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The local strength of microscopic alumina reinforcements

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ABSTRACT

We measure, using an adaptation of a method designed for ceramic ball bearings, the local strength of a brittle second phase that serves to reinforce a metal. The method uses focused ion beam milling and a nanoindentation device, and is free of artifacts commonly present in micromachined specimens. It is demonstrated on Nextel 610™ nanocrystalline alumina fibers embedded in an aluminum matrix composite. Results reveal a size effect that does not follow, across size scales, usual Weibull statistics: the fiber strength distribution differs between measurements at the microscale and macroscopic tensile testing. This implies that, in micromechanical analysis of multiphase materials, highly localized events such as the propagation of internal damage require input data that must be measured at the same, local, microscale as the event; the present work opens a path to this end.

1. Introduction

Many structural metallic materials combine a ductile matrix with a brittle second phase; important examples are silicon in cast aluminum, carbides in steel, or ceramic fibers or particles in composites. These multiphase materials constitute an interesting and now classical problem in micromechanics: as they deform, the matrix undergoes plastic deformation while the much stiffer, strong but brittle second phase remains elastic and takes up a disproportionate portion of the applied stress. Such two-phase materials produce one of the clearest manifestations of the plasticity size-effect ("smaller is stronger"), physical mechanisms of which have been the subject of a consistent research effort over the past two decades [1–5].

Comparatively, the brittle ingredient of multiphase structural materials has received far less attention and is less well understood. It is known that small brittle reinforcements embedded in metal deform elastically and then break stochastically; alternatively they debond if their interface with the matrix is weak. These events cause the two-phase material to accumulate internal damage, which in turn severely limits both its ductility and its strength. Conversely, if second phases and interfaces are strong, then the material that contains them can be strong, ductile and tough, at times remarkably so [6,7].

The strength of hard second phase particles is, hence, one of the main parameters that govern the mechanical performance of many engineering materials; yet measuring this strength has remained a challenge. Methods employed to date rest on assumptions or are subject to caution. For example, finite volumes of the multiphase material under stress can be sampled by means of neutron or synchrotron X-ray diffraction, or using internal damage detection methods such as synchrotron X-ray microtomography or acoustic emission [8–10]. These methods produce rich and relevant results; yet to obtain the stress of a single particle knowing the composite or the average phase stress requires micromechanical modeling, which in turn rests on idealizations of the material microstructure and/or load transfer mechanics [3,6,11,12].

An alternative is to test small brittle second phases directly. The mechanical testing of small particles is in fact a relatively wide area of research because the topic has relevance in comminution, a process that intervenes in many branches of industry (chemical, material, food, and pharmaceutical industries). Particle testing for strength can be conducted by crushing individual particles between two hard surfaces, or in fracture-by-impact tests [13–15]; however, the underlying mechanics of both methods is complex and still a subject of debate [16–19]. The mechanical behavior of small particles has been probed using nanoindentation, but the complex stress states produced complicate data interpretation [20–23]. Another approach to measure strength at microscopic or nanoscopic length scales is to employ techniques evolved from thin film and micro/nano-electromechanical systems technology [24–27]. Micromechanical testing samples can notably be

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produced by thin-film processes, or can be carved out of bulk material using focused ion beam (FIB) milling [27,28]. The most commonly conducted shaped-microsample mechanical tests, due to relative ease and flexibility in preparing the test specimens out of virtually any piece of a bulk material, include microbeam bending or miniature uniaxial testing [29–32]. Such tests are now nearly routine; however, the data they yield are subject to artifacts and hence cannot be trusted because specimen surfaces produced by ion-milling contain damage [28,33,34]. Finally, a few other methods have been proposed that measure stress within one phase of a multiphase material; here too strength measurements are problematic given the fact that the phase surface was altered by polishing [35–37].

Here we present a micromechanical testing approach by which one can measure, free of micromilling artifacts and directly, the local strength of individual small second phase elements in a multiphase metallic material. The novelty of the approach presented here lies in that (i) it can be adapted to variously shaped convex second phases (thus, it is not restricted, for example, to thin films) and (ii) it probes the strength of material, the surface of which is unaffected by micromachining or polishing.

The method was inspired by a recently proposed solution to the problem posed by the mechanical characterization of macroscopic ceramic spheres or cylinders used in bearings: such smooth spheroids are strong, brittle, and are as difficult to grip for loading in tension or bending as are convex second phases in metallic materials [38–40]. The idea behind the test is to machine a wide notch, so that compressive loading can put the remaining ligament outer surface into a state of localized tensile stress. Besides the fact that fracture in this specimen configuration takes place in material the surface of which is unaffected by FIB micromilling, the method has the advantage that applied loads are small enough not to cause the brittle microphase to shatter upon fracture. This in turn enables fractographic analysis and the identification of fracture-inducing flaws.

