Subsurface Damage of Monocrystalline Germanium Wafers by Fixed and Free Abrasive Lappings

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Abstract: The subsurface damage (SSD) layers of monocrystalline germanium wafers lapped by three different ways were measured and compared by the method of nanoindentation and micro morphology. Three ways such as ice-fixed abrasive, thermosetting fixed abrasive and free abrasive lappings are adopted to lap monocrystalline germanium wafers. The SSD depth was measured by a nanoindenter, and the morphology of SSD layer was observed by an atomic force microscopy(AFM). The results show that the SSD layer of monocrystalline germanium wafer is mainly composed of soft corrosion layer and plastic scratch and crack growth layer. Compared with thermosetting fixed abrasive and free abrasive lappings, the SSD depth lapped with ice-fixed abrasive is shallower. Moreover, the SSD morphology of monocrystalline germanium wafer lapped with ice-fixed abrasive is superior to those of two other processing ways.

Key words: subsurface damage(SSD); nanoindentation; fixed abrasive lapping; monocrystalline germanium wafer

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0 Introduction

Lapping monocrystalline germanium wafers is an important step before polishing, in order to improve the flatness and uniformity of workpiece surfaces. Surface damage (SSD) layers induced by lapping directly affect the image quality, laser-induced damage threshold, stability, service life and other important performance indicators of optical elements[1-3]. Therefore, it is necessary to make a comparative study on the processing methods and the processing results.

SSD can be divided into visible damage and invisible damage according to visibility. Visible damage mainly refers to the surface cracks, scratches and other defects, while invisible damage refers to the crack layer below the surface, the residual stress layer below the crack layer and so on[4-5]. Crack can be divided into dynamic crack and static crack by the formation, and the inertial force and the relative rate affect the propagation of SSD in the machining process[6]. In most of SSD studies, the object is dynamic crack. Static crack is produced by the extrusion and impact between workpiece and a single abrasive grain in the polishing pad or polishing liquid, but static crack is disorganized and difficult to be observed because of the complexity of internal material properties and structures[7].

Research methods for measuring SSD can be divided into destructive and non-destructive. Due to higher requirements of sample preparation, more advanced testing equipments and higher cost, generally, non-destructive testing technologies (e. g. acoustic microscopy and laser microscopy) are needed when the requirement of subsurface damage is strict[8-9]. Destructive testing technology is a common means of detection which

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is a direct, effective and easy operation, but its measurement results are not accurate enough and it will cause secondary damage to workpiece and make waste of material. The common destructive testing methods include chemical etching method, angle polishing method, magnetorheological finishing method and so on[10-12]. In this paper, the nanoindentation method is proposed to detect the SSD of germanium wafer, and the depth and composition of SSD will be analyzed.

1 Experiment

1.1 Experimental material and instruments

The SiC abrasive has the average size of 1.5 μm with a purity level of more than 99.9%, and monocrystalline germanium wafers used have a diameter of 50.8 mm and a thickness of 0.5 mm with a purity level of more than 99.9%.

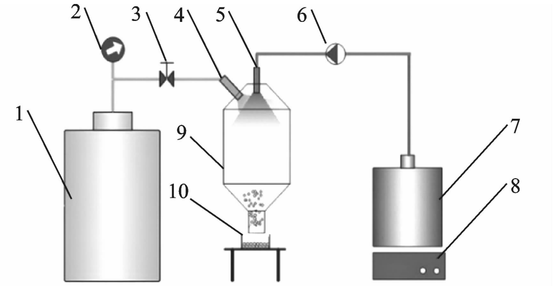
As shown in Fig. 1, the ice lapping experiment is performed on an ice lapping apparatus. The pressure is controlled by an air cylinder and the temperature in the polishing zone is controlled below 0 ℃ with a cold air generator. The SSD depths are measured by nanoindenting using a G200 nanoindenter. The morphologies and roughness of the lapped surface are observed and examined on an atomic force microscopy (AFM).

Fig. 1 Photograph of ice polishing apparatus

1.2 Preparations of lapping pad and lapping liquid

1.2.1 Preparation of ice-fixed abrasive lapping pad

As shown in Fig. 2, the preparing system is mainly composed of polishing liquid supply system, liquid nitrogen supply system, heat exchanger system and ice grain reserve system. When producing ice particles, the liquid nitrogen is fed into the heat exchanger system and the polishing liquid supplying system is opened at the same time. Waiting for 10—15 min, the ice particles will be collected in the outlet of the heat exchanger. Placing ice particles in a mold evenly and exerting a pressure of 10 kg, we can obtain an ice-fixed abrasive lapping pad. The photograph of ice-fixed abrasive polishing pad is shown in Fig. 3.



