Acta Materialia 105 (2016) 165–175

Contents lists available at ScienceDirect

Acta Materialia

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

Full length article

Microscopic strength of silicon particles in an aluminium–silicon alloy

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ARTICLE INFO

Article history:
Received 2 November 2015
Received in revised form
2 December 2015
Accepted 2 December 2015
Available online xxx

Keywords:
Al–Si alloys
Silicon
Particle
Fracture
Focused ion beam (FIB)

ABSTRACT

A microscopic three-point bending test that measures the strength of faceted particles of high aspect ratio is developed and used to probe individual coarsened plate-like silicon particles extracted from the eutectic Al-12.6%Si alloy. Focused ion beam milling is used in sample preparation; however, the tapered beam cross-section and multistep preparation procedure used here ensure that the particle surface area subject to tension in mechanical testing is free of ion beam damage. Results show that coarsened silicon particles in aluminium can reach strength values on the order of 9 GPa when they are free of visible surface defects; such high strength values are comparable to what has been reported for electronic-grade silicon specimens of the same size. By contrast, tests on eutectic silicon particles that feature visible surface defects, such as pinholes or boundary grooves, result in much lower particle strength values. Reducing the incidence of surface defects on the silicon particles would thus represent a potent pathway to improved strength and ductility in 3xx series aluminium casting alloys.

1. Introduction

The link between the microstructure and the mechanical properties of aluminium-silicon (AlSi) based alloys is a subject that has been investigated extensively for several decades [1–15] because it is both relevant and broad. It is relevant because more than 90% of today's aluminium castings are based on AlSi, a binary system which provides for excellent castability at low cost, leading it to be widely used, for example in the automotive industry [16]. The subject is broad because the properties and morphologies of engineering AlSi alloy microconstituents can be varied greatly with alloy composition, and with casting and heat treatment procedures [4–6,8,17–25]. The subject is also interesting because aluminium-silicon alloys are, together with metal matrix composites, an attractive model system for the study of damage and fracture in two-phase ductile-brittle materials [1,3,6,7,10,26–28].

In the simplest case, namely a binary AlSi alloy, the microstructure consists of a ductile aluminium matrix reinforced with brittle silicon particles. These two phases are also the main (but not the only) phases in more chemically complex 3xx series aluminium casting alloys. In most of these alloys, the fracture of silicon particles plays a dominant role among the various factors that determine the alloy's deformation and fracture properties [3,5–7,15,21,24,25,29–36].

Yet, little is known of the intrinsic strength of Si particles within aluminium [37,38]. To date, the strength of silicon particles in AlSi alloys has been assessed mainly by indirect methods. These evaluate average properties of the silicon phase by taking measurements on the alloy and then interpreting data via a model that links properties of the inclusions with those of the alloy.

The strength distribution of silicon particles has in this way been estimated by coupling dispersion hardening models, which were used to calculate the average stress in the particles from macroscopic alloy flow properties, with a measurement of the volume fraction of broken particles, which was produced using light microscopy along polished sections of tensile samples [8,30,39]. Caceres et al. [30] and Wang et al. [8] calculated in this way a Weibull reference strength of 3 GPa for eutectic silicon particles having a 4 μm representative diameter in heat treated A356 and A357 alloys. The strength of the particles was widely distributed, however, given that particle fracture was observed to begin at (average estimated) particle stresses of about 500 MPa. Using the same approach, Kiser et al. [39] estimated 215 MPa as an average strength of the Si phase, without discrimination of primary from eutectic silicon particles, in the form of coarser particles within a hypereutectic Al–20Si alloy heat-treated to the T4 condition. In an investigation on the plastic deformation of A356 and A357 alloys with and without Sr-modification, Wang [9] estimated the average tensile stress on the silicon particles as a function of the alloy plastic strain. Their results show a change in the shape of the curve at a

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http://dx.doi.org/10.1016/j.actamat.2015.12.006

particle stress level in the range of 600–800 MPa (depending on the particle aspect ratio). This was associated with the onset of particle cracking and matrix plastic relaxation.

Huber et al. [10] developed a void nucleation and growth model using a near-eutectic, Sr-modified, AlSi alloy with silicon particles of shape near that of a spheroid. In the model, void nucleation was attributed to fracture of the silicon particles and the critical stress value for particle fracture in the model, or in other words the average strength of the particles, fitted the experiments with a value of 550 MPa. In a comparatively early work, Coade et al. [1] used a X-ray diffraction technique to measure the average strain of the silicon particles in a sodium-modified A356 alloy submitted to bending. The strain was used to estimate the average stress in the particles and the fracture stress of the particles was estimated to be 230 MPa. It is noteworthy that, as the authors state in the paper, such a fracture stress would imply surface cracks 2–4 μm deep on the particles, which is admittedly inconsistent given that the particles size was also on that order. Along the same line, in a more recent work Finlayson et al. used a neutron diffraction technique during tensile testing to measure the average strain in the silicon particles of Sr-modified A356 alloys heat treated to T4 and T6 conditions [38]. To evaluate the strength of the silicon particles, a macroscopic applied plastic strain of only 0.01 was considered (strains larger than that were claimed to yield too large uncertainties), which resulted in average particle stress values of 220–330 MPa. At that level of applied plastic strain only 1–2% of the particles would be fractured, which makes this assessment a strength estimate of only the weakest particles in the alloy [38].

