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# Characterization of anisotropic wet etching properties of single crystal silicon: Effects of ppb-level of Cu and Pb in KOH solution

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**Abstract**

We investigated the effect of ppb impurity level of Pb and Cu in KOH solution on the etching characteristics of Si for a number of crystallographic orientations using a hemispherical specimen. With the ppb-level of Cu, the location of the maximum etch rate shifted from the vicinity of {2 1 1} to {1 1 0}, i.e. the anisotropy changed, while the etched surface became rough showing fine textures regardless of the orientations. With the addition of the ppb-level of Pb, the etch rate decreased at almost all orientations, while the morphology of all etched surfaces did not show any substantial change. Because the ppb-level of Pb in KOH solution reduced the etching reaction uniformly for all orientations, the anisotropy in the etch rate did not change. These results support our hypothesis that: (i) Cu in the solution deposited onto the Si surface as fine particles and acted as etching masks, and (ii) the reduction–ionization of Pb is in competition with the etching reaction of Si and leads to a change in the etch rate.


*Keywords:* Silicon; Anisotropic etching; Impurity; Etch rate; Surface roughness

## 1. Introduction

Anisotropic wet chemical etching of Si in alkaline solutions is a key technology for fabrication of Si microstructures such as diaphragms for pressure sensors or cantilevers for acceleration sensors. A smooth surface of Si after etching and a constant etch rate lead to the precise and stable production of these sensors [1,2]. However, the etching characteristics are unstable under certain conditions. Many studies have been done to control the etching properties. Campbell et al. observed the change of Si{1 0 0} surface roughness in KOH solutions with a variety of chemical reagent makers, of which the purity differed [3]. Hein et al. investigated the effects of the ppm to percentage level of metal impurities on the etching of Si{1 0 0} [4]. We have previously reported that the ppb-level of Cu in KOH solution roughens both Si{1 1 0} and {1 0 0} surfaces, and the etch rate of Si{1 1 0} and {1 0 0} is affected both by the ppb-level of Cu and Pb [5,6].

In addition, Monte Carlo simulations by Gosalvez and Nieminen showed that the variety of micron-scale features observed in the orientation-dependent surface morphology during etching had its origin at the atomistic scale and the pyramidal hillocks on Si{1 0 0} was the result of local stabilization of distributed apex-atoms by metal impurities from solution [7]. In this work, we further investigated the separate effect of Pb and Cu on the order of hundreds of ppb in KOH solution on the etching characteristics of Si for a number of crystallographic orientations using a hemispherical specimen experiment, an original technique developed by Sato and Shikida [8,9].

## 2. Experimental method

### 2.1. Specimen

We used a hemispherical single-crystal silicon specimen to evaluate the effect of the ppb-level of Cu or Pb in KOH solution for a number of crystallographic orientations as shown in Fig. 1. The surface was initially polished into a mirror, whose roughness was 0.005–0.007 mm (Ra). The radius of the hemisphere was 22 mm, and its sphericity was less than 10 mm. Etch rates were calculated from the change in the hemispherical specimen by

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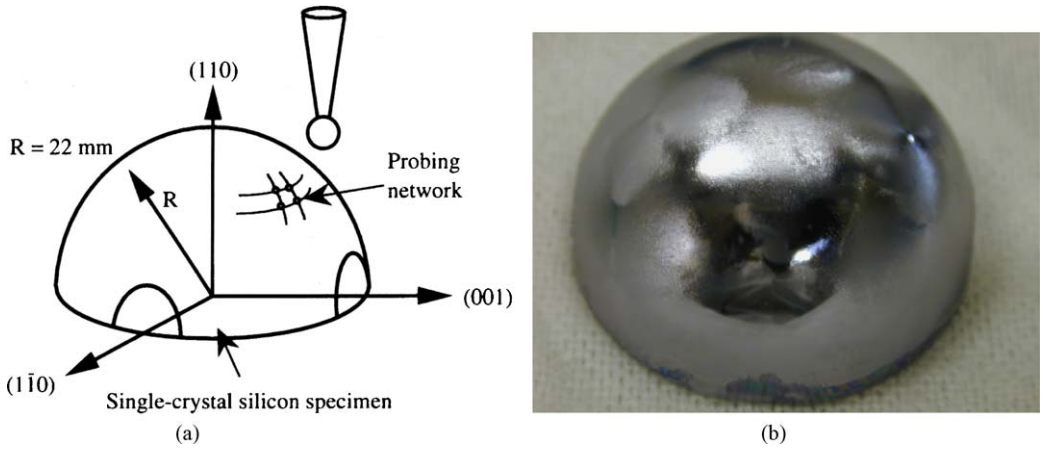


Fig. 1. Hemispherical single-crystal silicon specimen to evaluate for a number of crystallographic orientations: (a) schematic diagram and (b) photograph of the specimen after etching.

measuring the profile before and after etching. The spherical profile measurement was carried out using a three dimensional measuring machine (UPMC550-CARAT (Carl Zeiss)).

