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Study on variations in ductile-brittle transition points at nano-scale mechanical machining by thin film

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Abstract
In the semiconductor industry, single-crystal materials with uniform properties are widely used. Due to their brittle characteristics, these materials are prone to brittle fracture and typically processed using lithography rather than mechanical machining commonly used in material processing. Recent studies have shown that ductile machining of single-crystal materials is possible by applying extremely low loads to form nano-scale patterns. However, the critical load at which brittle fracture occurs in single-crystal materials is ultra-low, limiting the size of patterns that can be manufactured. We investigated how a ductile material on single-crystal silicon can increase the thrust force at the ductile-to-brittle transition and thus expand the range of ductile machining. We compared the critical thrust force at which brittle fracture occurs between uncoated single-crystal silicon and single-crystal silicon coated with approximately 150 nm of Ag ductile thin film. The Ag thin film was transferred at 70°C and 90°C respectively, resulting in different adhesion. We conducted mechanical machining using a nanoscratch tester capable of applying precise ultra-low loads, employing a 90° conical single-crystal diamond machining tool. After machining, we observed the machined nano-scale patterns using a scanning electron microscope (SEM) to evaluate the points of ductile-brittle transition The ductile-to-brittle transition of single-crystal silicon coated with the Ag thin film occurred at significantly higher thrust force. When comparing the pattern width, the uncoated silicon showed approximately 2.20 µm while the single-crystal silicon coated with Ag thin film at 90°C exhibited approximately 2.81 µm. The specimen coated at 70°C, which had a higher adhesion, showed a pattern width of 2.89 µm. These results indicate that the range of ductile machining patterns can be broadened through a process of coating a ductile thin film, followed by its removal after machining.
Ductile machining, Ductile-to-brittle transition, Single-crystal silicon, Nano-scale, Adhesion

1. Introduction

Single-crystal silicon is extensively utilized in various fields, including semiconductors and displays[1], due to its uniform properties, such as excellent electrical conductivity[2], optical properties[3] and low defect density[4]. However, its high brittleness makes it prone to brittle fractures, leading to a reliance on etching processes for pattern fabrication instead of conventional machining methods, despite their high productivity.

Recent studies on single-crystal silicon machining have revealed that ductile machining without brittle fracture is achievable under extremely low applied loads, demonstrating the feasibility of machining single-crystal silicon[5,6]. Nevertheless, the critical load for machining remains much low, limiting the size of the fabricated patterns.

To overcome this limitation, this study aims to broaden the range of ductile machining by delaying the ductile-brittle transition through coating a ductile thin film onto the surface of single-crystal silicon. The coated thin film serves as a temporary layer to enhance ductile machining and can be removed after processing if necessary. Specifically, an Ag thin film was coated on single-crystal silicon, and machining was performed using a nanoscratch tester which has a similar working principle to an ultra-fine machining system. The thrust force at the ductile-brittle transition point was regarded as the range of ductile machining.

2. Experimental methods

The two coated single-crystal silicon specimens were fabricated by coating a 150 nm thick Ag thin film onto the surface of single-crystal silicon coated with (3-trimethoxysilyl)propylethylenediamine, classified as an adhesive self-assembled monolayer (SAM) at 70°C and 90°C. The single-crystal silicon wafer is referred to as uncoated Si.

The coated Si and uncoated Si were intended to be machined within the load range that would not induce brittle fracture in single-crystal silicon. However, conventional machining equipment has limitations in applying ultra-low loads and lacks superior load resolution; instead, a nanoscratch tester, which offers micro-newton load resolution, was used to perform ultra-fine machining as shown in Figure 1 under the conditions specified in Table 1. Additionally, thrust force was measured with high resolution during machining, allowing for the analysis of machining behavior at the nanoscale. Each machining was conducted three times.

After the ultra-fine machining, the machined patterns were observed using a Scanning Electron Microscope (SEM) to decide the points of ductile-brittle transition. The thrust force at the ductile-brittle transition point could be defined as the range of ductile machining. The pattern width at this point were the maximum pattern size. The average value were used to ensure reliability of the results.

