A Study on the Fabrication of Micro Groove on Si Wafer using Chemical Mechanical Machining

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Materials are either removed from or added to a device, usually in a selective manner with using thin and/or thick film manufacturing processes that transfer the lithographic patterns into integrated circuits (ICs) or three-dimensional micromachines. This study deals with material removal by chemically assisted mechanical micromachining. Two methods are used chemical mechanical machining method are introduced in this paper. One, mechanically assisted chemical etching, is applied to fabricate a micro beam such as cantilever, and another is chemically assisted mechanical micromachining to fabricate microstructure such as micropattern, microchannel. The results are discussed.

Key Words : Si Wafer, Microstructure, Chemically Assisted Mechanical Micromaching, Mechanically Assisted Chemical Etching Method

1. Introduction

Recent technical advances in integrated circuits, infrared optics, etc., have given single point diamond turning (SPDT) of semiconductor materials, much attention as an important technology to achieve precision manufacturing requirements such as high form accuracy, better surface finish, and lower cost. Examples of SDPT of semiconductor materials extend from infrared mirror surface finishing to thinning of silicon on insulator (SOI) wafers. Precision machining of brittle materials, particularly, machining of single crystal semiconductor materials such as silicon and germanium has been gaining technological attention due to its importance in the application to advanced to optics such as aspherical lens, binary optics, etc., and in integrated circuit technology. In general, machining of brittle materials contains immensely different material removal mechanisms compared to the material removal mechanisms of ductile materials such as the aluminum, and copper. The material removal mechanism in the ductile machining process is mostly from shearing, whereas the material removal mechanism of brittle material consists of many brittle failure phenomena such as crack, brittle fracture, cleavage, etc. Although these brittle material removal mechanisms generate high material removal rate per unit of energy consumed, in many precision and ultraprecision applications, brittle material mechanisms are not desired since it is very difficult to control the dimension, the amount of material removal and the damage induced to surface and substrate. To avoid brittle failure damage in the surface, ductile regime machining is commonly practiced in machining brittle materials that requires ultraprecision material removal or finishing operations. Ductile regime

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machining occurs when brittle material is machined at very shallow depths of cut, typically within a few microns or less. Using the ductile mode or ductile regime machining technique, brittle materials and advanced ceramics such as silicon, can be machined with the material removal process similar to plastic, ductile removal process with continuous chip formation and free from brittle damages. It has been found that for ductile regime machining of brittle materials including advanced ceramics, the ductile regime machining condition is influenced by the parameters such as crystallographic orientation, tool nose radius, rake angle, machining rate and depth of cut. The depth of cut at which the transition of cutting mode changes from ductile to brittle is known as the critical depth of cut, h_c, Fig. 1.

Although naturally brittle and macroscopic material behaviors are similar, brittle amorphous materials such as glass are different from advanced ceramics such as single crystal silicon when it comes to explaining the ductile to brittle transition at critical depth of cut. Although the exact physics of ductile regime machining is not clearly understood to date, the traditional theory of the ductile/brittle transition mechanism is based on the energy balance between the two different material removal mechanisms. It seems that there are pre-existing micro cracks within the material. Based on this theory, ductile machining is possible at a small depth of cut, when the strain energy stored in a specific volume is less than the energy required for propagating the pre-existing cracks. However, in single crystal semiconductor materials, micro cracks are almost nonexistent. Therefore, the traditional energy balance theory is not applicable to single crystal semiconductor materials.