1—Liquid nitrogen tank; 2—Pressure gauge; 3—Safety valve; 4—Atomizing nozzle of liquid nitrogen; 5—Atomizing nozzle of lapping liquid; 6—High pressure pump; 7—Storage tank; 8—Magnetic agitator; 9—Heat exchanger; 10—Forming mold

Fig. 2 Schematic diagram of ice grain rapid preparation system

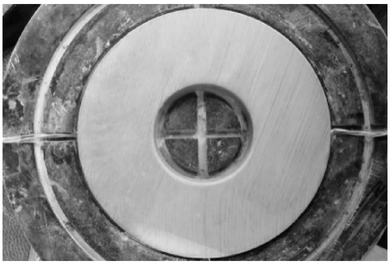


Fig. 3 Ice-fixed abrasive lapping pad

1.2.2 Preparation of thermosetting fixed abrasive lapping pad

The SiC abrasive of the average size of 1.5 μm and resin are mixed at the ratio of 1∶3. After adding the right amount of magnesium sulfate, curing agent CHP, and coupling agent, the mass fraction of the SiC powder should be 15%. The mixture is mixed and applied to the heat curing template. The polished PC board is pressed against the mold, which will be heated and pressed in the SN-50T vulcanizing machine. After 1 h, the pad can be taken out, and the photograph is shown in Fig. 4.

Fig. 4 Thermosetting fixed abrasive lapping pad

1. 2. 3 Preparation of free abrasive lapping liquid

Compared with the preparation of the lapping pads, the preparation of free abrasive liquid is simpler. After adding a small amount of emulsifier OP-10, triethanolamine and catalyst, the SiC mixture with 1. 5 μm, whose concentration is 15%, is mixed in a ball mill for 30 min. In the process of lapping, in order to ensure that the SiC lapping fluid is mixed evenly, the lapping fluid should always be in a state of mechanical agitation.

1. 3 Experimental procedure

Monocrystalline germanium wafers are pretreated with free abrasive to achieve uniform surface roughness, and the average roughness of the three wafers are 115. 0, 115. 1 and 115. 2 nm. Finally, the wafers are lapped in the hypothermia ultra-precision lapping machine with ice-fixed abrasive, thermosetting fixed abrasive and free abrasive, respectively. The detailed lapping conditions are given in Table 1.

Table 1 Parameters under lapping conditions

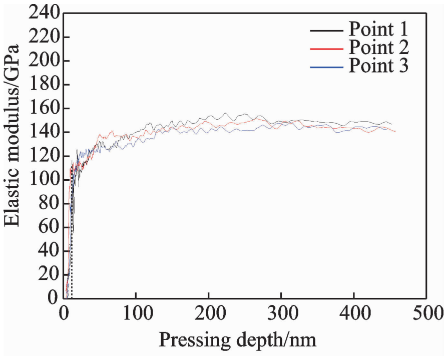
Parameter	Abrasive size/μm	Concentration/%	Temperature/°C
Value	1. 5	15	−3(ice-fixed), ambient temperature(others)
Parameter	Pressure/kg	Time/min	Velocity/(r • min^{-1})
Value	2	50	80

2 Results and Discussion

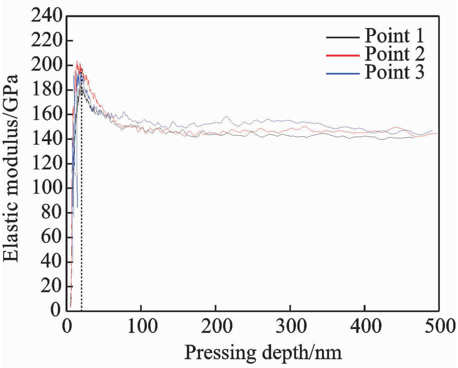
2. 1 Analysis of nanoindentation method

After the above three samples are cleaned for 5 min in an ultrasonic cleaning machine and then dried, nanoindentation test are conducted. This test adopts a G200 nanoindenter, of which the blunt tip radius is 100 nm and indentation depth is 500 nm. Three points of each sample are taken, and the relationships between the elastic modulus and indentation depth are shown in Fig. 5 and the analytical model is shown in Fig. 6 (The standard modulus value of germanium is (140±10) GPa).

