

Improvement in smoothness of anisotropically etched silicon surfaces: Effects of surfactant and TMAH concentrations

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Abstract

We investigated the anisotropic etching properties of single-crystal silicon using tetramethyl-ammonium-hydroxide (TMAH) water solutions containing poly-oxethylene-alkyl-phenyl-ether (NC-200) as a surfactant. When the surfactant was added at 0.1% of the total volume of the etchant, the etched surface morphologies drastically changed, along with the anisotropy of the etching rate. We found that by using the surfactant at the low TMAH concentration region, a smooth mirror-like surface can be etched in both (1 0 0) and (1 1 0) orientations simultaneously. Although the addition of the surfactant reduces the etching rate, we show how this procedure can be used to improve the roughness of an etched surface without significantly increasing the overall processing time.

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1. Introduction

Anisotropically wet etched surface morphology is important when fabricating microstructures on single-crystal silicon. The roughness of micro-machined surfaces influences the functionality of micro-system devices. One way of changing the etching anisotropy of single-crystal silicon is to use different additives in the etching solutions. It is empirically known that some surfactants [1,2] and alcohols [3,4] are effective at improving the smoothness of etched surfaces and decreasing the amount of undercutting. We have reported the anisotropy in the etching properties of silicon for a number of orientations by using 25% TMAH water solutions containing different surfactants belonging to the NC series (NC-100, NC-200 and NC-300, products of Lion Co.) [5]. Although creating a smooth mirror-like surface in silicon (1 0 0) was easy, doing the same in (1 1 0), whose surface is usually covered with a striped zigzag morphology, was difficult.

In this work, we measured the anisotropic etching properties of single-crystal silicon using different concentrations of

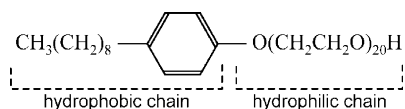
TMAH water solutions containing the surfactant NC-200. In the low TMAH concentration region, we obtained a smooth mirror-like surface in both the (1 0 0) and (1 1 0) orientations simultaneously.

2. Experimental method

To investigate the effects of the surfactant NC-200 on the etching properties of single-crystal silicon in different concentrations of TMAH water solution, we used two types of samples: wafer chips and a hemispherical specimen [6]. We used silicon chips with (1 0 0), (1 1 0) and (1 1 1) surface orientations to evaluate the effects of the surfactant on the etching rates and the etched surface morphologies. The etching rates were measured from the etching depth using a surface profiler DEKTAK3 ST (VEECO). The etched surface roughness was measured using a non-contact 3D optical profiler ZYGO (Zygo Co.) and an optical microscope. We also used hemispherical single-crystal silicon specimens to determine the etching properties of all crystallographic orientations [6]. Etching rates were calculated from the change in the surface profile of the specimen before and after etching. The measurements were made using a 3D surface-measuring machine UPMC550CARAT (CARL ZEISS Co.) with a mechanical contact probe.

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The surfactant NC-200 is nonionic containing 100% poly-oxethylene-alkyl-phenyl-ether. The structural molecular formula is:



One end of the molecule is hydrophilic, and the other is hydrophobic.

The etching conditions were as follows:

- 5–25% TMAH water solution,
- concentration of NC-200: 0.01–1.00% volume of total solution. Normally 0.1%,
- etching temperature: 60 °C.