Table 1 Parameters for Ultra-Fine Machining

Machining tool	90° conical single-crystal diamond tool
Machining direction	[110] crystalline orientation
Loading mode	Progressive
Maximum normal load	50 mN
Machining length	1 mm
Machining speed	1 mm/min

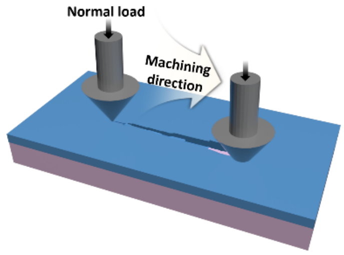

Figure 1. Schematic diagram of machining using a nanoscratch tester

3. Results and discussion

Observation of the machined patterns using SEM revealed that all three specimens underwent ductile machining under lower normal load. However, when the normal load increases beyond a certain level, a sudden brittle fracture occurs, as shown in Figure 2. This leads to the discontinuous coexistence of brittle fracture and ductile machining. Since it represents the transition point from ductile machining to brittle fracture, this point can be identified as the ductile-brittle transition. The average thrust force at these points were 19.91 ± 0.26 mN for uncoated single-crystal silicon, and 29.59 ± 1.09 mN (process temperature: 70°C) and 25.90 ± 3.50 mN(process temperature: 90°C) for coated single-crystal silicon, as shown in Figure 3(a). This indicates that the ductile-brittle transition occurred at a significantly higher thrust force in coated single-crystal silicon compared to uncoated single-crystal silicon. This result suggests that the adhesion between the thin film and single-crystal silicon suppressed crack propagation, thereby delaying brittle fracture in the single-crystal silicon.

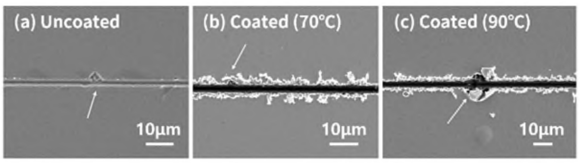

Figure 2. SEM images of the ductile-brittle transition points of (a) Single-crystal silicon, (b) Single-crystal silicon coated with Ag at 70°C, and (c) Single-crytal silicon coated with Ag at 90°C (All arrows indicate the first brittle fracture point)

Within the coated silicon specimens, the silicon coated at 70°C exhibited slightly higher thrust force at the ductile-brittle transition points compared to the silicon coated at 90°C. When the Ag thin film is coated on silicon at high temperatures, residual stress occurs during the cooling process after coating due to the difference in the thermal expansion coefficients between the two materials. This residual stress reduces the adhesion, and as the process temperature increases, the residual stress becomes larger. Consequently, the adhesion of the specimen coated at 90°C is slightly lower than that of the specimen coated at 70°C. Therefore, the thrust force at the ductile-brittle transition point decreases as the coating temperature increases.

The delay in the ductile- brittle transition is expected to expand the range of machinable pattern sizes. The average pattern width at the ductile-brittle transition are presented in Figure 3(b). For the coated specimens, the pattern width that can be machined increased by approximately 30%. There also were slight variations in the pattern width depending on the processing temperature, which were attributed to the effects of residual stress. This study demonstrates that the range of nanoscale machining for single-crystal silicon can be expanded through a process of coating a ductile thin film, followed by the removal of the film after machining. Future studies will investigate replacing the coating with a more cost-effective material.

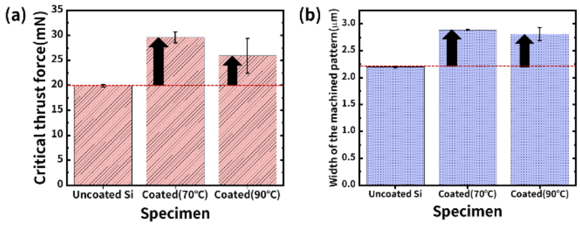

Figure 3. Variations of (a) thrust force at the ductile-brittle transition points and (b) pattern width at the ductile-brittle transition points

4. Conclusions

In this study, by coating the surface of single-crystal silicon with an Ag thin film, the ductile-brittle transition was delayed, thereby broadening the range of ductile machining and pattern size. The detailed results are as follows:

1) Both uncoated and coated Si exhibited brittle fracture; however, the ductile-brittle transition of the coated Si was delayed, resulting in brittle fracture occurring at higher thrust forces.
2) The silicon coated at 70°C presented slightly higher thrust force compared to the silicon coated at 90°C, which can be attributed to the increase in interfacial residual stress caused by higher coating temperatures.
3) As the ductile-brittle transition was delayed, the pattern width of coated Si capable of ductile machining increased by approximately 30%. This result indicates that the machinable range of single-crystal silicon can be expanded through ductile thin film coating and its subsequent removal.

Acknowledment

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