

Nano micro-scratch machining by atomic force microscope (AFM) for investigating fundamental characteristics of polishing

Shinsuke Matsui¹⁾ and Ken-noshin Kimura¹⁾

¹Chiba Institute of Technology

Matsui.shinsuke@it-chiba.ac.jp

Abstract

Nano-micro scratch machining by atomic-force-microscope (AFM) with various tip materials was applied for investigating the fundamental characteristics of polishing. The AFM tip can be assumed to a nano-shaped cutting edge of a polishing abrasive. The load and trajectory of the machining tip are controlled by the AFM system. 512 scratches with 0.04- μm intervals were machined on a 20 \times 20- μm area on the end plane of an optical fiber (made of pure SiO₂). SiO₂, Al₂O₃, ZrO₂, CeO₂, and diamond were used as AFM tip materials to simulate a polishing abrasive. The machining environment was water or alkaline solution (with pH of 10). Therefore, chemical aspects (which are important in regard to polishing) could be studied. Furthermore, the amount of machining damage was estimated by measuring the optical return loss of the optical-fiber end. Amount of wear of the tips was measured with FE-SEM. The FE-SEM results reveal that the diamond and CeO₂ tips in pure water machine the deepest scratched area. Although the diamond tip causes large subsurface damage, the CeO₂ tip causes very little subsurface damage. Moreover, it machines deeper scratches under the higher-pH condition by the SiO₂ tip. However, the SiO₂ tip causes very little subsurface damage in the higher-pH condition. The amount of abrasion on the SiO₂ tip is about the same as the amount of machining. On the other hand, the amount of abrasion on the CeO₂ tip is 37 times lower than the amount of machining. It is therefore concluded that AFM nano-scratch machining simulates the chemical aspects of polishing very well.

Atomic force micrometer, optical fiber, polishing and machining damage

1. Introduction

Atomic-force-microscope (AFM) nano-scratch machining for fundamental polishing characterization has been previously studied [1], [2]. The advantages of using an AFM for machining are available a well-defined cutting edge, trajectory, and process load. This so-called "nano-micro machining" is thought to reveal the elementary steps of the polishing, whose mechanism is not yet well understood [4]. As a specimen, the end plane of an optical fiber, made of highly pure silica glass, is used. The chemical aspects of the polishing can also be investigated with various tip materials under varied scratching atmospheres. Furthermore, an optical-fiber retro-reflection measurement can be used to characterize the subsurface machining-damage layer by machining the core part of the optical-fiber end plane.

In this study, SiO₂, Al₂O₃, ZrO₂, CeO₂ and diamond are used as tip materials. Among these materials, diamond and Al₂O₃ are harder than SiO₂. CeO₂ and ZrO₂ abrasives, which have similar hardness as the SiO₂ specimen, are said to have fairly high polishing rate due to chemical action. Moreover, the influences of environmental liquid, tip abrasion, and subsurface damage are investigated.

2. Experiments

AFM scratch machining of the optical-fiber end is shown in Fig. 1. A 20 \times 20- μm square area of the end plane of the optical-fiber (including the optical core located at the center of optical fiber) was scratch machined. This core (with diameter of ten μm) is the region of light-signal propagation in a single-mode

optical communication fiber. The scratching area consists of 512 scratch lines. Scratch speed was one second per line. liquid environment of pure water and KOH-dissolved alkaline water (pH=10) was prepared by. The subsurface damaged layer was characterized by measuring retro-reflection of the end plane of the optical- fiber. Previous studies [3] showed that the optical

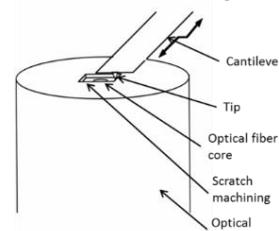


Fig. 1: Schematic diagram of scratch machining on optical-fiber end

index of the diamond silica glass of the subsurface damaged layer was from 1.45 to 1.53 in spite of the variation of the diameter of the diamond core. In contrast, increasing the abrasive diameter increases the thickness of the damaged layer. These increases cause various return losses in the case of various abrasive diameters.

The cutting-tool tips are made of SiO₂, Al₂O₃, ZrO₂, CeO₂, or diamond. Curvature radii of the tips were 0.2 μm for SiO₂, Al₂O₃, ZrO₂, and CeO₂ and 0.1 μm for diamond. Applied load was 80 μN , which is higher than that used in a previous experiment, so that tip wear could be measured more accurately. [2] The tips also were measured with FE-SEM (field-emission scanning electron microscope) before and after scratch machining to estimate the amount of tip wear.

3. Results

Machining depths and optical return losses for various tip materials are listed in Table 1. The scratch atmosphere is pure water. Before machining the end planes of the optical fibers were finished by fine silica polishing. The optical return losses are from 55 to 57 dB, which can be said to be almost no machining damage. The diamond tips machine to depths of 4.2 nm with return loss of 40.8 dB on average. This decrease in return loss suggests the machining damage causing by silica material plastic deformation. On the other hand, the return loss of the planes machined with the CeO₂ tip is 56.3 dB, though it machines deeper than the diamond tip. Machining

Table 1: Machining characteristics with various tip materials in pure water (applied load: 80 μN)

Tip material	Abrasive depth (nm)	Optical return loss (dB)
Diamond	4.2	40.8
Al ₂ O ₃	0.5	54.7
SiO ₂	2.8	52.4
CeO ₂	4.4	56.3
ZrO ₂	1.7	55.5