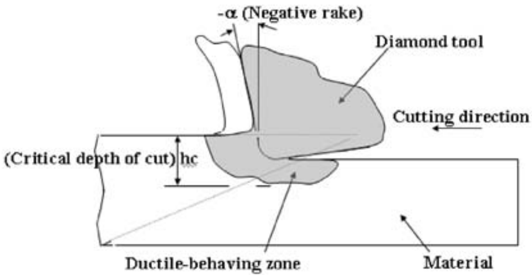

Fig. 1 Schematics of ductile regime machining and critical depth of cut, h_c

Very limited numbers of published articles are available on theoretically establishing a model for the ductile regime machining of defect tree brittle materials. Bifano et al. established a relationship between the critical depth of cut (h_c) as a function of material properties using a fracture mechanics approach as given in equation, as in (1).

$$h_c = 0.15\left(\frac{E}{H}\right)\left(\frac{K_c}{H}\right)^2 \tag{1}$$

where E the Young's modulus, H the hardness of the material and K_c the fracture toughness. Most other theoretical research is based on the molecular dynamics (MD) approach. Using MD, Shimada et al. suggested that the surface layer changes to an amorphous structure in defect free brittle material machining (Shimada et al., 1996). Shibata et al. argued that at a sub-nanometric cutting depth, silicon machining can be examined using the MD approach established for copper, since both materials have the same slip system of {111}, ⟨110⟩. MD simulation research on copper was first proposed by Ikawa et al, followed by Shimada et al. and by Rentsch et al. (1996). Recently, an empirical study on the influence of machining parameters in SPDT of silicon was reported by Syn et al. of the Lawrence Livermore National Laboratory (LLNL). Syn et al. conducted a series of silicon diamond turning tests and surveyed the influence of machining parameters on tool wear and subsurface damage in long distance cutting. Other previous research has shown that the parameters that affect the ductile/brittle behavior include loop stiffness of the machine, tool geometry, cutting speed, interaction between the tool and the crystallographic orientation of the material, hydrostatic pressure, etc.

2. Experimental

2.1 Mechanically assisted chemical etching

The motivation of this work is to extend the capabilities of MCMP to fabricate surface micro structures and micro poly silicon beam such as cantilever. Similar to MCMP, mechanically assisted chemical etching process was suggested in this work. The process is based on the change in the surface energy of the silicon surface at locations where there has been a physical interaction with a sharp tool. Essentially, the frictional energy dissipated along the region of the tool path provides the difference in the surface energy with its surroundings. Then, the entire surface is exposed to a chemical etchant which preferentially react with the location where there has been a physical interaction with the tool. The reaction causes the formation of fine patterns on the silicon surface. The integrity of the fabricated line patterns, however, was not adequate enough for the process to be used for ultraprecision applications. In order to overcome this problem, another process which utilized direct mechanical machining of the resist on the silicon wafer was developed (Lee et al., 2001). The process relies on removing a thin layer of resist on the silicon surface where line patterns are desired. The method of resist removal was based on single asperity machining. Once the resist has been removed, the Si substrate was etched to form grooves on the silicon surface. To achieve good surface integrity by using the technique, it is important to understand the effects of the critical load for the resist removal, feed rate, and the surface damage characteristics. Also, the physical and chemical states of the workpiece surface such as surface roughness must be assessed. This experiment focuses on identifying optimum mechanical machining conditions to achieve effective cutting of the resist layer.

The three-dimensional experimental setup was designed for this work, as shown in Fig. 2. The resolution of the X, Y, and Z axis was 0.1 μm, and the Z axis with an attached PZT moving part had a resolution of 0.01 μm. The mechanical tool to remove material was located on the attached PZT moving part. The workpiece was fixed by a vacuum, and the dynamometer was settled below the workpiece is holder part. Dynamometers inform researchers about the contact of the mechanical tool and the working force. The cutting speed was able to be controlled from 0.01 to 5 mm/sec, and the depth of cut was controlled from 0.1 μm to 100 μm by the PZT. The specifications of the experimental setup are shown in Table 1.

