Subsurface Integrity Research in Ultraprecision Manufacturing

Jiwang Yan

Department of Mechanical Systems and Design, Tohoku University
Aoba 6-6-01, Aramaki, Aoba-ku, Sendai, 980-8579, Japan
yanjw@pm.mech.tohoku.ac.jp

Abstract: Although “surface damage” in ultraprecision machining has been investigated extensively in the past decades, “subsurface damage”, i.e., internal defects of machined materials, is a new research focus. For an externally “perfect” surface, the depth and nature of the subsurface damage influences the mechanical, optical and electronic performance of the surface. The production of subsurface damage-free components is an essential task for both manufacturing industry and multidisciplinary researchers. In this talk, fundamental research results on subsurface integrity from the author’s group will be introduced. Firstly, the formation mechanisms of subsurface damage in ultraprecision machining will be discussed for various kinds of hard brittle materials, and then the characterization and evaluation techniques of subsurface damage will be reviewed. Finally, a newly developed laser damage recovery process, which is effective for improving subsurface integrity, will be introduced, and some future directions in this research area will be briefly mentioned.

Keywords: Ultraprecision machining, subsurface damage, surface integrity

1. Introduction

Ultraprecision machining of hard brittle materials in the micro-nano scale is an area of focused research interests. The manufacturing of defect-free components on these materials is indispensable for production of precision optics, optoelectronic elements, and micro electromechanical systems. It has been well accepted that a nominally hard brittle material can be deformed and removed in a ductile manner in ultraprecision machining processes, such as cutting, grinding and lapping, yielding continuous chips and extremely smooth surfaces [1]. However, these mechanical machining processes will inevitably cause subsurface damage to the crystalline structures due to the contact stress between the tool and the workpiece. It is the depth and nature of the subsurface damage that influences the mechanical, optical and electronic performances of the final products. Therefore, “subsurface integrity” is an essential aspect of ultraprecision machining technology. In this talk, experimental and simulation results on microstructures and mechanical properties of machining-induced subsurface damage will be presented, and nondestructive characterization of the subsurface damaged layers using micro laser Raman spectroscopy will be introduced. Moreover, a nano-pulsed laser irradiation technology for reconstructing the machining-damaged lattice structure will be demonstrated. In this abstract, the results for single crystalline silicon, which is a dominant semiconductor material, will be shown as an example.

2. Subsurface damaging mechanism

A number of previous authors used cross-sectional transmission electron microscopes (TEM) to observe the machined subsurface structures. TEM studies of diamond-turned silicon surfaces revealed that a thin (a few tens to one hundred and a few tens nm-thick) amorphous layer was formed by machining, below which is a region of crystal about a few μm deep. The depth and the microstructure of the subsurface damage layer were found to be strongly dependent on the machining conditions, such as tool take angle and depth of cut, and crystalline orientation of the workpiece [2]. It is interesting that for a highly negative rake angle, the thickness of the amorphous layer is approximately equal to the depth of cut.

An abundance of literature has demonstrated that silicon undergoes phase transformation under hardness indenters and in other situations where high hydrostatic pressure exists. It is generally accepted that a structural transformation from diamond cubic (Si-I) into a metallic state β-Sn (Si-II) that could occur under the indenter during loading due to high hydrostatic pressure (10~13 GPa). Material around the indenter would then become sufficiently ductile to sustain plastic flow. After the indenter is unloaded, the pressure-induced metallic phase does not transform back to the diamond cubic structure, but instead, changes to an amorphous phase or other metastable phases [3].

