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In-situ strength of individual silicon particles within an aluminium casting alloy

M.G. Mueller*, G. Žagar, A. Mortensen

Laboratory of Mechanical Metallurgy, Institute of Materials, École Polytechnique Fédérale de Lausanne (EPFL), Station 12, CH-1015 Lausanne, Switzerland

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ABSTRACT

Measurements of local strength are performed *in-situ* on individual silicon particles that constitute the second phase of aluminium alloy A356. Particles are shaped using Focused Ion Beam (FIB) milling such that, upon the application of a compressive force on the particle, a volume of material unaffected by FIB milling is subjected to bending. Silicon particles in this commercial aluminium casting alloy are shown to be capable of locally sustaining tensile stresses as high as 16 GPa, i.e., approaching theoretical strength. The reason why such strengths are not reached by most alloy Si particles is shown to be the presence of specific surface defects, the effect of which is assessed. The most deleterious defects are interfaces between merged silicon crystals; therefore, eliminating these might lead to significantly enhanced strength and ductility in this widely-used casting alloy family.

1. Introduction

In heterogeneous materials composed of hard brittle particles embedded in a soft ductile matrix, mechanical damage generally consists in particle fracture, particle-matrix interfacial decohesion, or matrix voiding. In Al-Si alloys, a prototypical example of such materials representing the vast majority of aluminium-based casting alloys, early stages of damage are typically dominated by the fracture of silicon particles. These constitute the main alloy second phase and can take a variety of forms, typically of intricate networked or isolated particles, depending on the alloy composition and processing history [1–5]. The strength of Si particles initially grown from the Al-Si eutectic reaction, potentially modified by subsequent heat treatment, and embedded in aluminium is thus a key parameter underlying the performance of most cast aluminium alloys; however, its measurement and the identification of pathways towards its improvement do not, to date, have clear answers.

Silicon particles in Al-Si alloys are microscopic and irregular in shape, features that have long been major impediments to probing them mechanically. Thus, the main approach to characterize their strength has been to estimate their strength (or strength distribution) from measured macroscopic alloy properties.

A classical approach consists in relating the estimated (average) stress in the silicon phase to the fraction of broken particles, itself measured along polished sections of the probed material or using X-rays or neutrons (for diffraction and/or tomography), after deformation to varying levels of macroscopic strain. Typically, a Weibull strength distribution is extracted for the population of silicon particles [3,6,7]. To estimate the stress in the silicon particles while the multiphase material is strained, different strategies have been implemented. Caceres and Griffiths [3] exploited a dispersion hardening model that uses the particle average aspect ratio and volume fraction as the main parameters that characterize the silicon phase [8]. Kiser et al. [9] calculated the stress in the particles from a constitutive law derived from a finite element analysis [10]. Nishido et al. [11] used an expression based on assimilation of the Si phase to an equivalent Eshelby inclusion, while Huber et al. [12] used an extension of the Eshelby theory to estimate the stress in silicon particles, deriving from this the silicon particle strength by using it as the fitting parameter of their particle fracture-induced void nucleation model.

These strength estimations are indirect assessments of the averaged behaviour of the silicon phase within Al-Si alloys. This is also a feature of studies where X-ray or neutron diffraction techniques were used to quantify the stress in the silicon phase [13,14]. An alternative approach, which probes particles directly, was proposed in a study where scratch tests were performed on a deep-

* Corresponding author.
E-mail address: martin.mueller@epfl.ch (M.G. Mueller).

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etched Al-Si alloy to estimate the average bending stress experienced before fracture by the protruding particles [15].

A common feature of all of those investigations is that, by measuring average strength values, they do not provide insight into the actual fracture mechanism of individual particles, so as to shed light on the reasons why silicon particles are found in those studies to exhibit strength values of only 0.1–3.5 GPa [3,6,7,9,11–15], which are well below the theoretical strength of Si, at 17–27 GPa [16] - a question that has indeed been formulated (either explicitly or implicitly) repeatedly in the literature on Al-Si alloys [11,13,14,17–22].

Strength measurements of individual silicon particles within Al-Si alloys are, by comparison, rare. Harris et al. [21] demonstrated the application of a micro-Raman technique to follow the stress state in a single particle during alloy mechanical testing; however, the measured particle strengths are subject to artefacts since the particles were polished (as required for the method). Recently, coarsened silicon particles extracted from an eutectic Al-Si alloy were probed individually using a microscopic three-point bending technique [23]; results showed that silicon particles can be actually much stronger than had been determined before. Also, the study showed the deleterious influence of specific surface features on the strength silicon particles [23]. The present work builds on, and complements, that contribution: here, tests are conducted on (smaller) silicon particles within the commercial aluminium casting alloy A356 using a different micromechanical testing method inside a Scanning Electron Microscope (SEM). Particularly, the weakening effect of different defect types found in particles within Al-Si and Al-Si-Mg alloys [24] (and likely in other Al-Si casting alloys too) is confirmed and quantified. Results also highlight the enormous strength potential of Si particles reinforcing aluminium if these can be produced free of those defects.