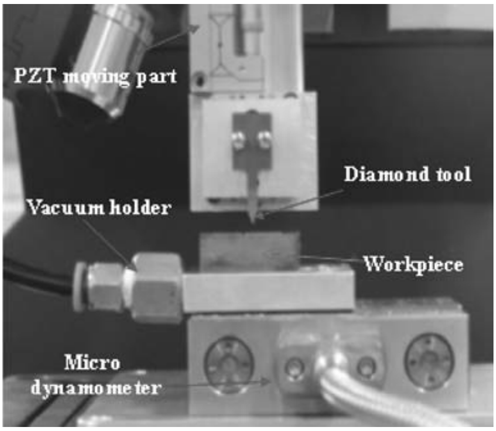

Fig. 2 Experimental setup

Table 1 Specification of experimental setup

X, Y, Z resolution	0.1 μm
PZT (minimum incremental distance)	0.01 μm
Vacuum holder	50mm Hg
Calibrated range	<25N

2.2 Chemically assisted mechanical micromachining

The main purpose of this experiment was to verify that the chemically reacted layer of the silicon material affects mechanical micromachining. The formation of the reacted layer on the surface of material is thought to reduce brittle fractures during micromachining of silicon. To investigate this, chemicals with different concentration were prepared and after a leaching procedure, as the difficult chemical concentration with other variables were fixed, the mechanical properties of the reacted surface were measured in terms of thickness and hardness change; and tested through a method of scratching, indentation, and friction coefficient. To generate a chemically reacted layer, the workpiece is immersed in a bath. While the workpiece was dipped in the bath, the

Table 2 Experimental conditions for generation of chemically reacted layer

Parameters	Conditions
Chemical solution	KOH
Concentration (wt%)	0, 5, 10, 20
Buffer chemical	Acetic acid
Dipping time (min)	5, 10
Temperature	Room temperature

chemical solution was agitated by a stirrer. To prepare the specimens, Si (100) wafers were cleaned in the ultrasonic cleaning bath, followed by dipping in potassium hydrate (KOH) solution to form a reacted layer. The concentrations of KOH were adjusted as 0wt%, 5wt%, 10wt% and 20wt%, as written in Table 2. The pH of the solution was fixed at 12 to exclude the effect of pH by controlling the buffering chemical, acetic acid. In general, the resulting byproduct of the reaction between KOH and Si is hydrated silicon, $Si(OH)_4$, which shows ductile characteristic, as written in equation (2).