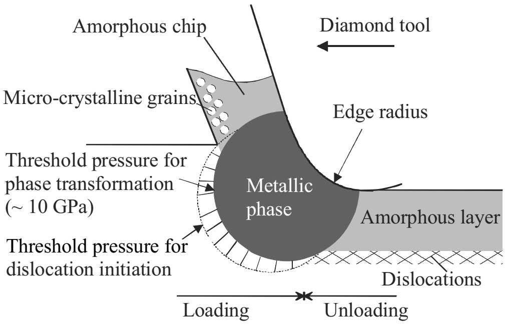

Fig.1 Schematic model of subsurface damage formation

Similarly, in ultraprecision machining, the hydrostatic pressure can be sufficiently high to make silicon undergo phase transformation. From the viewpoint of both phase transformation and dislocation, a model of subsurface damage mechanism in ductile machining of silicon is proposed as schematized in Fig. 1. As soon as the tool advances into the material (loading), a phase transformation from diamond cubic structure (Si-I) to metallic phase (Si-II) occurs in the material surrounding the tool tip. This part of the material becomes sufficiently ductile to sustain plastic flow which facilitates ductile-mode material removal. After the tool has passed (unloading), the metallic phase does not transform back into the diamond cubic structure, but rather, changes into an amorphous phase. As a result, the final subsurface damage layer contains an amorphous phase rather than the metallic phase. The metallic phase does not transform to other metastable phases presumably because the unloading speed in cutting is by far higher than that used in the indentation tests. In Fig. 1, as the distance from the tool tip increases, the pressure decreases and finally drops below the threshold pressure required for phase transformation (~10 GPa). However, the pressure may still be high enough to facilitate dislocation initiation in silicon if a sufficiently high shear stress exists. As a result, a dislocation layer is generated beneath the amorphous layer as the tool passes [2]. The phase transformation of silicon during machining has double-faced effects. On the one hand, due to the transformation from diamond cubic to metallic structure, the material around the tool becomes sufficiently ductile to sustain plastic flow. This facilitates the ductile regime machining. On the other hand, the phase transformation from metallic to amorphous gives rise to the residual amorphous layer on the produced surface, which affects the subsurface integrity.

3. Characterization of subsurface damage

Subsurface damages, especially amorphous layer, will significantly influence the mechanical, optical and electrical functions of silicon parts. For example, when considering micromechanical applications where surface contacts and/or frictions exist, the mechanical properties of the amorphous layer, such as hardness, elasticity and plasticity, become very important. The subsurface damages also influence the subsequent wafer manufacturing processes. That is, a machining operation always involves multiple tool passes due to the cross feed; thus, with the exception of the first cut, all subsequent cuts are made upon an amorphous material and not the starting crystalline material. From this point of view, it is essential to clarify the mechanical properties of this amorphous layer and to quantitatively measure the thickness of this layer.

In a previous work [4, 5], we performed nanoindentation tests on ultraprecision ductile-machined silicon wafers. As the indentation behavior is dominated by the machining-induced amorphous layer for low loads and shallow depths, this situation can be simply considered as a thin film of amorphous silicon formed on a pristine crystalline substrate. Therefore, if a suitable indentation load is used, it may be possible to detect the presence and the mechanical property of this amorphous layer by nanoindentation. We found that when the maximum load is smaller than a critical value, the hardness of the diamond-turned silicon wafer becomes distinctly lower than that of the pristine silicon. In this case, the indentation response will be mainly due to the amorphous layer; thus, it can be concluded from these results that the machining-induced amorphous silicon is softer than diamond-cubic silicon. This surface-softening phenomenon is very different from conventional metal machining, where the near-surface layer always becomes harder due to the work-hardening effects. The work-hardening effect of metal is due to dislocation activities; while the surface-softening effect during silicon machining is caused by phase transformation. It was also found that the machining-induced amorphous silicon has significantly higher micro plasticity than diamond cubic silicon. Therefore, it is the property of the amorphous phase that in effect dominates the machinability of silicon. From this aspect, we can say that when dealing with ductile machining technology the mechanical properties of the amorphous layer should be considered.

To measure the depth of the phase transformation layer resulting from machining, we performed laser micro Raman spectroscopy analysis (Fig.2). Laser Raman can detect the presence of amorphous silicon as well as residual stresses in silicon wafers [6]. For bulk crystalline silicon (c-Si), the triple degenerate optical phonons display in the first-order Raman spectrum a sharp peak at the Raman shift of 521 cm^{-1}, and for amorphous silicon (a-Si), the first-order Raman spectrum reflects the phonon density of states and presents an optical band peak at 470 cm^{-1}. In most of previous works, qualitative detection of structural changes of materials has been carried out using Raman shift information, whereas the intensity information of the Raman scattering has not been effectively used. In our work [7], we use the Raman intensity to characterize the thin amorphous layers on the crystalline bulk material. In order to compare the intensities of Raman scattering from the amorphous region and that from the crystalline region, we propose a new parameter, namely Raman intensity ratio, to represent the relative significance of the two phases. We used the Gaussian distribution for fitting amorphous peaks and the Lorentzian distribution for fitting crystalline peaks, respectively. A strong correlation was identified between the Raman intensity ratio and the depth of amorphous layer, and the correlation was approximately linear within the experimental range. Therefore, by utilizing the linear relationship, it is possible for us to quantitatively predict the depth of amorphous layers using the Raman intensity ratio. This method can be used for quantitative measurement of subsurface damage depth of silicon. This measurement method is very quick, inexpensive and completely nondestructive. It can be done at room temperature, in air and without contacts, and can be used for on-machine measurement and mapping of specific surface areas.

