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The local strength of individual alumina particles

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ABSTRACT

We implement the C-shaped sample test method and micro-cantilever beam testing to measure the local strength of microscopic, low-aspect-ratio ceramic particles, namely high-purity vapor grown α-alumina Sumicorundum® particles 15–30 µm in diameter, known to be attractive reinforcing particles for aluminum. Individual particles are shaped by focused ion beam micromachining so as to probe in tension a portion of the particle surface that is left unaffected by ion-milling. Mechanical testing of C-shaped specimens is done *ex-situ* using a nanoindentation apparatus, and in the SEM using an *in-situ* nanomechanical testing system for micro-cantilever beams. The strength is evaluated for each individual specimen using bespoke finite element simulation. Results show that, provided the particle surface is free of readily observable defects such as pores, twins or grain boundaries and their associated grooves, the particles can achieve local strength values that approach those of high-perfection single-crystal alumina whiskers, on the order of 10 GPa, outperforming high-strength nanocrystalline alumina fibers and nano-thick alumina platelets used in bio-inspired composites. It is also shown that by far the most harmful defects are grain boundaries, leading to the general conclusion that alumina particles must be single-crystalline or alternatively nanocrystalline to fully develop their potential as a strong reinforcing phase in composite materials.

1. Introduction

Particle fracture is well-known to be one of the primary causes for the premature failure of particulate composites or multiphase alloys (Babout et al., 2004; Beremin, 1981; Brechet et al., 1991; Ghosh and Moorthy, 1998; Gurland, 1972; Lewandowski et al., 1989; Li et al., 1999; Llorca et al., 1993; Mummery et al., 1993; Mummery and Derby, 1994; Pandey et al., 2000; Scarber and Janowski, 2001). It is also known, from both experiment (Bonderer et al., 2008; Bouville et al., 2014; Hauert et al., 2009a; Kouzeli et al., 2001; Krüger and Mortensen, 2014; Le Ferrand et al., 2015; Miserez et al., 2006, 2004a, 2004b; Miserez and Mortensen, 2004; Munch et al., 2008) and micromechanical theory (Hauert et al., 2009b; Tekoglu and Pardoen, 2010), that stronger, more ductile, and tougher particulate composites are produced if the intrinsic strength of their particulate reinforcement is increased.

It is in this light surprising that so little attention has been paid to quantifying the strength of hard particles used in composites or found as coarse precipitates in alloys. One of the main reasons for this is that the strength of a particle is

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inherently difficult to measure since, unlike a fiber, it cannot be gripped. To date the strength distribution of particulate reinforcements has generally been estimated indirectly, by coupling more or less elaborate micromechanical models of two-phase composite material behavior with direct or indirect observations of particle fracture (Babout et al., 2004; Brockenbrough and Zok, 1995; Caceres and Griffiths, 1996; Eshelby, 1957; Hauert et al., 2009a; Kiser et al., 1996; Lewis and Withers, 1995; Li et al., 1999; Majumdar and Pandey, 2000; Mochida et al., 1991; Wallin et al., 1986; Wang, 2004; Wang et al., 2003) or using X-ray and neutron diffraction to estimate average strains in reinforcing particles at the onset of particle cracking (Coade et al., 1981; Finlayson et al., 2007; Mueller et al., 2008).

Comparatively, only a few studies report direct measurements of the fracture stress in individual reinforcing particles. Pioneering efforts can be found in Harris et al. (2007), Joseph et al. (2015), Ogiso et al. (2007), Webb and Forgeng (1958) and Yoshida et al. (2005). Newer techniques give access to more direct measurements, free of extraneous flaws that were often introduced in the particles in earlier methods. Recent developments in three-dimensional synchrotron X-ray diffraction enable now the determination of micrometer-scale grain-resolved elastic strains within multiphase materials (Sedmák et al., 2016); however, to the best of our knowledge this technique has not been used to measure the local stress at fracture in reinforcing particles. Another recent method used to measure the strength of individual microscopic particles combines focused ion beam machining (FIB) with microscopic particle bending tests (Feilden et al., 2017; Mueller et al., 2016). This approach provides access to unambiguous strength data of individual reinforcement particles; however, it is only suitable for particles of relatively high-aspect ratio, i.e., for fibers or platelets.