All studies cited in the previous paragraphs require a micromechanical model to deduce the stress on the Si particles either from the measured composite stress or from the measured average Si particles lattice strain. This in turn raises two issues, namely (i) the validity of approximations made in constructing the micromechanical models, and (ii) the fact that such models give a value for the average stress exerted on the particles, which may differ significantly from the stress exerted on those particles that fracture at a given point of the composite's deformation history. As a result, precisely when and why silicon particles in AlSi alloys fracture is at present still poorly understood. Measuring locally the strength of individual silicon particles can provide direct insights into the matter; however such a measurement is a significant challenge that has only been reported in a couple of studies.

One approach that has been proposed to achieve this is based on the micro-Raman technique [35,40], specifically a method developed by Narayanan et al. [41], which allows to relate the shift of the silicon peak in the Raman spectrum with the in-plane stresses of a (111) silicon wafer. Applying this on silicon particles in a AlSi alloy thus requires a well-polished surface of bending [40] or compression [35] test samples of the alloy, along which a silicon particle identified as having a (111) orientation can be spotted. The main drawback of this approach is that the particle on which the stress is measured is affected by polishing, which introduces defects (scratches) on the particle. With this technique, Joseph et al. found that eutectic silicon particle fracture occurred at stresses in the range 500–1000 MPa, whereas Harris et al. reported a value of 600 MPa. Another approach to the direct measurement of alloy particle strength was described by Riahi et al. [42], where the strength of silicon-rich intermetallics in a modified AlSiFeCuNiMgMn alloy was measured by means of an instrumented scratch test conducted on a surface of the alloy. The particles, protruding relative to the aluminium matrix by a few micrometres after a surface deep-etch, were bent to fracture by the sliding indenter. The approach, however, was not applied to silicon particles (to the best of our knowledge).

In summary, the strength of the silicon phase in AlSi alloys has been mostly assessed using approaches that measure averaged back-calculated phase properties, and differences in measured strength values have been interpreted in terms of average geometrical and morphological features of the Si phase such as size, aspect ratio and interconnectivity. Reasons why Si particles are as strong or as weak as they are found to be, or in other words structure/property relations in these particles viewed as a material with its own strength-limiting defects, have not yet been explored in depth. In the present work, we probe the local strength of eutectic silicon particles individually by means of a microscopic three-point bending test, results of which give direct measurements of individual particle strength. We use the method on particles where no defects are noted and also on particles containing observable defects, and we measure thereby the effect of such flaws on their strength.

2. Materials and methods

2.1. Method

The flexural strength of plate-like silicon extracted from a binary AlSi alloy is measured in this work using a microscopic three-point bending test conducted on particles that are removed from the alloy by selective leaching the aluminium matrix. Prior to leaching, the alloy is heat-treated to coarsen the particles; this causes them to adopt naturally a variety of plate-like shapes showing flat surfaces oriented along (111) planes of the Si crystal [43–46]. We use one such flat surface, as it presents itself after etching, as the probed surface subjected to peak tensile stress during the bend test.

The sides of the specimens are shaped by focused ion-milling to turn the particles into straight beams amenable to bend testing: this inevitably causes material in the corners of the lower beam surface to be altered by the ion beam. In early measurements conducted with beams that had parallel sides and rectangular cross-sections, we found that fracture surfaces can betray crack initiation at, or near, the beam corner, i.e. from a portion of irradiated and gallium-implanted material which is likely not to be representative of silicon as it is within the alloy (Supplementary Information gives an example of such a fracture surface). This problem was alleviated by giving the beams a trapezoidal (tapered) cross section, with the wider side of the beam subjected to tension during the test. This alters the stress distribution, causing tensile stresses to decrease as one approaches the edge of the beam. Fig. 1 illustrates this by showing results of finite element simulations, conducted as described below, on two beams: one with a near-rectangular cross section and the other with a cross section typical of tests conducted here. As seen, whereas in the former the first principal stress is uniform along the X axis, in the latter the stress at the mid-span edge is 10% lower than the peak stress and decreases rapidly in the Z direction. This is representative of all specimens tested in this work: the minimum difference in computed stress between the edge and the peak stress in the centre was always between 10% and 20%. With such trapezoidal beams, thus, the region of the samples that is exposed to peak values of applied tensile stress during the test does not include material that was altered by focused ion beam milling, which is situated along the sidewalls of the sample. This is an important feature of the present test method, which we describe in more specific detail in the following sections.

We note in passing that such trapezoidally tapered specimens are often used in fracture toughness testing; however, in such tests the taper is oriented the other way around (i.e. with the narrower end at the location of peak tensile stress). This is practiced where crack growth stability is sought, since in this orientation, as the crack advances, its front broadens, decreasing the driving force.