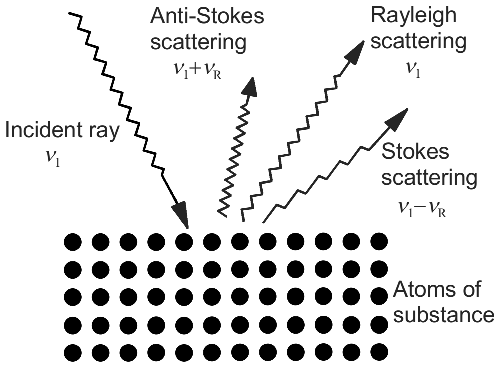

Fig.2 Principle of laser Raman analysis for subsurface damage

4. Subsurface damage recovery by laser irradiation

Removing subsurface damage from silicon substrates is essential for producing high-reliability silicon parts. This issue has become a subject of concentrated research interests from multidisciplinary research communities and industries. Currently, after the machining processes, etching and chemo-mechanical polishing (CMP) processes are carried out to remove the subsurface damaged layers. However, due to the poor controllability of processing depth, the deterioration of substrate form accuracy becomes a big problem. Other issues, such as increase in production cost, and environmental pollution due to the chemical waste fluids, are also serious problems.

In our group, we proposed reconstructing the lattice structure of the damaged layer by laser irradiation [8, 9]. The processing mechanism is schematized in Fig. 3. As amorphous silicon has a remarkably higher absorption coefficient of laser light than crystalline silicon, there will be sufficient absorption of laser in the near-surface layer to form a thin liquid silicon film. The liquid layer is metallic and has a much higher absorption rate, and thus becomes thicker and thicker. The top-down melted liquid phase finally extends below the deeper dislocated region. During the period of melting, an initially rough surface becomes a smooth one due to the surface tension effect of the liquid layer. This is similar to the manner in which a free droplet of liquid naturally assumes a spherical shape to achieve a minimum surface area to volume ratio. For a plane wafer, the surface area reaches a minimum when the liquid thin film becomes completely flat at the surface. After the laser pulse then, environmental cooling will result in a bottom-up epitaxial regrowth from the defect-free crystalline region which serves as a seed for crystal growth. The epitaxial regrowth mechanism in this case is different from that in laser annealing technology, where amorphous silicon is usually sputtered on substrates of different materials, such as glass or sapphire. In such cases, no lattice-matched crystal seed exists, thus poly-crystal structures will be formed. In this work, it is expected that by using the material self-organization phenomena induced by the nanosecond laser pulses, we might be able to achieve both a perfect single-crystal subsurface structure and an extremely smooth surface at the same time.