$$\equiv \text{Si-OH} \Leftrightarrow \text{Si-O}^{-} + H^{+} \qquad (2)$$

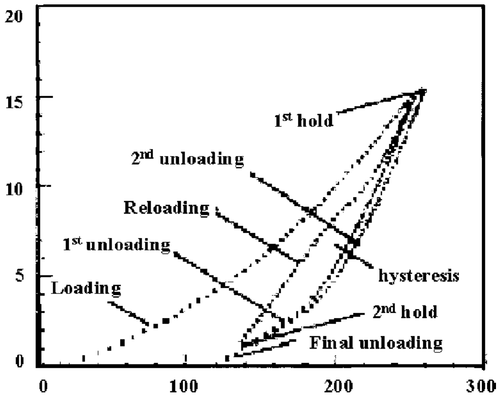

Fig. 3 A representative load-displacement curve

3. Discussion

3.1 Mechanically assisted chemical etching

Single crystal silicon and silicon based materials (e.g., SiO_2 and poly silicon films) are the most common materials used in MEMS/NEMS. Single crystal silicon and silicon based materials are brittle and potential candidate materials, especially poly silicon, for MEMS/NEMS applications because of their isotropic mechanical properties and high corrosion resistance. For the mechanical properties of single crystal silicon, poly silicon, and SiO_2 film, hardness and elastic modulus were measured. For hardness and elastic modulus measurements, multiple loading and unloading steps are performed to examine the reversibility of deformation, ensuring that the unloading data used for analysis purposes are mostly elastic [11]. A typical indentation experiment consists of eight steps: approaching the surface; loading to peak load; unloading 90% of peak load; reloading to peak load; holding the indenter at peak load; unloading 90% of peak load; holding the indenter after 90% unloading; finally unloading completely. The first hold unloading characteristics. The second hold step was included to remove the influence of creep on the incorporate the corrections due to thermal drift. Figure 3 shows a representative load-displacement curve of an indentation made at 15 mN peak indentation load and the hardness and elastic modulus as a function of indentation depth at various peak loads for an undoped Si (100). The undoped Si (100) exhibited hysteresis in displacement during cyclic loading and unloading. Hysteresis observed in the unloading curve at low loads was due to a pressure-induced phase transformation from its normal diamond cubic form to a β-tin phase. This phase transformation resulted in a decrease (about 22%) in volume, which affects the indentation displacement during loading. The hardness and elastic modulus data of the undoped Si (100) and the poly silicon film were comparable. The undoped Si (100) and the poly Si film exhibited hardness of about 12 GPa. Hardness and elastic modulus were estimated from indentation test for all samples. SiO_2 film was used as the resistance for the Si (100) workpiece since it has the proper characteristics for microfabrication, such as high friction and surface damage behaviors as mentioned previously. This process sequence for the creation of a simple trench structure is illustrated in Fig. 4. A resistant layer of (SiO_2) was deposited on a silicon substrate (Fig. 4(A)). The resistant layer was repeatedly removed at the same position by the mechanical tool with the machining load of 5 mN, below the critical load of SiO_2 film (Fig. 4(B)). A selective wet etching process was followed (Fig. 4(C)). The conditions for the wet etching process and mechanical machining are shown in Table 3. Finally, The Si trench structure (Fig. 4 (D)) was fabricated after removing the resistant layer (SiO_2). This technique is applicable to combinations of thin films and lateral dimensions where the substrate can be etched without significant etching of the microstructure. The fabricated Si trench structure using a mechanical assisted chemical etching process is presented in Fig. 5.

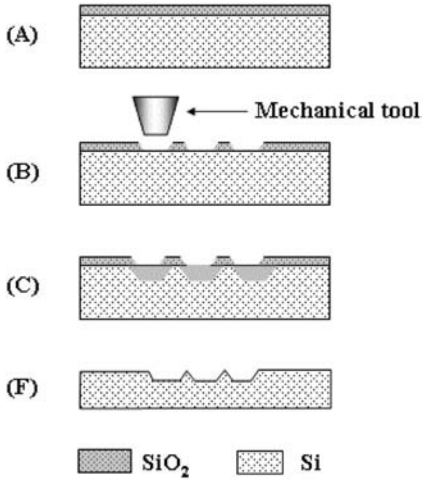

Fig. 4 Basic mechanically assisted chemical etching process (A) resist layer deposition, (B) Mechanical removal, (C) Chemical wet etching, (D) Resist layer removal and Si trench structure)

Table 3 Conditions for fabrication of Si trench structure

Mechanical	Tool	Radius Material	50 μm PCD
	Parameters	Cutting speed Cut load	100 μm/sec 5mN
Chemical	Solution	KOH Ambient temp.	50wt% 20℃
	Etching	time	30 min

Poly silicon beam was fabricated by the mechanical assisted chemical etching process. For the fabrication of a poly silicon beam, the appropriate fabrication step is a pattern transfer to the thin isolation film as shown Fig. 6, illustrating a wet pattern transfer to a 1 μm thick thermal SiO_2 film with a 1 μm resistant layer.

For opening the window to etch SiO_2, the mechanical tool removed the poly silicon film within the critical load. In addition, the working load was 10 mN, below the critical load of a poly silicon film. The conditions for fabricating of

