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Fatigue pre-cracking and fracture toughness in polycrystalline tungsten and molybdenum

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HIGHLIGHTS

- A precracking scheme for fracture toughness measurements in W and Mo is provided.
- Precracking requires compression fatigue followed by tension (3PB) fatigue loading.
- Tension (3PB) fatigue minimizes or reduces the effect of residual tensile stresses at the crack tip.
- Precracking and fracture toughness of W and Mo greatly depend on specimen orientation.
- The present fracture toughness of W and Mo is in the variation of the literature data.

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ABSTRACT

Fatigue pre-cracking performance and fracture toughness in polycrystalline tungsten (W) and molybdenum (Mo) have been investigated in relation to grain boundary (GB) configuration with respect to the crack advance direction. Sub-sized, single edge notched bend (SENB) specimens with three different orientations, R-L (ASTM notation) for a forged Mo rod and L-S and T-S for a rolled W plate, were pre-cracked in two steps: fully uniaxial compression fatigue loading to provoke crack initiation and its stable growth from the notch root, and subsequent 3-point bend (3PB) fatigue loading to extend the crack. The latter step intends to minimize the influence of the residual tensile stresses generated during compression fatigue by moving the crack tip away from the plastic zone. It is shown that fatigue pre-cracking performance, especially pre-crack extension behavior, is significantly affected by the specimen orientation. The R-L orientation, giving the easiest cracking path, permitted crack extension completely beyond the plastic zone, while the L-S and T-S orientations with the thickness cracking direction of the rolled plate sustained the crack lengths around or possibly within the plastic zone size due to difficulty in crack advance through an aligned grain structure. Room temperature fracture toughness tests revealed that the 3PB fatigued specimens exhibited appreciably higher fracture toughness by about 30% for R-L, 40% for L-S and 60% for T-S than the specimens of each orientation pre-cracked by compression fatigue only. This indicates that 3PB fatigue provides the crack tip front out of the residual tensile stress zone by crack extension or leads to reduction in the residual stresses at the crack tip front. Strong dependence of fracture toughness on GB configuration was evident. The obtained fracture toughness values are compared with those in the literature and its strong GB configuration dependence is discussed in connection with the appearance of pop-in.

1. Introduction

Tungsten (W) and molybdenum (Mo), the group VIA transition metals, possess 6 valence electrons of the 5 d and 6s orbitals: the

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5 d contributes to strong covalent bonding, and the 6s to metallic bonding. As a result, they exhibit many advantageous properties such as high melting points, good thermal conductivity, low tritium inventory, etc, and hence W is planned to be used as divertor (full W divertor) in ITER (International Thermonuclear Experimental Reactor) [1] and rotating solid targets for the high intense spallation neutron source in ESS (European Spallation neutron Source) [2].

Frontier technologies such as ITER and ESS utilize or go with extreme environments. However, practical use of W and Mo is limited to non-structural applications because W and Mo exhibit intergranular brittleness, which is peculiar to group VIA and accelerated by exposure to intense heat loading and heavy irradiation environments (severely embrittled by recrystallization and irradiation). In order to employ W and Mo as high temperature structural materials in extreme environments, it is essential to significantly improve their resistances to embrittlement by recrystallization and irradiation and construct data base of the mechanical properties of W and Mo materials, particularly the fracture toughness value. Therefore, research efforts have been made on the development of embrittlement tolerant W and Mo materials [3–6] and acquisition of their fracture toughness data [7–23].

Fracture toughness test specimens require a sharp pre-crack [24]. Pre-cracks are normally introduced by tension-tension fatigue (tension fatigue) from notches as a starter. A sharp crack in W and Mo, however, once formed at a notch root under tension loading, becomes unstable and grows so rapidly to fracture that it is in practice impossible to control crack growth and thus sustain an adequate crack length. Instead of tension fatigue, employed pre-cracking methodologies in W and Mo specimens have been cyclic compression [7,15], thermally induced cracking [14], bridge indentation [9] and compression and subsequent tension loading (each monotonic loading) [11]. Sharp notches, intended to imitate real cracks, have also been produced in W and Mo specimens by razor blade polishing [16] or FIB (focused ion beam) engraving [21] for fracture toughness tests.