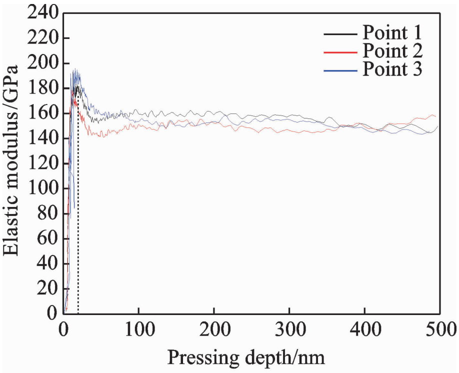
The following conclusions can be drawn from Fig. 5:(1) The elastic modulus values of monocrystalline germanium wafers are finally stabilized and the values range from 140 GPa to 150 GPa, which indicates that the probe reaches the base through the subsurface damage layer. (2) The



(a) Free abrasive lapping



(b) Thermosetting fixed abrasive lapping



(c) Ice-fixed abrasive lapping

Fig. 5 Relationship between elastic modulus and indentation depth

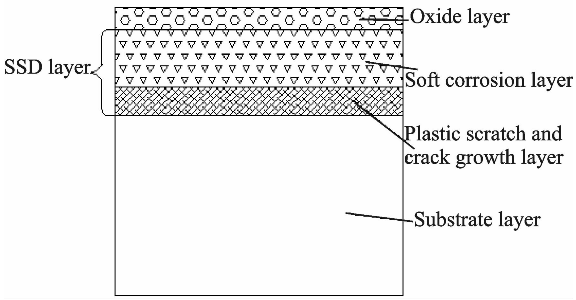


Fig. 6 Analytical model diagram

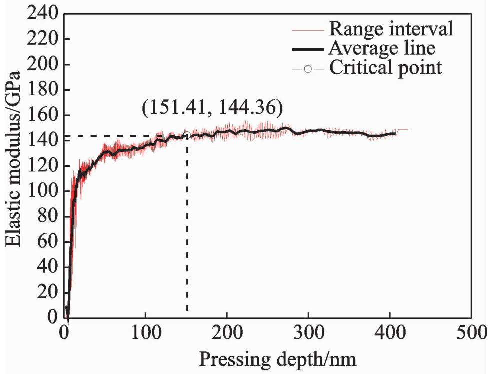
trends of three curves in each sample are consistent and the data is concentrated, so the chance of data can be ruled out. In order to facilitate the analysis, the analytical models are established, as shown in Fig. 6, which divide the germanium wafers into four layers, respectively, such as oxide layer, soft corrosion layer, plastic scratch and crack growth layer, and substrate layer. The soft corrosion layer and the plastic scratch and crack growth layer are referred as SSD layer.

As seen in Fig. 5, at the start of the press, the values of elastic modulus of germanium wafers lapped by three ways increase rapidly, which is due to the existence of an oxide layer on the surface of the three samples. Fig. 5(a) shows that the elastic modulus value of the oxide layer of germanium wafer is smaller than that of the substrate layer after the free abrasive lapping, but from the Figs. 5(b, c), the elastic modulus values of the oxide layer are larger than that of the substrate layers after the ice-fixed abrasive and thermosetting fixed abrasive lapping. This is because when lapping with the free abrasive, a small amount of emulsifier OP-10, triethanolamine and catalyst are added into the lapping fluid, which makes the chemical action of free abrasive strong and makes the mechanical action weak. After lapping, the chemical action of residues inhibits the oxidation of the germanium wafers, so the oxide layer is relatively thin (about 12 nm), resulting in a increase in the elastic modulus value. In fixed abrasive lapping, the main way of removing is the mechanical cutting action of abrasive and the slurry is mainly for cooling and lubrication, whose chemical effect is weak, so the oxide layers are also relatively thick (about 20 nm). In the process of pressing the probe into the oxide layer slowly, the values of elastic modulus increase rapidly and finally exceed the values of the substrate layer.