### 2.2. Etching condition

The specimen was etched at 110 °C in 34 wt.% KOH without addition, with addition of 360 ppb Pb and with addition of 360 ppb Cu. The etching treatment was performed in a polytetrafluoroethylene (PTFE) vessel with heater. In order to prevent the change of KOH concentration due to evaporation, the vessel was covered with the PTFE plate as shown in Fig. 2. Pb or Cu ions were added to the solution as nitrates, respectively. Nitrates used were commercially available reagents supplied for atomic absorption analysis.

## 3. Results

### 3.1. Etch rate

Fig. 3 shows the contour maps of etch rates without addition, with 360 ppb Cu and with 360 ppb Pb. The differences of the etch rate between no addition and 360 ppb Cu or 360 ppb Pb are shown in Fig. 4. Fig. 5 shows the change of etch rates from the top of the hemisphere, i.e. the {1 1 0} plane, to the {1 0 0}, {3 1 1}, {3 1 2} and {1 1 1} plane without addition and with addition of 360 ppb Pb or 360 ppb Cu.

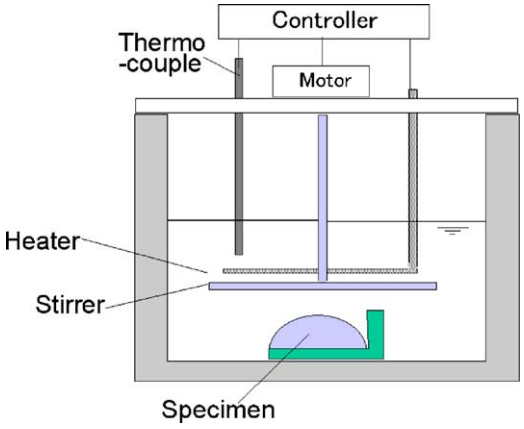


Fig. 2. Experimental apparatus for etching.

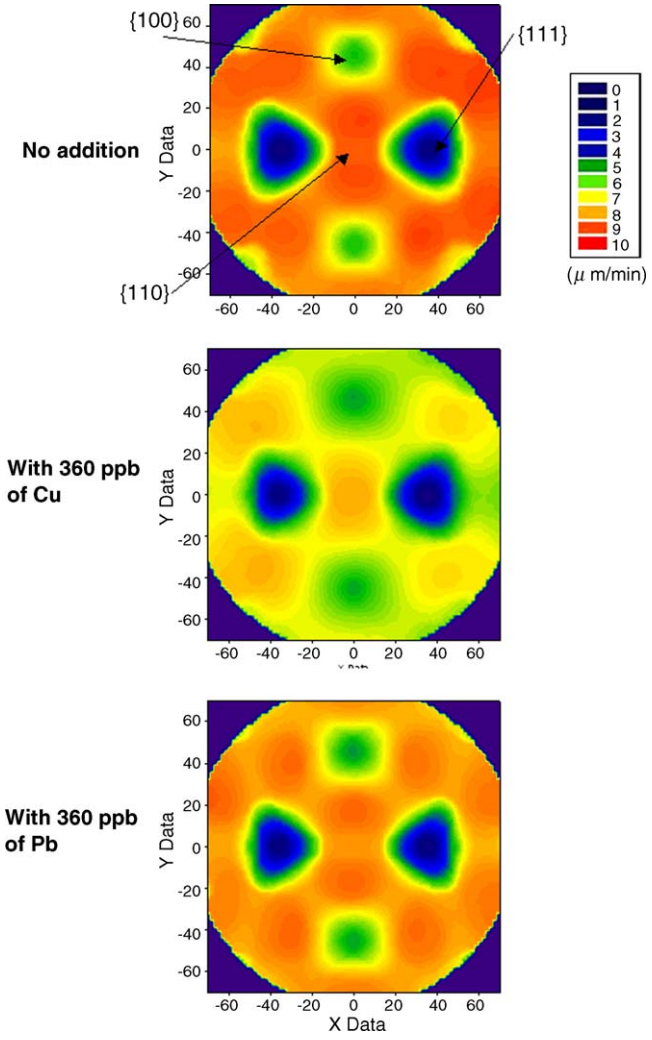


Fig. 3. Contour maps of etch rate without an addition, with 360 ppb Cu and with 360 ppb Pb.