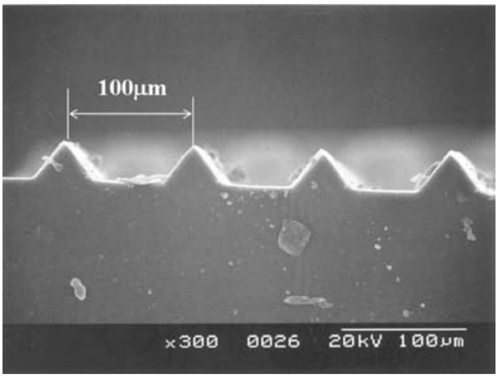

Fig. 5 Fabrication of Si trench structure

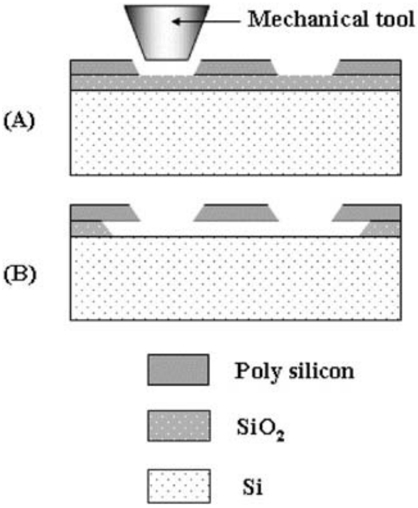

Fig. 6 Fabrication process of poly silicon beam using mechanically assisted chemical etching process

Table 4 Conditions of mechanical micromachining.

Mechanical tool	Material	Diamond, WC
	Radius (μm)	5, 10, 50
Machining parameters	Machining speed (μm/sec)	10, 100, 500, 1000
	Working load (mN)	10～50

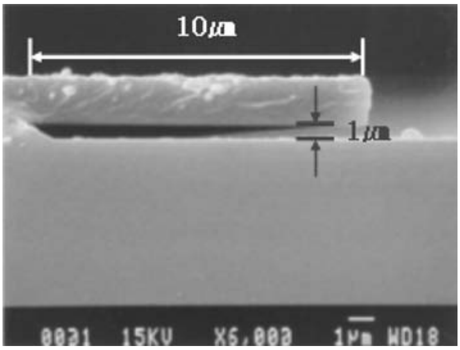

Fig. 7 SEM image of fabricated poly silicon beam. (A) Sectional diagram of posited SiO_2 (1 μm), poly silicon film (2 μm) on Si substrate. (B) Section diagram of poly silicon micro-beam (10 μm)

poly silicon beam are shown in Table 4. For etching the resistant layer, an isotopic etching buffered with HF was used. This solution etches SiO_2 at a rate of 100 nm/min, and the creation of an opening to the underlying substrate takes about 10 min. It is possible to optically monitor the progress of etching. From the experimental results of poly silicon films, poly silicon beams were fabricated as shown in Fig. 3.10

3.2 Chemically assisted mechanical micromachining

The chemical solution generally etches the surface material; as a result, the chemical attacked surface has a higher binding energy than the raw material. When the concentration of the solution was strong, the grains of materials were removed from the raw material. Therefore, if the chemical solution was controlled, the material was easily removed by a mechanical tool. This was the motivation of this work.

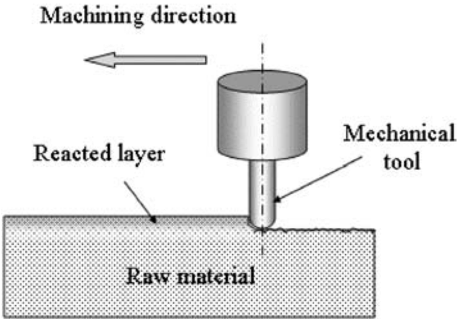

Fig. 8 Chemically assisted mechanical micromachining.

The basic procedure of chemically assisted mechanical micromachining is described as the following. First, the chemical solution reacts with the surface of the material.

Second, the reaction produces the chemically reacted layer, which might have changed mechanical properties.

