Available online at www.sciencedirect.com
ScienceDirect
Acta Materialia 58 (2010) 439–448
Acta MATERIALIA
www.elsevier.com/locate/actamat

Stronger silicon for microsystems

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Received 22 January 2009; received in revised form 4 September 2009; accepted 9 September 2009
Available online 8 October 2009

Abstract

Few studies have deliberately varied the microstructure of microfabricated polycrystalline silicon (polySi) to examine their effects on resulting mechanical performance and reliability. In the present study, the tensile strength distributions of four microfabricated polySi variants were examined, corresponding to two different grain sizes (285 nm vs. 125 nm) in both the undoped and heavily P-doped conditions. Microtensile tests revealed that the coarse-grained materials exhibited significantly lower characteristic strengths (1.48–1.76 GPa) compared to the fine-grained material (2.80–2.83 GPa). The difference in strength was attributed largely to preferential etching of grain boundary grooves that were considerably more pronounced in the coarse-grained material. The presence of phosphorous doping had a less pronounced effect on strength values, lowering the characteristic strength of coarse-grained material by merely 16% and having little or no effect on the fine-grained material.
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Keywords: Lithography; Chemical vapor deposition; Fracture; Tension test; Ultrafine-grained microstructure

1. Introduction

Much of the prior work on the development of MEMS processing routes has focused on fabricating structures with controlled dimensions and minimal residual stress. The processing techniques, derived largely from the microelectronics industry, have not been optimized with respect to structural properties such as modulus, fracture strength, fatigue resistance, and fracture toughness. The present study examines possible processing pathways to improve the tensile strength of polycrystalline silicon (polySi). The motivating hypothesis for this work is that the tensile strength of polySi can be improved by identifying processing conditions to minimize the extent of critical defects. Specifically, our initial expectation based on preliminary experiments and existing literature was that small changes in the microstructure and/or doping conditions could improve polySi's mean strength by a factor of two or more by affecting the formation and size of failure–critical defects. Establishing connections between processing conditions, microstructure, critical defects, and structural reliability may assist in the future development of highly reliable MEMS materials.

Prior work on the fracture strength of polySi has largely been limited to a few established processing methods such as the SUMMiT V^{TM} process at Sandia National Labs and the PolyMUMPs process at MEMSCAP, Inc. For this reason, there have been relatively few studies which explicitly vary the microstructure (i.e. grain size, distribution and crystallographic texture) of microfabricated silicon to examine the effects of microstructure on properties such as fracture strength. An important exception is the work of Ballarini and co-workers [1], who compared the bending strength of amorphous silicon (a-Si) and polySi with a 100 nm grain size. They found that, on average, a-Si was nearly twice the strength of the polySi (9.7 GPa compared to 4.9 GPa average strength). This study highlights the notion that microstructure plays an important role in determining the resulting mechanical properties. Microstructure governs fracture strength via three possible avenues:

(1) by modifying the material's resistance to fracture, i.e. altering the toughening mechanisms;

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doi:10.1016/j.actamat.2009.09.022

(2) by imparting local intragranular stress risers, most pronounced near grain boundaries and triple junctions, due to the constraining elastic crystallographic anisotropy of neighboring grains in the polycrystal;
(3) by affecting the size, shape, distribution, or nature of failure-critical defects.

1.1. Resistance to fracture

In Ballarini's comparison of a-Si and polySi, fracture toughness was measured for both materials. In spite of their very different average strength values, the average fracture toughness of these two materials was found to be statistically indistinguishable, $\sim$1.0 MPa$\sqrt{\text{m}}$, suggesting that microstructure may not play an important role in toughness in this particular case. As a counter-example, Cho et al. [2] experimentally measured the fracture toughness values of several polysilicon tensile bars that had been fabricated by MEMSCAP's Multi-User MEMS Processes (MUMPS) and precracked by radial corner cracks extending from nearby indentation in the sacrificial oxide prior to release. They found that the fracture toughness from specimen to specimen varied by 50%, from 0.843 to 1.225 MPa$\sqrt{\text{m}}$. This large variation in fracture toughness was attributed to local crack tip variations in crystallographic orientation, grain boundary toughness, and crack tip shielding conditions. Numerical cohesive zone modeling by Foulk et al. [3] of the grain-bridging effect in brittle microstructures also illustrates various scenarios where grain boundaries can influence the material's macroscopic toughness.

Under most typical silicon MEMS processes, doping is not expected to have a substantial effect on fracture toughness. Cook has recently shown, through indentation toughness experiments, that neither heavy *n*-type P-doping or *p*-type B-doping has a substantial effect on silicon's intrinsic toughness [4]. Although doping levels can affect dislocation mobilities, there is little reason to believe that diffusionally driven dopant content would have a dramatic effect on the intrinsic toughness, unless the dopant poisons the grain boundaries causing a transition from transgranular to intergranular fracture. Son et al. [5] showed that the doping process can, under other conditions, have a notable effect on both fracture strength and fracture toughness. In comparing boron implantation, phosphorous implantation, and $POCl_3$ diffusion, they found that the diffusion-doped material had the highest strength and fracture toughness. They suggest that the implantation method causes ion damage to the lattice, lower intrinsic toughness, and hence lower strength, which pervades even if the films are well annealed.

1.2. Local microstructural stresses

In a separate work, Ballarini et al. [6] simulated the role of polycrystalline elastic anisotropy associated with random crystallographic texture in an equiaxed Poisson–Voronoi tessellated microstructure on the local strain energy release rate at the tip of a long crack. They found that materials with strong elastic anisotropy and non-cubic crystal symmetry could be sensitive to the local microstructure with energy release rates affected by up to 40% in extreme cases. However, for cubic materials with weak elastic anisotropy such as silicon, the elastic anisotropy of the microstructure was found to have a negligible effect on the local crack tip stresses or driving force for failure.

1.3. Critical defects

The critical strength-limiting defects in most brittle MEMS materials are surface flaws induced by either the chemical etching process [7] or annealing. In polySi, grain boundaries can be sites for critical defects [8]. Grain boundary grooves, also called grain boundary depressions [8], are the result of either localized preferential etching or thermal surface diffusion [9] and often represent the largest surface flaws. In single-crystal Si, Alan et al. [10] showed that the addition of a tetramethylammonium hydroxide (TMAH) etch after a standard potassium hydroxide (KOH) etch reduced the root-mean-squared surface roughness from 1.5 nm to 0.4 nm. This reduction in surface roughness increased the strength of the KOH + TMAH etched material by $\sim$25% over the baseline strength of the KOH-etched material. Similarly, Miller et al. [11] showed that the average strength of MEMSCAP SOIMUMPS single-crystal silicon could vary by 50%, depending on the extent of etch-induced sidewall and edge defects.

The etch-induced surface defect structure and oxide thickness of both single-crystal and polycrystalline silicon has been shown to be strongly affected by anodic oxidation/galvanic corrosion when silicon is in intimate contact with a metal layer such as gold [12–15]. This galvanic corrosion can result in a highly porous, extensively oxidized surface layer, and the extent of surface damage has been shown to correlate well with observed degradation in the fracture strength, which can be reduced by >90% under extreme conditions [16,17].

Doping may also affect the size and shape of critical defects. Dopant content has long been known to affect etching or thermal grain-boundary grooving processes (see for example Ref. [18]). With few exceptions, the presence of dopant exacerbates the defect formation process, especially when the dopant is locally segregated to grain boundaries.

2. Method

2.1. Polysilicon microfabrication

The polysilicon materials for the present study were produced at Sandia National Laboratories' Microfabrication Facility in Albuquerque, NM. Four wafers were examined

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