Nanoindentation by Using a Newly Developed Nanomachining Instrument

Wei Gao* , Robert Hocken**, John Patten**, John Lovingood**, and Don Lucca***

* Department of Mechatronics and Precision Engineering, Tohoku University, Sendai, JAPAN
** Center for Precision Metrology, The University of North Carolina at Charlotte, Charlotte NC
*** School of Mechanical and Aerospace Engineering, Oklahoma State University, Stillwater OK

INTRODUCTION

Single-point diamond turning (SPDT), which was originally developed for military use, has been improved to an ultra-precision machining technology for mass production of precision parts of ductile materials, such as computer hard disk substrates, print drums, polygon mirrors, etc. Recently, great efforts have being made to extend diamond turning to brittle materials such as germanium, silicon and even ceramics, and to push SPDT technology to a higher level of nanometer form accuracy and atomic surface finish. For this purpose, nanocutting experiments need to be carried out to understand the machining mechanism in the nanometer range[1].

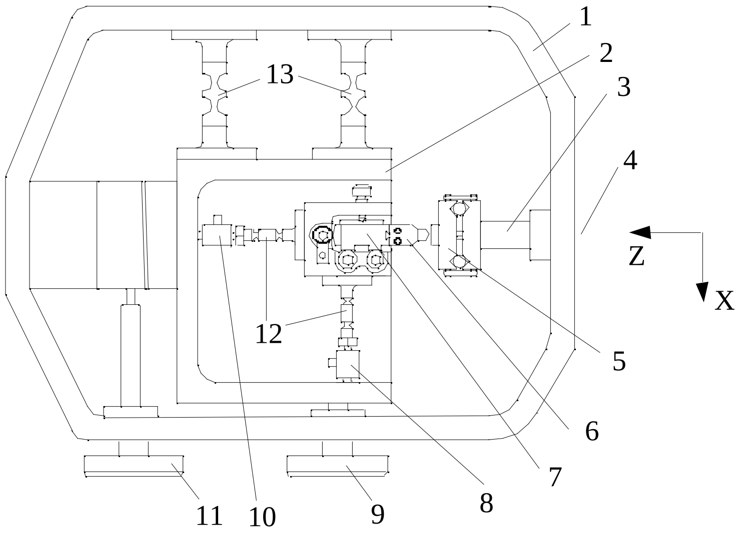
A nanomachining instrument has been designed and built in the Center for Precision Metrology, The University of North Carolina at Charlotte[2]. This instrument, which is basically a single point diamond planer, was developed to perform two-dimensional nanocutting experiments especially on brittle materials. In this paper, we present some results of nanoindentation experiments using this nanomachining instrument.

DESCRIPTION OF THE INSTRUMENT

Figure 1 shows the schematic of the nanomachining instrument. A PZT tube scanner of 12.7 mm in diameter and 25.4 mm in length is employed to accomplish a maximum depth of cut of 4 μm with 0.1 nm resolution, and length of cut of more than 20 μm. The flexural stiffness and the axial stiffness of the PZT scanner are 6 x 10^6 N/m and 7 x 10^7 N/m , respectively. The sample is kinematically mounted on the PZT scanner, and the tool is held stationary during cutting. The infeed motion is monitored by a capacitance probe with a measurement range of 2 μm so that the nonlinearity and hysteresis of the PZT can be measured and compensated. There are two force sensors to measure thrust and cutting forces with a resolution of better than 0.2 mN. The stiffness of the structural loop is designed to be close to that of the PZT scanner. The cutting area is set to be very close to the top surface of the sample so that the machining process can be observed by optical microscopes and scanning electron microscopes.

EXPERIMENTAL SETUP FOR NANOINDENTATION

Nanoindentation experiments can be simply realized by replacing the cutting tool with a diamond



1: Main frame
2: Sub-frame
3: PZT
4: Capacitance probe
5: Sample holder
6: Tool shank and tool
7: Tool holder
8: Cutting force sensor
9: Y-directional fine positioning system
10: Thrust force sensor
11: Z-directional fine positioning system
12: Force decoupling flexures
13: Main flexures