Finally, the reacted layer with/without a substrate layer is machined by the mechanical tool. In the chemically assisted mechanical micromachining process, the chemical function changes the material property. For example, a hard brittle material surface, such as a Si wafer, changes into a hydrated layer due to a KOH solution. Figure 8 shows a machining schematic diagram of the suggested process, and the mechanical conditions are also shown in Table 4. The evaluation method of the chemically reacted layer is suggested. The layer generated by a chemical solution was analyzed in terms of its thickness, hardness change, and its mechanical property. The friction coefficient was measured to evaluate the dynamic properties of the surface. The generation of the plow by the relative action of tip and substrate changes the dynamic fiction coefficient. The test results show that the friction coefficients of the surfaces reacted on by KOH solution were increased over two times compared with bare surfaces below 5 mN (Fig. 9). As the scratch distance increased from 60 μm (measured corresponding load was 10 mN) to 150 μm, the differences in the friction coefficient of the surfaces were smaller because deep penetration decreases the effect of a chemically reacted layer. Accord-

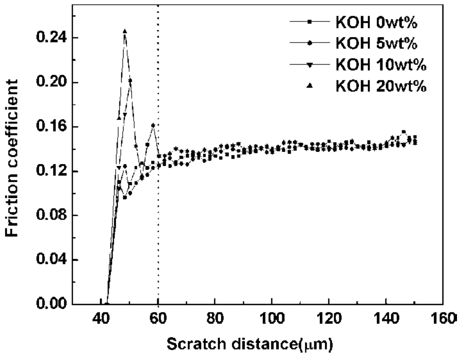

Fig. 9 Relation between scratch distances and friction coefficient

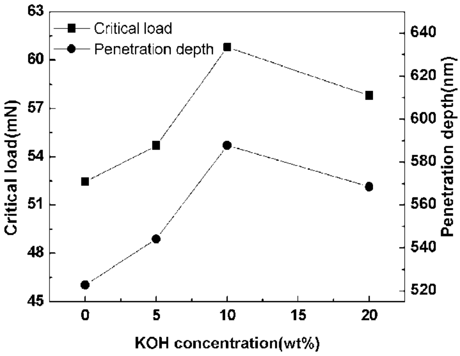

Fig. 10 Penetration depth and critical load as various chemical solutions

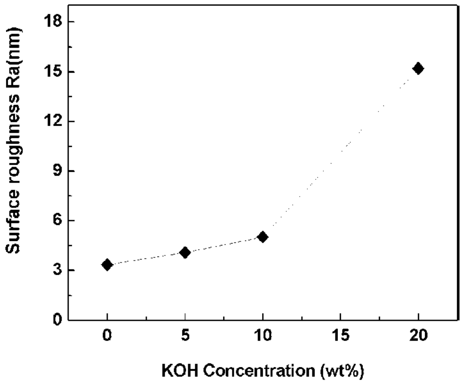

Fig. 11 Surface roughness of chemically reacted surface of Si wafers

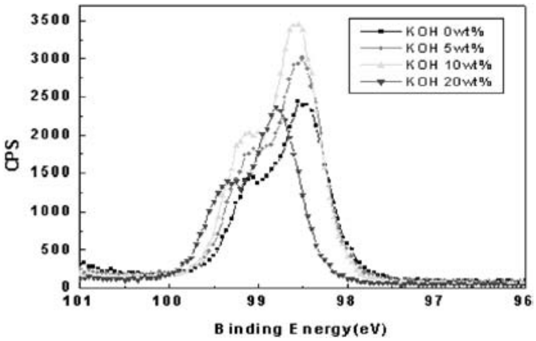

Fig. 12 XPS spectrum of Si wafers

ing to the result, it is thought that the maximum working force should be limited below 10 mN for this study. The scratch test results of the chemically reacted layer are shown in Fig. 10. The critical load for the fracture of the material increased until the concentration of KOH reached 10wt%, and then the load decreased at 20wt% concentration. Also, the penetration depth had the same trend as the critical load. Because of an increase in critical load and the formation of a weakened surface layer, crack resistance was enhanced and also critical penetration depth was increased. However, the silicon was etched in a solution of 20wt% concentration. Hence, the etching of the material at the surface of silicon reduces the thickness of the reacted layer, which is thought to be the reason for the drop in crack resistance. Therefore, 10wt% KOH concentration and 10 mN working force was chosen for the micromachining condition of silicon material. With an increase in chemical solution concentration, surface roughness is shown in Fig. 11. The material surface with a chemical concentration that increased gradually changed into a coarse surface, from 5.02 nm (KOH 10wt%) to 15.20 nm (KOH 20wt%), three times as coarse. It is thought that it results in a pitting phenomena caused by chemical etching. Therefore, it was necessary to decide the optimal domain concentration for the chemical solution to apply in mechanical micromachining. To help choose the optimal domain concentration for machining, the binding energy of the material surface was investigated by on X-ray diffractometer. In general, EDS is used for elemental analyses