The method is in principle suited for any convex inclusion of brittle material that can be carved by FIB milling. To develop the method and explore what it teaches on a material of engineering significance, we have chosen to use ~12 μm diameter nanocrystalline alumina fibers embedded in an aluminum matrix as a testbed material. Reasons for this choice are (i) that these fibers are an engineering material with a regular convex shape and an isotropic microstructure (which in turn eases data interpretation), and (ii) that continuous fibers such as these can also be tested for strength using conventional tensile testing of macroscopic samples. Hence, strength data obtained here by means of the present microscopic testing method can be confronted to strength data reported in the literature from another testing method conducted on the same fibers [41–43].

As will be seen in what follows, the two testing methods yield different strength distributions for the same material: the reason is obviously that the tested volumes of material are so different from one test to the other (tested areas are centimeters long in tensile tests, ten or so micrometers long in the present method). This difference, in turn, points to the fact that size-scaling of strength data, such as given by Weibull statistics, cannot be used to extrapolate strength distributions in brittle second phases across dimensional scales. Phenomena that are driven by highly localized stresses, such as the propagation of damage from one brittle inclusion to the next or crack tip processes, are governed by different defects and hence different strength distributions than are phenomena such as damage initiation across large volumes of material subjected to a homogeneous state of tensile stress. We show here how strength distributions pertinent to the former, microscale, can be measured directly, by coupling microtesting of carved C-shaped brittle phase regions with bespoke finite element simulations, taking due account of friction effects since these take particular importance at the microscale of the present strength measurement method.

2. Methods

2.1. Notched sample preparation

We probe here high-strength nanocrystalline Nextel™ 610 alumina fibers produced by 3M™ (St. Paul, MN, USA). These fibers are in particular used to reinforce a pure aluminum matrix composite wire of ~2 mm in diameter, also produced by 3M™. The Nextel™ 610 fiber is 99.5% α-Al_2O_3. The fibers are ~12 μm in diameter and their typical microstructure is characterized by equiaxed grains with a mean diameter of ~65 nm [43]. The orientation of the fibers in a composite wire is such that the fiber axis is roughly collinear with the wire axis.

Prior to FIB micromachining of the test samples, the composite wire was prepared so as to enable cutting a rectangular notch perpendicular to the axis of individual fibers. First, a ~1 cm long segment of the composite wire was cut using a diamond cutting wheel (Accutom-50, Struers, Denmark) and mounted in epoxy resin. The mounted wire was then ground and polished along two planes, one cross-sectional and the other longitudinal, the latter placed roughly midway across the wire. This produced a sharp ~90° edge passing roughly through the center of the wire. Next, the fibers were exposed by deep etching the aluminum matrix with 20 wt. pct. NaOH for approximately 1 h at room temperature, followed by rinsing in distilled water. Fibers that remained loosely attached near the ~90° edge after deep etching were manually removed, using sharp tweezers under an optical microscope. The result of this procedure is a sample showing several tens of micrometer of exposed fibers, which lower down are embedded in the aluminum matrix, and whose top was gently polished during final metallographic preparation while side surfaces are in pristine condition (Fig. 1a and b).

Fibers to be probed were selected based on their geometrical separation from neighboring fibers; indeed, some space around the fiber is required for access of the ion beam during milling, and also for the fiber to be free to bend during the mechanical test. The notch and the rooftop were then FIB-milled into each of the selected fibers with the beam direction oriented parallel to the polished surface of the wire.

The first feature to be machined is a several μm-wide rectangular notch oriented in a way such that the notch faces are parallel or perpendicular, respectively, to the fiber axis. Then a two-sided roof is machined along the top of the fiber, with its edge situated eccentrically relative to the ligament neutral axis (Fig. 1c). Note that these two features can be milled also in a fiber that is located (when looking along the ion beam path) behind another fiber (Supplementary Fig. S1); such was the case for ~3/4 of the specimens tested in this work.

The FIB machining process was performed either with a Zeiss NVision™ 40 (Oberkochen, Germany), or (more seldomly) with a FEI Nova 600 NanoLab, both being dual beam (SEM/FIB) instruments. Both FIBs featured a 30 kV Ga+ gun and were used with currents of 6.5 nA for the initial coarse milling steps, subsequently reduced to 1.5 or 0.7 nA for the final steps. Prior to the FIB machining process, a ~5–10 nm carbon layer was deposited using a Cressington™ 208 Carbon Coater (Watford, England, UK) to avoid charging of the alumina fibers while irradiating with electron or ion beams. Before testing, each FIB milled fiber sample was extensively imaged with SEM in order to retrieve the characteristic dimensions (Fig. 1c and Supplementary Table S1) that are needed for the finite element modeling and data analysis.