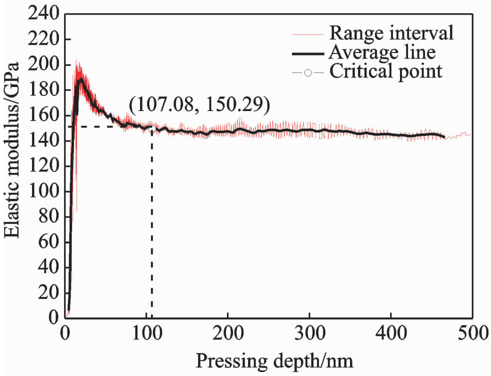
Fig. 5 shows that the elastic modulus value of germanium wafers lapped with free abrasive increases slowly and continuously, but there is a falling back of the value of wafers lapped with ice-fixed abrasive and thermosetting fixed abrasive after reaching the pole, which is because the probe reaches the SSD layer. In free abrasive lapping, the strong chemical action induces a thick soft corrosion layer and a thin plastic scratch and crack growth layer, but the total SSD layer is thicker than those of the two other lapping ways. Therefore, in the process of pressing the probe slowly through the subsurface, the elastic modulus value rises slowly and finally tends to be stable. On the contrary, ice-fixed abrasive lapping and thermosetting fixed abrasive lapping induce a thinner soft corrosion layer and a thicker plastic scratch and crack growth layer, but the total SSD layer was thin, which makes the elastic modulus value come down after reaching the pole and tend to be stable.

As shown in Figs. 5(b) and (c), the falling speed of the elastic modulus values of the germanium wafer lapped with thermosetting fixed abrasive is slower than that of the wafer lapped with ice-fixed abrasive. This is because abrasives protrude above a fixed abrasive polishing pad, and during the process of loading, the exposed abrasive will be pressed directly into the surface of the workpiece. In addition, abrasives are heat cured at lapping pad, so the bonding strength is large and the abrasive cutting action on the workpiece is strong[13]. However, the abrasives in the ice-fixed abrasive lapping pad are completely dispersed in the ice particles and highly consistent with the pad through compaction. Moreover, the ice grains are easy to melt during the lapping process, in which rolling friction will be produced between the shedding abrasives and the surface of the workpiece[14]. Therefore, the mechanical action of thermosetting fixed abrasive lapping pad is stronger than that of ice-fixed abrasive lapping pad. The plastic scratch and crack growth layer of the germanium wafer lapped with thermosetting fixed abrasive is thicker than those of the two other lapping ways, so the transition process is slow before the probe is pressed into the substrate layer.

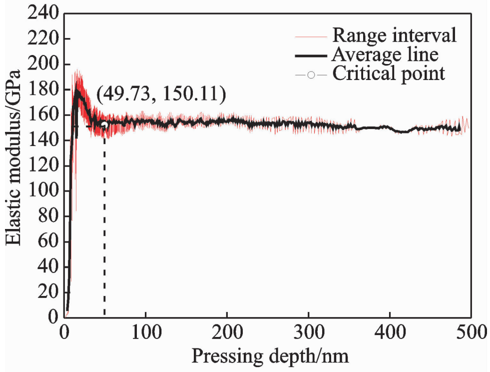
Fig. 7 shows the range intervals, average lines and critical points of the elastic modulus in the three kinds of lapping. As shown in Fig. 7(a), the probe enters into the substrate layer of the wafer lapped with free abrasive at 151.41 nm, so the SSD depth is 139.41 nm (The thickness of the oxide layer is minus). Figs. 7(b) and (c) show that the SSD depths are 87.08 nm and 29.73 nm lapped with thermosetting fixed abrasive and ice-fixed abrasive, respectively. Therefore, it can be considered that the SSD depth of the germanium wafer lapped with ice-fixed abrasive is less than those of thermosetting fixed abrasive lapping and free abrasive lapping.



(a) Free abrasive lapping



(b) Thermosetting fixed abrasive lapping



(c) Ice-fixed abrasive lapping

Fig. 7 Range interval, average line and critical point of the elastic modulus in the three types of lapping

2.2 Analysis of profile and data

After lapped by the three methods, three germanium wafers are etched for 5 min with hydrofluoric (HF) acid (40%v.) to remove the oxide layer and expose SSD layer. The morphologies of SSD layers are observed by AFM after ultrasonic cleaning and drying. Selecting three areas on each sample as the scanning areas, as shown in Fig. 8, they are the central region 1 and two symmetric regions 2, 3 at 5 mm from the edge, of which the area size is 20 μm × 20 μm.