Figure 1 Schematic of the nanomachining instrument

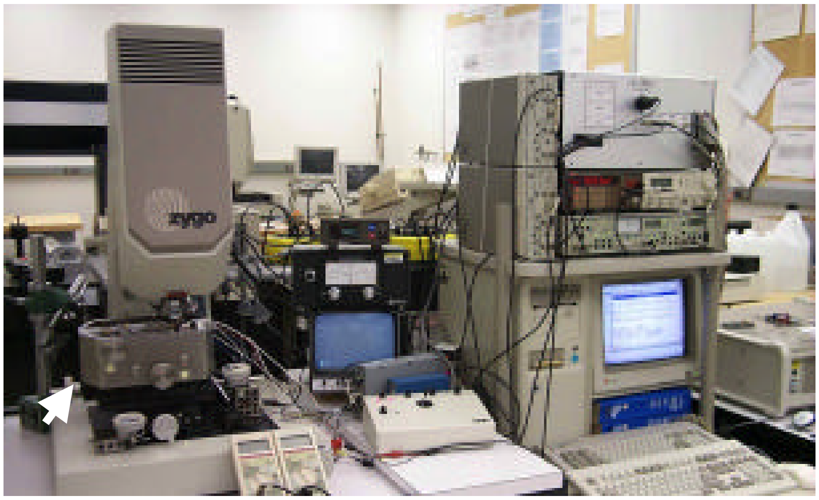
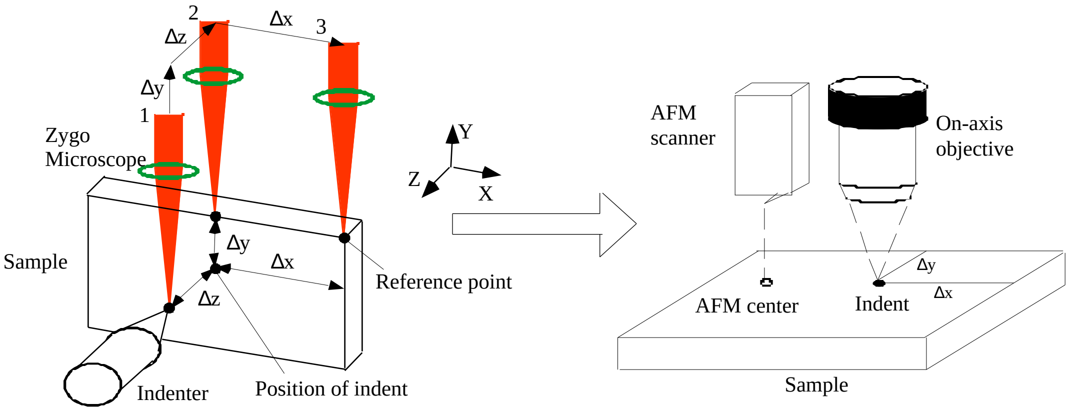


Figure 2 Photograph of the experimental setup for nanoindentation



(a) Measuring the indent position on a Zygo (b) Imaging the indent on an AFM

Figure 3 Locating and imaging the indent

indenter. The load and the indentation depth can be measured by the thrust force sensor and the capacitance probe, respectively.

Figure 2 shows a photograph of the experimental setup for nanoindentaion. In nanoindentation experiments, it is desirable to know the position of the indent so that the indent can be easily imaged by an AFM. For this purpose, we mounted the instrument on a Zygo interference microscope. By using the high magnification optical microscope, we can clearly observe the tool-sample interface. As shown in Figure 3(a), the position of the indent on the sample can be measured precisely by respectively focusing the microscope on the indenter and a reference corner point on the top surface of the sample, and measuring the moving distances of the focal point. Once we obtain the coordinates Δx and Δy of the indent relative to the reference corner point, we can easily find the indent when it is imaged by an AFM. A PSI AutoProbe XL AFM system was used to image the indent. As can be seen in Figure 3(b), the PSI AFM provides an on-axis, high magnification view of the sample, making it easy to select an area of interest on the sample surface. After the on-axis view is focused on the indent based on the coordinates Δx and Δy, the system automatically moves the motor-driven sample stage so that the indent can be located in the AFM center, and can be imaged by the AFM.

MAKING TOOL-SAMPLE CONTACT

In nanoindentation experiments, it is important to establish sample-tool contact to nanometer accuracy. To make the tool-sample contact, we first manually move the tool toward the sample until the gap between the tool and the sample is within several microns by using the fine positioning system of the instrument with the visual feedback from the optical microscope. Then the sample is automatically moved closer to the tool by