2. Materials and methods

2.1. Material

The particles probed in this work are Si particles produced in the course of conventional alloy processing within aluminium casting alloy A356 (Alu Metall Guss AG, Gontenschwil, Switzerland), of composition limits: 6.5 to 7.5 wt% Si, 0.25 to 0.45 wt% Mg, max 0.20 wt% Fe, max 0.20 wt% Cu, max 0.10 wt% Mn, max 0.10 wt% Zn, max 0.20 wt% Ti, max 0.05 wt% of others (each), max 0.15 wt% others (total), balance Al. The alloy was remelted and cast as a rod 15 cm high and 2 cm in diameter using a copper permanent mould. Heat-treatment was then conducted at 540 °C for 6 h, these being standard parameters for the solutionizing step of this alloy's T6 heat-treatment schedule [25]. The resulting microstructure is shown in Fig. 1a on a polished section of the material, where the silicon particles are dark and the α-aluminium phase is bright.

To expose the silicon particles within the microstructure, the aluminium phase was selectively dissolved to a depth of a few tens of micrometres by soaking a polished sample of the alloy in a mixture of phosphoric acid 85%, acetic acid 100% and nitric acid 70% in volume ratios 83:5.5:5.5 for 2 h. Fig. 1b–c shows the topography after this deep-etching procedure, where the partly protruding silicon particles are readily visible.

2.2. The mechanical testing method

We probe the strength of individual particles a few micrometres in size, produced within the alloy after standard alloy processing steps detailed above. As will be seen, test geometries on different particles vary; however, the general idea in all cases is that the particle be shaped using a Focused Ion Beam (FIB) such that a well-defined part of the particle that was not ion-milled be subjected to bending upon the application of a compressive force using the tip of an instrumented (tungsten) needle. This defines a portion of the particle surface that is subjected to tensile stress, which is where fracture will eventually take place.

A main premise of the method is that the surface where strength is probed be left in its pristine condition; it thus must be unaffected by FIB milling and its shape thus cannot be altered. This, together with the fact that particles are all different from each other, both morphologically and in terms of their disposition in space, makes it impossible to prepare specimens of the same shape and the same dimensions out of the many different particles within the alloy. Instead, each particle is shaped individually exploiting its own characteristics. This makes each specimen probed in this work unique in terms of its geometry. There are, nevertheless, two common features to most particles tested here (exceptions are described in the next subsection). One is a deep, rectangular, notch micromilled with the FIB so as to define a remaining ligament that will be subjected to bending upon the subsequent mechanical testing (Fig. 2c). The second is a roof at the top of the particle, introduced to ease load application.

The method to probe the strength of brittle particles used in this work is thus an adaptation of the notched sample test developed on alumina fibres 12 μm in diameter in Ref. [26]. That work introduced at a microscopic scale the concept of milling a notch to produce, upon the application of a compressive force, bending in a pristine ligament of material. There are, nevertheless, differences between the methodology that was developed in Ref. [26] and that of the present work: (i) the particles tested here are randomly oriented and significantly more irregular morphologically; (ii) they are also nearly one order of magnitude smaller.

The small size and irregular shapes of particles tested here make it essential to be able to see the sample in real time and have sufficient tilt and rotation capability in performing sample positioning and alignment adjustments prior to mechanical testing; for these

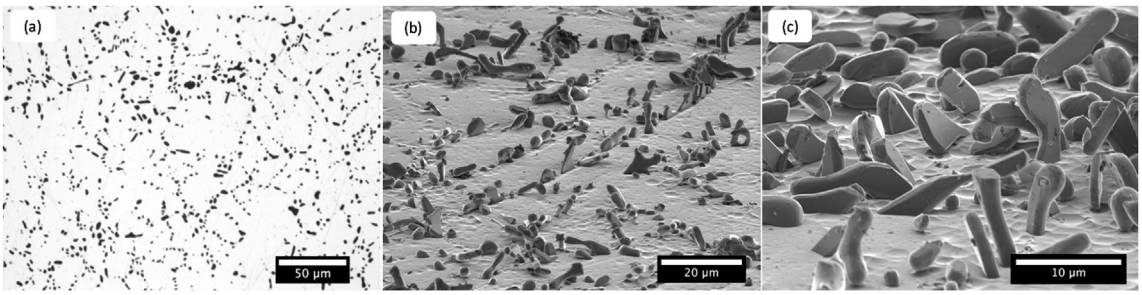

Fig. 1. Microstructure of the alloy A356 heat-treated 6 h at 540 °C used in this work. (a) Optical micrograph of a polished section. (b–c) SEM images showing silicon particles exposed after a deep-etching procedure.