Composites Part A 112 (2018) 365–370

Contents lists available at ScienceDirect

Composites Part A

journal homepage: www.elsevier.com/locate/compositesa

Mullite-zirconia-fibre/molybdenum-matrix composites: Strength and damage tolerance

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ARTICLE INFO

Keywords:
A. Metal-matrix composites (MMCs)
B. Strength
B. Damage tolerance
D. Mechanical testing

ABSTRACT

Molybdenum-matrix composites reinforced with composite fibres containing mullite and zirconia in various proportions (including that corresponding to eutectic) were successfully produced by the internal crystallisation method. The composites are characterised by a high damage tolerance: the average notch sensitivity value is 0.71, while the maximum value of this parameter approaches 1. Composites with eutectic and off-eutectic fibre compositions are sufficiently strong at temperatures up to 1400 °C. The mean strength values are 178 MPa and 140 MPa at temperatures of 1300 and 1400 °C, respectively.

1. Introduction

Motivated by the need to create metal-based high-temperature materials to replace nickel superalloys—which are functional at temperatures no higher than ~1100 °C—researchers have sought to develop (i) refractory metal alloys and (ii) metal-matrix composites (MMCs).

The initial work of the 'Beyond nickel superalloys' programme has generated numerous interesting and useful results, primarily through finding ways to decrease the oxidation resistance of niobium- and molybdenum-based alloys [1–5]. The main problem in developing high-strength metal alloys, including those based on refractory metals, is obtaining alloy microstructures that reach the necessary balance between strength (creep resistance) and fracture toughness [6]. Introducing silicide particles into a molybdenum matrix increases the alloy strength and creep resistance [7], but concurrently leads to a decrease in fracture toughness [8,9].

The main advantages of MMCs are their strength and fracture toughness characteristics. If such composites are designed appropriately, their strength and fracture toughness can simultaneously increase with increasing fibre volume fraction [6,10] even though high-temperature fibres are normally brittle at room temperature. This is a result of the larger fracture zone that forms at the crack tip in brittle fibres, caused by the formation of multiple fractures away from the macrocrack propagation surface.

One fabrication route to high-temperature composites is the internal crystallisation method (ICM) developed some decades ago [6,11]. This method is capable of producing either model composites with molybdenum matrices [11] or oxide fibres [12], to reinforce metal [13–15], intermetallic [16], and ceramic [17] matrices. Recently, it was shown that this method can be used to obtain molybdenum-matrix composites of a practical importance. The oxidation rate of a molybdenum matrix was reduced by reinforcing it with oxide-containing fibres with particular chemical compositions [18,19].

In the present paper, the fabrication, strength, and fracture toughness of a new family of molybdenum-matrix composites reinforced with mullite–zirconia fibres, which are produced by using ICM, are briefly described. The chemical composition of the fibres was chosen based on the strength data of 2 mm-diameter eutectic mullite–zirconia rods grown from the melt [20]. The fibre microstructure will be described in a paper to be published elsewhere.

2. Methods

The methodology of the present work includes (i) the ICM step and (ii) a novel mechanical testing method for composite specimens that yields strength and damage tolerance data.

2.1. Internal crystallisation method

The ICM for producing oxide-fibre/molybdenum-matrix composites consists of three main steps. First, a molybdenum carcass with continuous internal channels is prepared by the diffusion bonding of an assembly of wire and foil. The process is performed in such a way as to prevent the gaps between neighbouring fibres being filled with the foil material. Consequently, the channel cross-section is formed by two planar and two concave lines; this is the shape of the resulting fibres. Then, the carcass is infiltrated with an oxide melt, driven by capillary

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https://doi.org/10.1016/j.compositesa.2018.06.023
Received 5 August 2017; Received in revised form 11 April 2018; Accepted 17 June 2018
Available online 19 June 2018
1359-835X/