Table 2: Machining characteristics with various tip materials in alkaline water (pH=10) (applied load: 80 μN)

Tip material	Abrasive depth (nm)	Optical return loss (dB)
Diamond	5.1	40.0
SiO ₂	12.2	58.0
Al ₂ O ₃	2.9	51.4

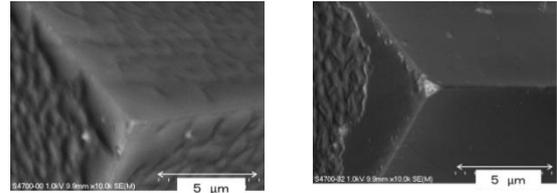
with the silica tip only reaches an abrasive depth of 2.8 nm with return loss of 55 dB. In this case, very little damage is generated under the machined plane. Furthermore, the alumina tip produced no scratch machining, so the return loss was unchanged.

The scratch-machining characteristics in high-pH liquid (pH=10) are listed in Table 2. The silica tip achieves deeper scratching to a depth of 12.2 nm. At pH 10, the silica surface slightly dissolves and forms HSiO³⁻ ions. It is thus supposed that the combination of this dissolution effect and scratch machining form a deeper scratched area. The diamond tip machined to a depth of 5.1 nm with return loss of 40 dB. The depth and return loss are similar to those in the case of pure water. It seems that the pH-10 liquid hardly influences the diamond machining compared to the silica machining. It is also revealed that the Al₂O₃ tip machines to a greater depth (2.9 nm) than that (0.5 nm) by machining in pure water. The return loss is 51.4 dB. Because machining depth with the 30-μN diamond tip is 2.9 nm, and return loss is 46 dB, the Al₂O₃-tip machining in pH 10 causes less damage to silica.

An Al₂O₃ tip before machining and after machining in 10 pH is shown in Figure 2. The figure confirms that the tip was well prepared (Figure 2(a)). The curvature radius of the tip is about 0.2 μm. The Al₂O₃ tip after machining in pH of 10.0 is shown in Figure 2(b). The figure reveals that abrasion is caused on the top of the tip.

Scratch-machining amount and tip-abrasion amount in pure water and pH of 10 are compared in Table 3. With the diamond tip, abrasion does not occur. In the case of the silica tip, the machining amount is about the same as the abrasion amount. Especially, at pH 10, they are nearly equal. In these cases, the

specimen material and the tip material are the same. It thus seems that the machining proceeds by the tip and work materials wearing together. Accordingly, very little subsurface damage occurs. On the other hand, in the case of



(a) before machining (top view) (b) after machining (top view)
Fig. 2: Al₂O₃ tip wear in pH-10 atmosphere

Table 3: Machining amount and tip-abrasion amount

atmosphere	Tip material	Machining amount (mol) (×10 ⁻¹⁴) A	Abrasive amount (mol) (×10 ⁻¹⁴) B	Ratio A/B
Pure water	Diamond	8.6	0	-
	Al ₂ O ₃	0.88	0.02	44
	SiO ₂	4.9	1.9	2.6
	CeO ₂	7.7	0.21	37
	ZrO ₂	3.0	0.63	4.76
pH = 10.0	SiO ₂	22.1	22.2	1.0
	Al ₂ O ₃	4.7	0.56	8.4

the CeO₂-tip machining, the machining amount is 37 times larger than the tip abrasion. In account of this result concerning CeO₂, namely, large scratch depth, very little subsurface damage, and hardness close that that of silica, it is concluded that the machining of silica by the CeO₂ tip proceeds under significant assistance of some kind of chemical action. Furthermore, in the case of Al₂O₃-tip machining, tip abrasion and machining occur, and the ratio of their respective amounts is 8.4. This result also suggests that some kind of chemical action also plays a role in machining in this case.

4. Conclusion

Atomic-force-microscope (AFM) nano-scratch machining was applied for characterizing fundamental polishing. As a result, it is revealed that the SiO₂, ZrO₂, and CeO₂ tips machine silica material while causing very little subsurface damage. The amount of abrasion on the SiO₂ tip is about the same as the machining amount. On the other hand, the amount of abrasion on the CeO₂ tip is 46 times lower than the machining amount. It is also revealed that the AFM nano-scratch machining simulates the chemical aspects of polishing very well.

Acknowledgment

This work was supported by JSPS KAKENHI Grant Number JP15K05731.

References

- [1] Matsui S, Ohira F, S. Umemura, K. Matsunaga, and K. Koyabu, Precision Science and Technology for Perfect surface, (1999) pp. 893.
- [2] S. Matsui and J. Kobayashi: Characterization of damaged layer of optical fiber end by AFM scratch machining, Journal of Abrasive Technology (in Japanese), **54** No. 10 (2010) pp. 607
- [3] S. Matsui, F. Ohira, K. Matsunaga, and Y. Kikuya: Proc. of 7th International Precision Engineering Seminar, (1993) pp. 699
- [4] L. M. Cook: Chemical processes in glass polishing, Journal of NonCryst. Solids, **120** (1990) pp. 152
- [5] N. Ozawa, M. Ishikawa, M. Nakamura, and M. Kubo: Polishing Process Simulation of SiO₂ by CeO₂ Abrasive Grain under Wet Environment, Surface Science, **33** (2012) pp. 351 (in Japanese)