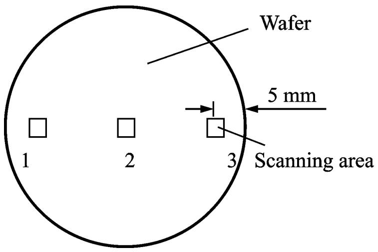
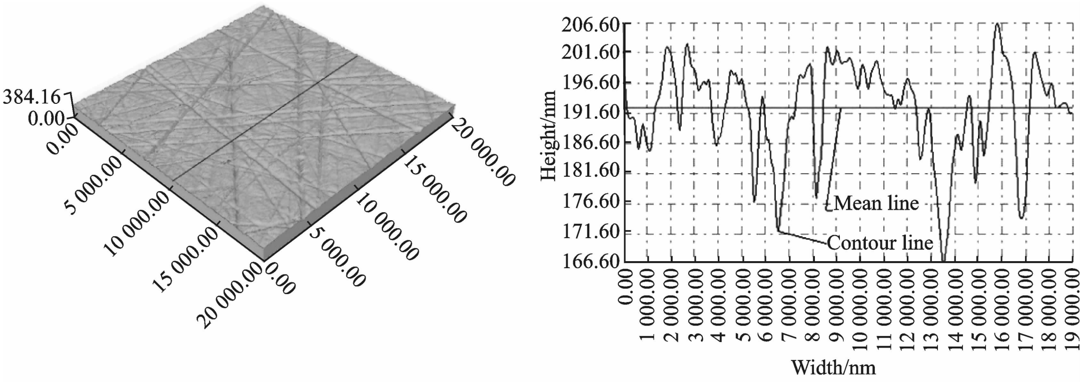


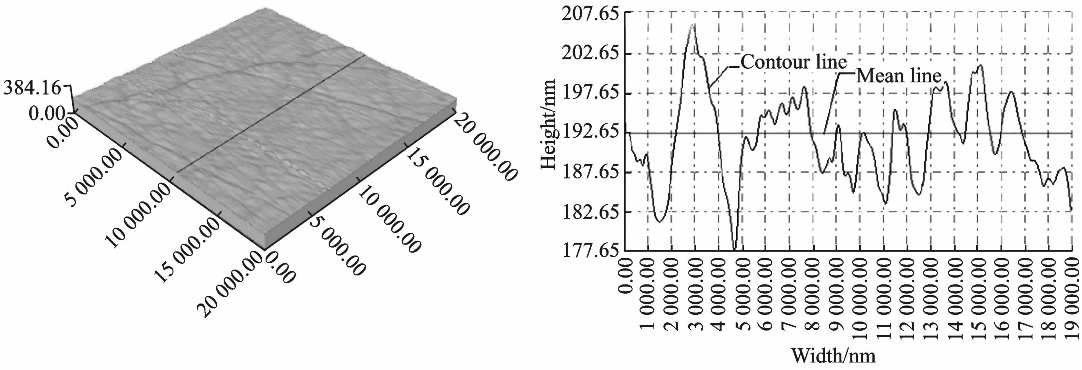
Fig. 8 Distribution of scanning areas

Fig. 9 shows the morphologies (left) and profiles (right) of the SSDs of the samples, and to ensure consistency, the center line is selected as the measurement region, of which the length is 19 000 nm.

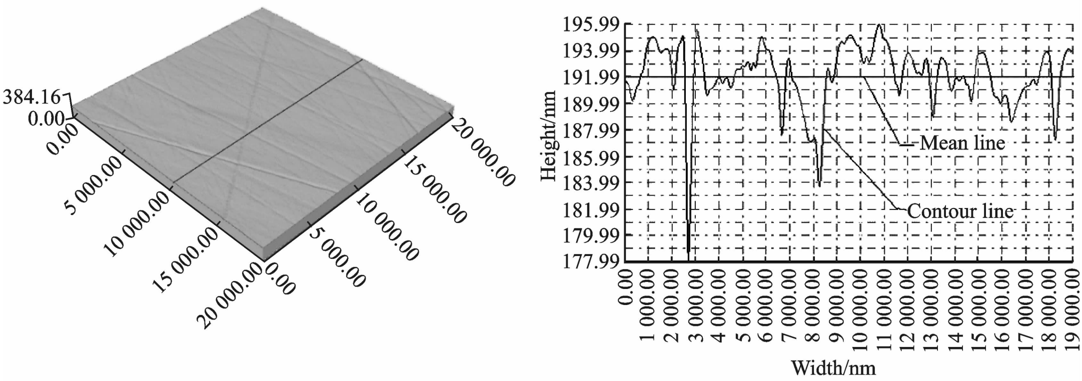
The following conclusions can be obtained from Fig. 9. (1) The amount of scratches and cracks on the subsurface of ice-fixed abrasive lapping is significantly less than those of thermosetting fixed abrasive lapping and free abrasive lapping. (2) From the view of overall volatility, lapped with free abrasive, the profile of the SSD layer is very large and very uneven. However, the overall fluctuation of the SSD layer is relatively stable and the majority of the surface profile is uniform after thermosetting fixed abrasive lap-