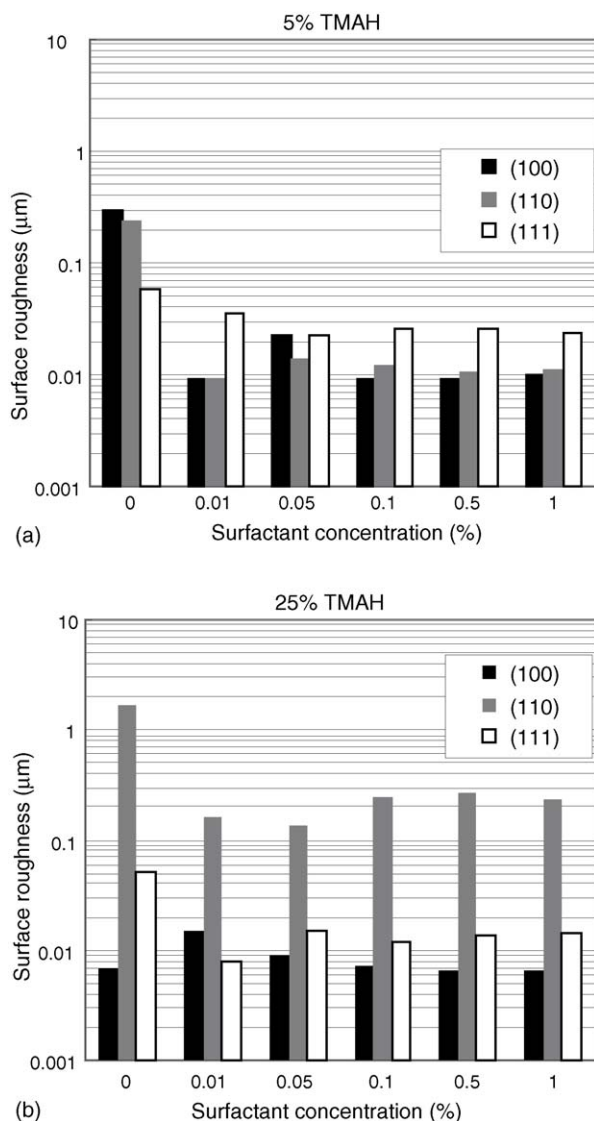


Fig. 1. Effect of surfactant addition on surface roughness for the three principal orientations. Etching temperature: 60 °C. Etching time: 4 h.

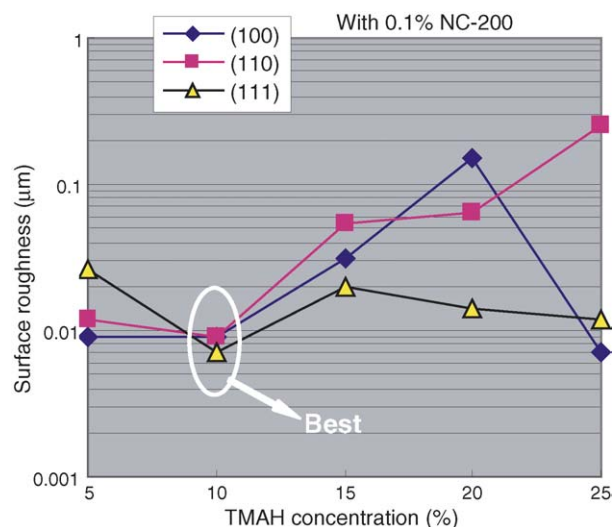


Fig. 2. Dependence of roughness of the three principal orientations on TMAH concentration with 0.1% NC-200 addition. Etching temperature: 60 °C. Etching time: 4 h.

3. Results

The addition of the surfactant reduced the etched surface roughness for the three principal orientations, as shown in Fig. 1. The initial surface roughness of the specimens was 2–5 nm. The etching time was 4 h. The surface roughness of silicon (1 1 0) decreased remarkably by adding the surfactant at both low (5%) and high (25%) TMAH concentrations. Although the (1 0 0) surface showed a similar decrease in roughness due to adding the surfactant at a low TMAH concentration, such an effect was only slightly observed at the higher TMAH concentration. On the other hand, the roughness of the (1 1 1) surface was not affected by the surfactant at either low or high TMAH concentrations. Since the roughness of (1 1 1) and (1 0 0) surfaces at high TMAH concentrations is already quite minimal, further improvement in smoothness is difficult. The required amount of surfactant necessary for the effects was quite small, reaching saturation already at 0.1% of volume concentration. The change in roughness for the three principal orientations in different TMAH concentrations with 0.1% NC-200 added is shown in Fig. 2. Roughness was reduced in the lower TMAH concentrations. In 10% TMAH, the roughness was best for all three principal orientations simultaneously.

Although the surface roughness of (1 1 0) decreased at high TMAH concentrations after adding the surfactant (Fig. 1(b)), the roughness was still higher than that at low TMAH concentration with the surfactant (Fig. 1(a)). The surface morphologies of silicon (1 1 0) etched in 5% (low) and 25% (high) TMAH with and without 0.1% NC-200 are compared in Fig. 3. For the low TMAH concentration, the surfactant changed the surface morphology from zigzags (Fig. 3(a)) to a smooth, mirror-like finish (Fig. 3(b)). However, for the high TMAH concentration, the typical linear stripe structures characteristic of (1 1 0) persisted (Fig. 3(c) and (d)), although

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