Cyclic compression loading on notched specimens enables mode I pre-cracking with a stable crack growth for brittle and semi-brittle materials [25]. The driving force of compression fatigue pre-crack introduction is residual tensile stresses generated during unloading from the far field maximum compression stress. Since the studies by Suresh and coworkers [26], this pre-cracking technique has been applied to fracture toughness measurements in ceramics [27], WC-Co cermets [28], Mo [7] and W [15]. In view of easy cracking at grain boundaries (GBs) in W and Mo, pre-cracking performance may be greatly influenced by GB configuration with respect to the crack advance direction in the specimens that inherit anisotropic microstructures consisting of aligned and elongated grains. Because of limited availability in reports on cyclic compression pre-cracking and fracture toughness testing in W and Mo, studies of the pre-cracking conditions and crack front profiles in details are greatly desired for this pre-cracking technique to prevail for fracture toughness evaluations of these brittle metals.

Pre-crack introduction for fracture toughness testing requires the small scale yielding condition at the crack tip front. The ordinary methodology for this is to apply cyclic loads with a small value of stress intensity factor range ΔK on a notched specimen so that the introduced crack necessarily satisfies this condition. The application of cyclic compression with such small ΔK generates a minimal size of the residual tensile stress zone at the crack tip front but concomitantly limits the pre-cracking distance only with not much longer than the initial residual tensile stress zone size due to increasing degrees of contact of the crack planes with crack growth (crack closure effect) [26]. The range of ΔK employed in compression fatigue pre-cracking in W has been 20–45 MPa$\sqrt{\text{m}}$ [15], considered in small scale yielding, and has yielded a pre-crack length of only about 40 μm [19]. The experimental difficulty in dealing with an apparently short crack is associated with the confirmation of its formation from specimen surface observations during fatigue testing. Thorough-thickness introduction of such a short crack is practically also difficult and this could affect reliability and reproducibility in fracture toughness testing. Another obstacle in small ΔK compression fatigue pre-cracking is the necessity of sharp notch machining of the brittle metal, for which precise equipment and techniques are required.

The introduction of a longer pre-crack can be considered as a methodology for alleviation of these difficulties, alternatively providing an effective scheme of pre-cracking in W and Mo for fracture toughness testing. An appreciably longer pre-cracks (say longer than 0.2 mm) from a round notch root is sought with a sub-size specimen (3 mm in thickness, 5 mm in width) in this investigation but needs to resort to relatively large ΔK in compression fatigue. A concern for large ΔK in compression fatigue is that the crack tip front is accompanied by a large residual tensile stress zone, which may affect fracture toughness measurements. The minimization of the residual stress effect at the crack tip is essential to proper assessments of fracture toughness and for this the application of tension fatigue loading to the compression fatigue pre-cracked specimen is subsequently attempted prior to fracture testing. The introduced pre-crack length is compared with the estimated plastic zone size to inferentially identify the crack tip position associated with the plastic zone.

This study firstly intends to show the introduction of an appreciable length of a pre-crack by cyclic compression in single edge notched bend (SENB) specimens with three different orientations, taken from a rolled W plate and a forged Mo rod. Secondly, the cyclic compression pre-cracked specimens are subjected to cyclic tension for proper fracture toughness measurements: the influence of the pre-crack distance relative to the residual stress zone size is assessed in terms of the validity of the fracture test results. Finally, the effects of anisotropic microstructures on the fracture toughness are investigated with the pre-cracked SENB specimens of W and Mo and peculiar fracture behavior derived from the anisotropy is also highlighted.

2. Experimental

2.1. Materials and test specimens

Sub-sized fracture toughness test specimens (SENB) were cut out from a forged Mo bar (99.95% in purity, 40 mm in diameter), Fig. 1(a) and a rolled W plate (99.95%, 5 mm in thickness, 50 mm in width), Fig. 1(b). The Mo rod was produced by Plansee AG and the W plate by Toho Kinzoku Co., Ltd., Japan; both distributed by The Nilaco Corporation, Japan. The specimen dimensions (3 × 5 × 25 mm) and the notch configuration are shown in Fig. 1(c). The crack advance direction corresponds to the axis of the cylinder (R-L: ASTM notation) for the Mo specimens and the thickness of the plate for the W specimens, Fig. 1(a) and (b). The W specimens were taken in two directions from the rolled plate: notch plane perpendicular to and parallel to the rolling direction (L-S and T-S, respectively). The yield stresses (0.2% proof stress) measured by compression testing are 570 (R-L, Mo), 1450 (L-S, W) and 1550 MPa (T-S, W).

The notch was introduced by wire electro-discharge. Three notch widths (1, 0.5, 0.15 mm) were used to see the effect of notch root radius on pre-crack initiation; assuming the notch root as a half circle, the root radius is half of the width. The thinnest wire diameter available for electro-discharge slitting is 0.1 mm, which yields 0.15 mm notch width due to slight excess of material removal