occurred, and the fracture toughness values appear valid. However, in the surface crack tension specimens prepared from the forged bar, the P-COD curves indicated considerable subcritical crack growth, in spite of the fact that the fracture mechanics conditions indicated valid LEFM conditions. No valid K_{IC} data can be deduced, the K_{Ie} value is high. If a stress field intensity parameter K_{Ii} (for the load P_i at which the P-COD curve deviates from linearity, indicating the initiation of subcritical crack propagation during the tensile loading) is calculated according to Ref. 6, a value of 17 MPa.m^{1/2} is obtained.

The fracture toughness data for the various Mo specimens reflect the different material conditions (heat treatment, resulting microstructure and its anisotropy) and crack orientation (relative to the deformation axis).

Further investigations are necessary to reveal the influence of loading rate and test temperature on the fracture toughness data in view of the known strain-rate and temperature sensitivity of the mechanical properties of bcc materials. Preliminary tests with surface-cracked tension specimens of recrystallized TZM did not show any effect of loading rate on K_{Ie} for variations in loading rate between 20 and 650 MPa/min, Fig.4.

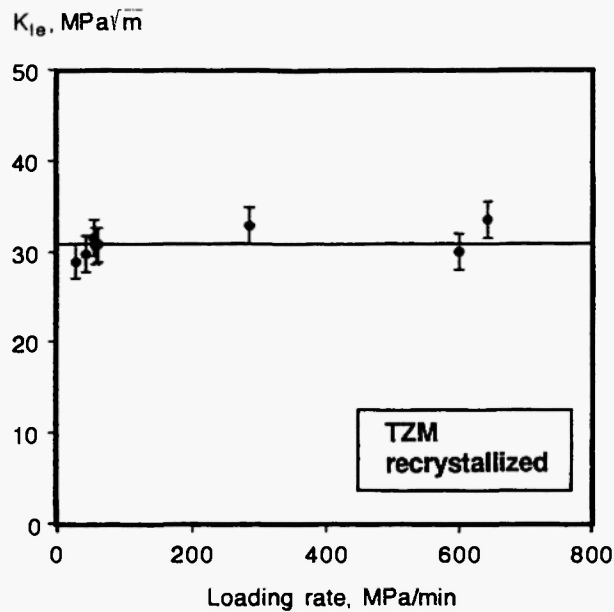


Fig. 4: Influence of loading rate on K_{Ic} values for surface crack tension specimens of recrystallized TZM

4. SUMMARY AND CONCLUSIONS:

The fracture toughness test procedures applied in this investigation should be of value for a general characterization of high-melting point metals and alloys in their practical product forms, such as forged bars, swaged rods, or plate products.

The method of testing surface-crack tension specimens yields with little testing efforts valid K_{Ic} values for low-toughness materials.

For high toughness materials which exhibit significant subcritical stable crack growth more elaborate analytical techniques are required.

ACKNOWLEDGEMENTS:

The authors thank Dr.H.Schmidt (Univ.Vienna) for his assistance in the precracking experiments of DCT-specimens and Mr.J.Femböck (Metallwerk Plansee GmbH) for valuable advice and comments.

LITERATURE

1. C.T.Wang and R.M.Pilliar, Short rod elastic plastic fracture toughness test using miniature specimens, *J.Mat.Science* 24 (1989) 2391
2. ASTM E 399-83
3. D.Broek, "Elementary Engineering Fracture Mechanics", M.Nijhoff Publ., Boston 1982, p.170
4. ASTM E 740-88
5. D.L.Chen et al, Determination of fracture toughness of forged rods: comparison of test results from a high-strength pm Al-Fe-Mo-Zr alloy, internal Plansee Report MPW92-B12 of 20.10.1992
6. C.W.Marschall and F.C.Holden, Fracture toughness of refractory metals and alloys, "High Temperature Refractory Metals", L.Richardson et al Eds., Gordon-Breach Science Publ., 1964,p.129
7. "Tensile properties, fracture toughness and ductile-to-brittle transition temperature of tungsten inserts", Southern Research Institute, SRI-EAS-88-162-6248-I-F, July 1988
8. S.Suresh and J.R.Brockenbrough, Theory and experiments of fracture in cyclic compression, *Acta Met.* 36 (1988) 1455
9. M.N.James et al, Fracture toughness testing of hard metals using compression-compression precracking, *J.Mat.Sci.* 25 (1990) 4810
10. "Stress Intensity Factors Handbook", Y.Murakami et al, Eds, Pergamon Press, Oxford, 1987, P.21
11. B.Weiss and R.Stickler, Review of the application of ultrasonic fatigue test methods for the determination of crack growth and threshold behavior of metallic materials, AIME Conf.Proceedings "Ultrasonic Fatigue", J.M.Wells et al, Eds, 1982, p.135
12. J.C.Newman, Jr. and I.S.Raju, An empirical stress-intensity factor equation for the surface crack, *Eng.Fract.Mech.* 15(1981) 185