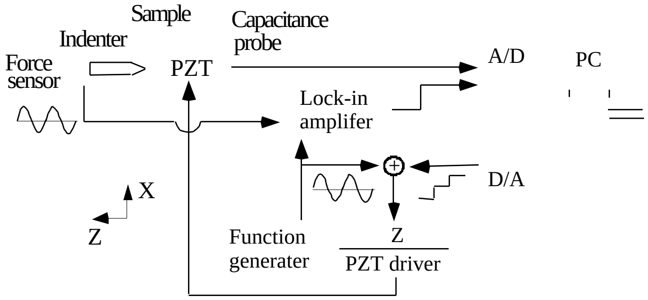


Figure 4 Diagram of making tool-sample contact

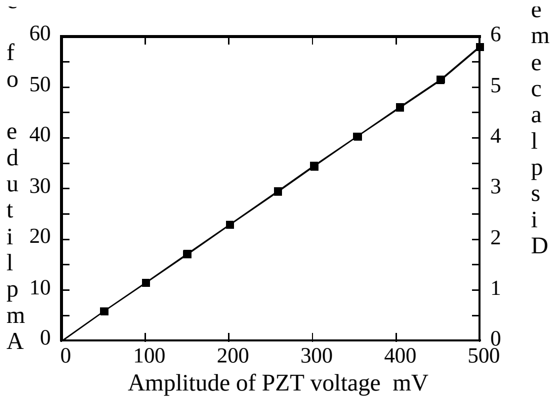


Figure 5 Output characteristics of the PZT

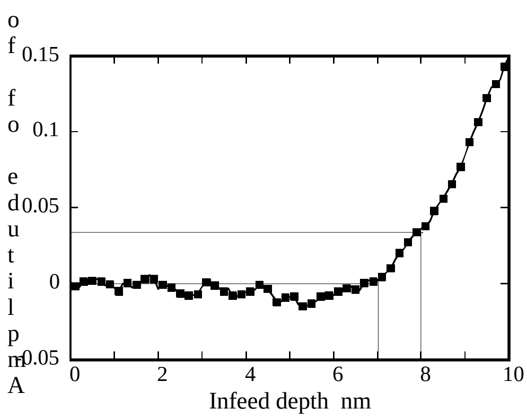


Figure 6 AC force output during making contact

using the PZT and small steps while monitoring the force sensor output. An increase of the force sensor output indicates the tool-sample contact, and the capacitance probe records the position of the PZT at the contact point.

The accuracy of establishing sample-tool contact is mainly determined by the sensitivity of the force sensor. Although the employed force sensor is quite sensitive, it is influenced by the system electronic noises and environmental mechanical vibrations. To improve the sensitivity of the force sensor, we convert the force to an AC signal. As can be seen in Figure 4, the sample is oscillated at a constant frequency by the PZT scanner when it is moved toward the tool. The output of the force sensor will correspond to the oscillation frequency of the sample once the sample comes in contact with the tool. The AC force sensor output is detected by a lock-in amplifier, which has the ability to detect and measure small AC signals down to a few nanovolts in the presence of noise.

In this case, the sample need to be oscillated at a stable amplitude of less than several nanometers. Figure 5 shows the characteristics of the PZT when being oscillated at small amplitude. The amplitude of the PZT displacement was measured by the capacitance probe. It can be seen that the output of PZT is stable in nanometer range.

Figure 6 shows the force sensor output during making tool-sample contact. The sample was oscillated at a frequency of 180Hz. It can be seen that the tool-sample contact can be made in nanometer accuracy.

INDENTATION EXPERIMENTS

Figure 7 shows the AFM image of indents on a silicon (111) surface. A diamond conical indenter with a radius of 5μm was used in the indentation experiments. Final depths are also shown in the figure. Figure 8 shows the load-displacement curve corresponding the number 2 indent in Figure 7. As can be seen in these figures that the developed instrument has the ability to perform depth-sensing nanoindentations.

Nanoindentations were also performed to evaluate the frame compliance (inverse of the frame stiffness) of the instrument. The frame compliance C_f can be expressed as [3]:

$$C_f = C_t - C_s = C_t + \frac{\sqrt{\pi}}{2E_r}\frac{1}{\sqrt{A}}$$

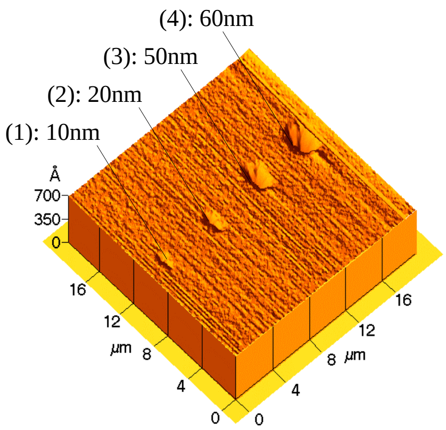


Figure 7 Indents on silicon

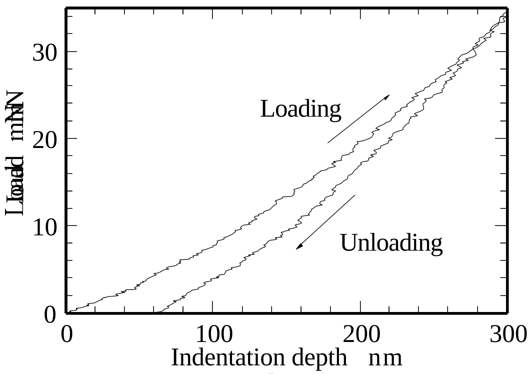


Figure 8 Indentation curve

Table 1 Measured frame compliance

NUM	C_t (nm/mN)	A (nm^2)	C_s (nm/mN)	C_f=C_t-C_s (nm/mN)
1	7.874	2916432	7.454	0.420
2	5.291	6943482	4.831	0.460
3	4.237	11072425	3.826	0.411
4	4.149	12735762	3.567	0.582
5	4.082	11946209	3.683	0.399
Ave.				0.454

where C_t and C_s are the total measured compliance and sample compliance, respectively. E_r is the reduced modulus that is calculated from the elastic moduli and Poisson's ratios of the sample and indenter. A is the projected area of the indentation, which is measured by AFM. Table1 shows the measured results. The frame compliance of the instrument was evaluated as 0.454nm/mN. Fused silica was used as the sample.

CONCLUSIONS

A newly developed nanomachining instruments has been used to perform nanoindentaion experiments. An experimental system has been constructed to make it easy to locate and image the indent. A technique has also been develop to make tool-sample contact with nanometer accuracy. Experimental results have shown that the developed instrument has the ability to perform depth-sensing nanoindentations. The frame stiffness of the instrument has also been evaluated from the indentation results.

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