We present here an alternative micromechanical testing approach, which also uses FIB-shaped samples and measures the local strength, directly and free of micromilling artifacts, in the surface region of particles of almost any convex shape. The approach is inspired from the parallel problem of mechanical characterization of ceramic bearings (Strobl et al., 2014; Supancic et al., 2009; Wereszczak et al., 2007a, 2007b), which we have recently extended to the microscale and demonstrated by measuring the local strength of strong and brittle reinforcing fibers embedded in a metal (Žagar et al., 2015). Here we extend this approach towards testing microscopic particulate composite reinforcements of irregular shape and low aspect ratio. Specifically, we report here observations of directly determined strength measurements conducted on Sumicorundum® (Sumitomo Chemical Co. Ltd., Tokyo, Japan) alumina particles of diameter in the range 15–30 μm, which have been shown to provide superior reinforcement in infiltrated aluminum- or copper-based metal matrix composites (Kouzeli et al., 2001; Krüger and Mortensen, 2014; Miserez et al., 2006, 2004a; Miserez and Mortensen, 2004). As will be seen, the measured strength of the alumina particles can be higher than what is measured for high-strength nanocrystalline alumina fibers, or in alumina platelets a few hundred nanometers thick that have been used in bio-inspired composites. Their strength approaches that of high-perfection alumina whiskers and microcrystals where specific defects are absent. We identify those specific defects as: (i) grain boundaries and grain boundary grooves, (ii) micropores and (iii) other shape irregularities. We show that random grain boundaries are particularly deleterious to the strength of alumina particles, leading to an overall conclusion that alumina has great potential as a particulate reinforcement in composite materials provided it is (i) smooth in shape and (ii) single- or nano-crystalline.

2. Materials and methods

2.1. Material

Particles investigated in this work were high-purity α-alumina particles (AA-18, Sumicorundum® grade) produced by Sumitomo Chemical Co. Ltd. (Tokyo, Japan) by in-situ chemical vapor deposition. The typical particle size is between 15 and 30 μm (Uchida et al., 1995). XRD analysis performed in our laboratory confirmed that the particles are entirely of α-alumina. Chemical analysis provided by the manufacturer states as main impurities Si ($<$ 50 ppm), Fe ($<$ 20 ppm), Na ($<$ 15 ppm), Mg ($<$ 10 ppm), and Cu ($<$ 10 ppm). The as-received powder particles have polyhedral shapes and consist generally of (i) single crystal particles with a low aspect ratio or (ii) particles consisting of two or more polyhedral crystallites linked along a grain boundary, which forms a groove at the particle surface. By investigating 200 SEM images of random Sumicorundum particles from this study, we estimate that roughly half of all the particles are single-crystalline while the other half is made of two or more crystallites forming grain-boundary grooves.

2.2. Micromechanical testing

2.2.1. C-shaped particle test method

To measure the local strength of the particles we extend to particles the approach presented in Žagar et al. (2015). The approach requires having particles partially embedded in a matrix that holds each particle in place during load application. To this end, a Sumicorundum particle – polymer matrix composite with a low fraction of the alumina particles (a few percent) was produced. The polymer matrix was then deep-etched, or alternatively in one specimen it was cracked, so as to cause particles partially embedded in the matrix to protrude by several micrometers out of the matrix. To prevent sample charging the FIB/SEM and to protect the surface of particles from FIB damage and/or redeposition, the sample with partially embedded particles was coated with $\approx$40 μm of thermally evaporated carbon. Selected particles were FIB milled to form a C-shaped particle specimen, outlined in Fig. 1 and featuring (i) two parallel and roughly vertical sides, (ii) a wide rectangular notch machined with the beam oriented perpendicular to the previously machined sides, and (iii) a roof situated at the top