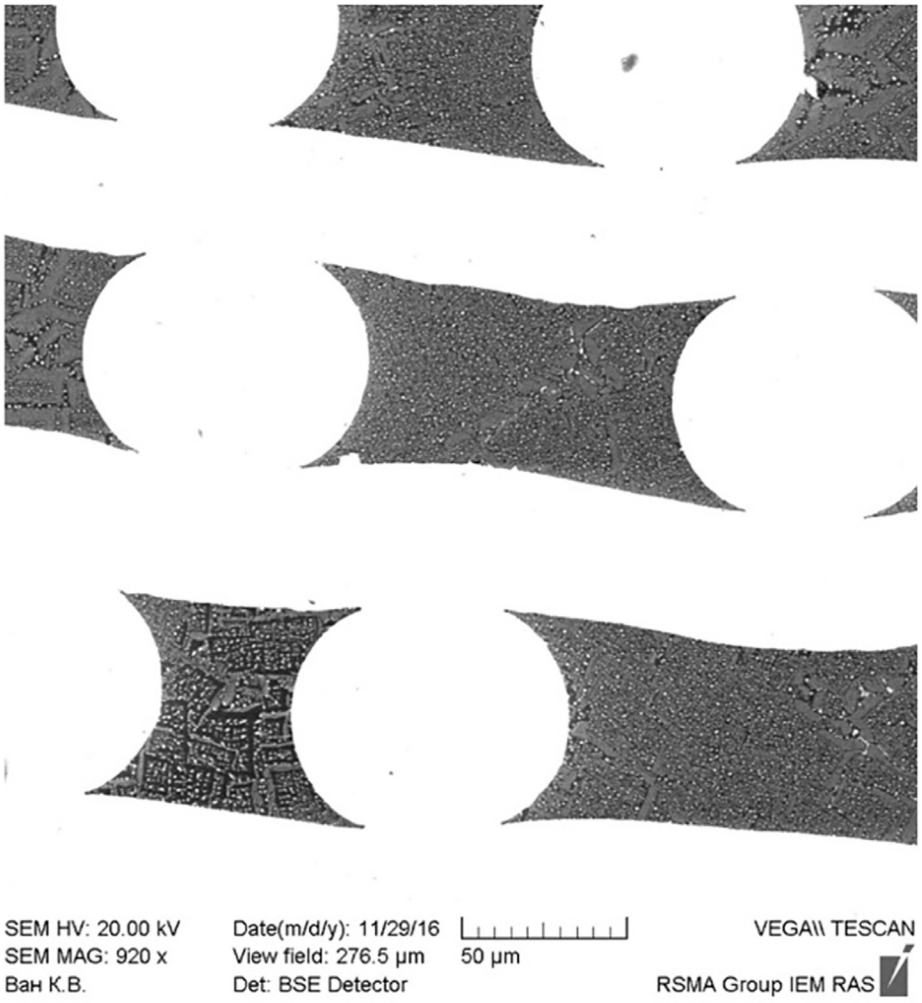

Fig. 1. Typical composite macrostructure.

force. The melt is then crystallised to form fibres in the channels of the oxide/molybdenum block by pulling the block up into the cold zone of the furnace. In the previous work on ICM cited in the Introduction, the oxide raw material was melted in a molybdenum crucible located below the molybdenum carcass. In the present work, the raw material was placed in a disposable molybdenum hopper located at the top of the carcass.

The typical macrostructure of the composite specimen obtained by ICM is shown in Fig. 1. In the present experiments, the foil thickness, wire diameter, and distance between the centres of neighbouring wires were 0.04, 0.08, and 0.16 mm, respectively. The corresponding geometrical calculation yields a fibre volume fraction of 40.5%. However, the real geometry can deviate from the ideal one (see Fig. 1), which leads to a decreased fibre volume fraction, so the real values for these specimens vary from ~35 to 40%.

The size of most of the specimens was ~5 × 15 × 65 mm. These are termed the original specimens. Also, some specimens were prepared with a size of ~5 × 5 × 65 mm.

Five reinforcing fibre compositions are shown in Table 1. Compositions E-1, E-2, and E-6 correspond to ceramics C_1, C_2, and C_3, respectively, which were grown from the melt; these were studied in Ref. [21]. E-1 corresponds to the mullite/ZrO_2 eutectic; E-2 is a zirconia-rich off-eutectic composition; and E-6 is a mullite-rich off-eutectic composition. The compositions of E-4 and E-7 are shifted from E-2 and E-6, respectively, towards zirconia. The main bulk of the experiments were performed with composites containing the E-1 and E-2 fibres.

Table 1
Compositions of the raw mixtures of oxides used to crystallize fibres reinforcing molybdenum matrix.

	ZrO_2	SiO_2	Al_2O_3
E-1 (C_1)	30	20	50
E-2 (C_2)	67	10	23
E-3	67	7	26
E-4	68	8	24
E-6 (C_3)	16	24	60
E-7	23	24	53

The raw fibre materials were prepared by (i) mixing the oxides in a proper ratio, see Table 1; (ii) cold pressing the oxide mixture to obtain a tablet with a diameter of ~12 mm and height of ~6 mm; and (iii) sintering the mixture in air at 1250 °C for 6 h.

During fibre crystallisation, which was performed in an argon gas atmosphere to suppress silica evaporation, the pulling-up rates were 10, 50, and 250 mm/min. These values are approximately equal to the fibre crystallisation rates.

2.2. Test methods

The original specimen shown in Fig. 2a was tested according to the procedure described in Ref. [10]. First, in a specimen denoted MXYZW, where M is a letter and X, Y, Z, and W are digits, a notch with a length