(a) Free abrasive lapping



(b) Thermosetting fixed abrasive lapping



(c) Ice-fixed abrasive

Fig. 9　AFM images of SSD layers (left) and profile (right)

ping, and the fluctuation of the SSD layer is very small and the whole is uniform after ice-fixed abrasive lapping. (3) Selecting the arithmetic average midline as reference, we can see that the maximum peak value of free abrasive lapping is 206.4 nm, 13.8 nm to the midline, and the minimum valley value is 166.60 nm, 26 nm to the midline. Meanwhile, the maximum peak value of thermosetting fixed abrasive lapping is 206.33 nm, 13.68 nm to the arithmetic average midline, and the minimum valley value is 177.65 nm, 15 nm to the midline. The maximum peak value of ice-fixed abrasive lapping is 195.99 nm, 5 nm to the arithmetic average midline, and the minimum valley value is 177.99 nm, 14 nm to the midline. From the above analysis, the morphology of the SSD layer of ice-fixed abrasive lapping is superior to those of thermosetting fixed abrasive lapping and free abrasive lapping.

From the above two aspects, comparing results of SSD layers of germanium wafers after lapping, we can know that fixed abrasive lapping is superior to free abrasive lapping. This is because free abrasive lapping is based on the threebody wear, which is among workpiece, abrasive and lapping pad. The relative motion of the above three contains respective movement, so the movement is complex and irregular and the cutting direction of abrasive is chaotic. Therefore, in the process of lapping, it is easy to cause that lapping is excessive or not enough. In addition, free abrasives exist in lapping liquid and have strong randomness, and the phenomenon of extrusion or bonding of abrasives is easy to occur, which seriously affects the quality of subsurface.

However, fixed abrasives are bonded to fixed abrasive pads, which makes abrasives not have the characteristics of randomness and movement. In others words, fixed abrasive lapping is based on two-body wear and the movement is relatively simple and easy to be controlled, so the lapping is relatively uniform. In addition, fixed abrasive lapping pads have a property of self-sharpening, wherein the self-sharpening of the thermosetting fixed abrasive lapping pad is performed by swelling mechanism of polymer and the self-sharpening of the ice-fixed abrasive lapping pad is performed by the melting in the process[15]. Therefore, the lapping effect of fixed abrasive lapping pad is better than that of free abrasive.

In terms of fixed abrasive lapping, the effect of ice-fixed abrasive lapping is better than that of thermosetting fixed abrasive lapping. Although thermosetting fixed abrasive lapping pads have self-sharpening, the experiments show that, compared with ice-fixed abrasive lapping pads, the self-sharpening of thermosetting fixed abrasive lapping pads is a bit poor and it is necessary to make a certain repair in lapping processes, which will affect the quality of machined surface. In ice-fixed abrasive lapping, as long as environmental temperature is reasonable controlled, the lapping heat generated between the motion of the workpiece and the pad will melt the surface of the lapping pad, which will take away old abrasives and expose new abrasives to achieve a more stable self-sharpening process. Therefore, the effect of ice-fixed abrasive lapping is superior to that of thermosetting fixed abrasive lapping.

3 Conclusions

A new and accurate approach is proposed to measure the SSD depth. Meanwhile, a new method is proposed to lap monocrystalline germanium wafers, which induces a lower amount of SSD.

The following conclusions can be drawn:

(1) The SSD layer of monocrystalline germanium wafer is mainly composed of soft corrosion layer and plastic scratch and crack growth layer.

(2) According to the nanoindentation method, lapped with ice-fixed abrasive pad, the SSD depth is 29.73 nm, while the SSD depths are 87.08 nm and 139.41 nm lapped with thermosetting fixed abrasive and free abrasive, respectively.

(3) Lapping method has a significant influence on the SSD of monocrystalline germanium wafer. Under the same lapping conditions, the ice-fixed abrasive lapping can effectively reduce SSD and improve the surface quality of the workpiece.

Acknowledgments

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