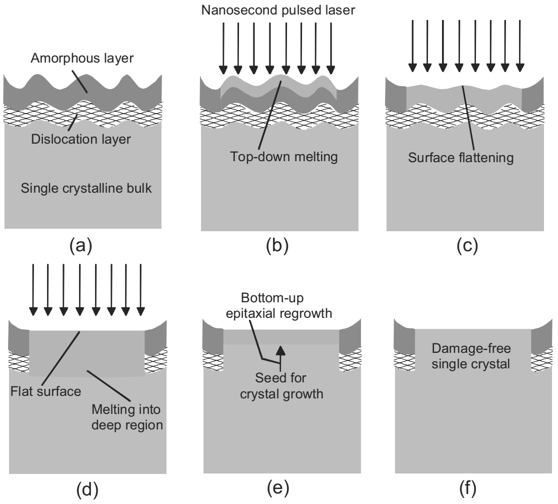

Fig.3 Mechanism of laser recovery of subsurface damage

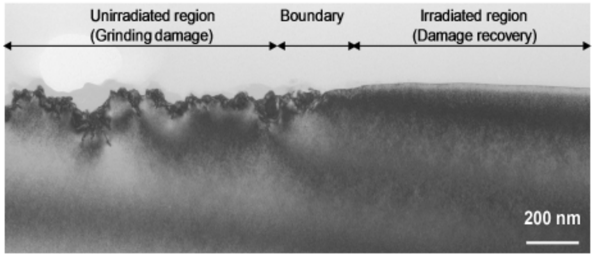

Fig.4 Cross-sectional TEM of silicon before and after later recovery

Fig.4 shows a cross-sectional TEM micrograph of silicon sample before and after laser irradiation. We can see that before laser irradiation, there is an amorphous layer beneath the ground surface, and below the amorphous layer there is a layer of dislocations. After laser irradiation, however, the amorphous layer and the dislocations have completely disappeared, indicating that the recrystallization of silicon was performed successfully. A perfect single crystalline structure identical to that of the bulk material has been achieved. From the results, we can also see that after laser irradiation, the surface roughness has been remarkably improved. That is, the grinding-induced tool marks on the wafer surface have been significantly flattened. The surface flattening phenomenon might be a result of surface tension effect of the melted silicon during the laser pulse. The proposed laser processing technique offers a number of advantages over the conventional chemo-mechanical processes: (i) It involves no material removal thus preserves the dimensions of the workpiece; (ii) It generates no pollutants; (iii) It enables selective processing and processing of complex shapes like aspherical and diffractive optical elements.

5. Summary

Ultraprecision machining of hard brittle materials in the micro-nano scale is technologically and economically important in modern society. Subsurface damage due to high-pressure phase transformation is an essential issue to deal with in ductile machining of semiconductor materials. Micro laser Raman spectroscopy has been demonstrated to be an effective means of nondestructive evaluation for the damaged subsurface layers. Laser is also effective to repair the subsurface damage. Perfect single-crystalline structure can be reconstructed on a machining-damaged silicon substrate through a single nanosecond pulse laser irradiation. The combination of ductile machining and laser irradiation may lead to significant improvement of subsurface integrity in manufacturing industry.

Further reading

[1] J. Yan, M. Yoshino, T. Kuriagawa, T. Shirakashi, K. Syoji and R. Komanduri: On the Ductile Machining of Silicon for Micro Electro-mechanical Systems (MEMS), Opto-electronic and Optical Applications, Materials Science and Engineering A, 297, 1-2 (2001) 230-234.

[2] J. Yan, T. Asami, H. Harada, and T. Kuriyagawa: Fundamental investigation on subsurface damage in single crystalline silicon caused by diamond machining, Precision Engineering, 33, 4 (2009) 378-386.

[3] J. Yan, H. Takahashi, X. Gai, H. Harada, J. Tamaki and T. Kuriyagawa: Load effects on the phase transformation of single-crystal silicon during nanoindentation tests, Materials Science and Engineering A, 423, 1-2 (2006) 19-23.

[4] J. Yan, H. Takahashi, J. Tamaki, X. Gai, H. Harada, and J. Patten: Nanoindentation tests on diamond-machined silicon wafers, Applied Physics Letters, 86, 5 (2005) 181913, pp. 1-3.

[5] J. Yan, H. Takahashi, J. Tamaki, X. Gai, and T. Kuriyagawa: Transmission electron microscopic observation of nanoindentations made on ductile-machined silicon wafers, Applied Physics Letters, 87, 11 (2005) 211901, pp.1-3.

[6] J. Yan: Laser micro-Raman spectroscopy of single-point diamond machined silicon substrates, Journal of Applied Physics, 95, 4 (2004) 2094-2101.

[7] J. Yan, T. Asami and T. Kuriyagawa: Nondestructive measurement of the machining-induced amorphous layers in single-crystal silicon by laser micro-Raman spectroscopy, Precision Engineering, 32, 3 (2008) 186-195.

[8] J. Yan, T. Asami and T. Kuriyagawa: Response of machining-damaged single-crystalline silicon wafers to nanosecond pulsed laser irradiation, Semiconductor Science and Technology, 22, 4 (2007) 392-395.

[9] J. Yan, S. Sakai, H. Isogai, and K. Izunome: Characterization of microparticles and oxide layers generated by laser irradiation of diamond-machined silicon wafers, Semiconductor Science and Technology, 26, 2 (2011) 025006, pp.1-7.