in different regions on the single crystal silicon surface. However, EDS analyzes a subsurface volume of material several microns thick, so it cannot be used to analyze changes in the immediate vicinity of the surface. Therefore, XPS was used for the chemical composition analyses of the sample surface. Figure 12 shows the results of the X-ray diffractometer. The binding energy was not changed in the domain of KOH 10wt%, but only the number of atoms increased. On the other hand, the binding energy was changed from 98.5 (eV) to 99 (eV) at KOH 20wt%. The reason is that a phase change occurred at the material surface.

From these results of material surface change as chemical concentration increased, it is thought that the optimal domain condition for this work is about KOH 10wt%. Microchannels are a basic structure in microfluidics. In general, the dimension required for microchannel is several hundred micrometers in width of the groove and several tens of micrometers in depth of the channel. Microchannel with 100 μm in width and 50 μm in depth was fabricated. Machining conditions for the fabrication of microchannels are presented in Table 5.

Table 5 Conditions for fabrication of microchannel

Material	Undoped Si (110) wafer	
Chemical	Solution	KOH 10wt%
	pH	12
	Buffer	Acetic acid
	Dipping time	During grooving
	Temperature	Room temperature
Mechanical	Tool radius	50 μm
	Machining velocity	10 μm/sec
	Working load	10 mN
	Penetration depth	0.2 μm/tool path

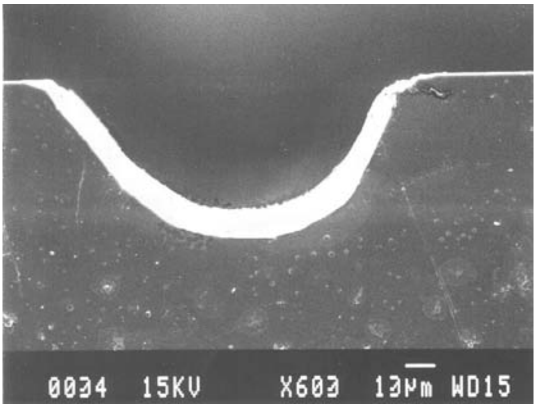

Fig. 13 Fabricated microchannel (Si wafer)

4. Conclusion

A Chemical mechanical micromachining process was proposed in this study. The process was divided into two methods: a mechanically assisted chemical etching process and chemically assisted mechanical micromachining. The motivation for the suggested process was to develop a machining process without a photo-lithographic mask in silicon machining and to give the advantages of flexibility and machine-ability to a machining system. The properties of materials, such as Si wafers, poly silicon, and SiO_2, were observed. From those studies, Si trench structures and poly silicon beam were fabricated by using mechanically assisted a chemical etching process. The basic principles of the chemically assisted mechanical micromachining process were introduced, and formation and analysis of a chemically reacted layer at different chemical concentrations were described. It is an important prerequisite that these requirements are correctly and thoroughly investigated and identified prior to being applied to silicon machining. Understanding the properties of materials, such as Si, poly silicon, and SiO_2, is indispensable when being combined with silicon machining and precision machining. To comprehend mechanical properties, the scratch test was carried out and the results were applied to the fabrication of microstructures, such as Si trenches and ploy silicon beams. Chemically assisted mechanical micromachining was directly applied to the machining of brittle material (silicon wafer) and did not used a photo-lithographic mask. It was applied in fabricating microchannels of 100 μm in width, 50 μm in depth. It is possible to fabricate a high aspect ratio microstructure with the suggested process.

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