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STUDY OF THE DISTORTED LAYER STRUCTURE OF SILICON WAFERS BY THE METHOD OF PLASMA-CHEMICAL ETCHING AFTER MECHANICAL MACHINING PROCESSES*

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Abstract – In this experimental work, by using the method of plasma-chemical etching, we have dealt with the causes of the creation of a distorted layer on the surface of silicon wafers during mechanical machining processes, in addition, the elucidation of connections between the structure of this layer and characteristic parameters of the mechanical strength of these wafers have been studied. Experimental results obtained at room temperature show that after cutting and grinding processes, the mean value of mechanical strength σ, which is apparently independent of the types of conductivity, is significantly lower than its theoretical value. Analysis of the dependence of mechanical parameters on the time of plasma-chemical etching indicates that the lower values obtained for the mechanical strength of silicon wafers is basically due to the existence of a distorted layer and corresponding internal stresses created on the surface of these wafers after mechanical machining. Plasma-chemical etching leads to an increase in σ value. Dependency of σ on the etching time is qualitatively described by the microstructure of the distorted layer and parameters of the micro relief surface of the wafers. Correlation between parameters σ, H, K and the microstructure of the distorted layer allows us to suggest the method of plasma chemical etching as a method of investigating the microstructure of the distorted layer after the mechanical machining processes.

Keywords – Mechanical properties, silicon wafers, cutting, grinding and etching

1. INTRODUCTION

The production of active elements of integrated circuits and structures by group technology on a single silicon wafer takes up a significant place in the fabrication of up-to-date semiconductor devices. It should be mentioned that production of reliable, high quality and economic devices and integrated circuits include different mechanical and thermal processing of initial semiconductor materials. Mechanical cutting, grinding, chemical etching, diffusion and etc. are related to this processing. As a rule, these operations lead to structural changes in both the bulk and surface of the wafers. In particular, as a result of mechanical cutting, grinding and polishing, a distorted layer is formed on the surface of the wafer due to the interaction of diamond powder granules with a machined surface. The thickness of the distorted layer is one of the basic factors which serves as a criterion for the quality of the machined surface of semiconductors.

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The existence of the distorted layer on the surface of the wafer sharply decreases the lifetime of charge carriers, increases the leakage current and strongly decreases the mechanical strength of the wafers.

Many works have been initiated to investigate the structure of the distorted layer and its influence on the mechanical strength of semiconductor wafers [1-3]. Analysis of results obtained by different authors shows that most of the depth of the distorted layer is created during the cutting of ingot into wafers. According to the work [1], the value obtained for the mechanical strength, after the removal of 116 μm from the surface of the wafer, is equal to 8.8 GPa.

In the work [2], maximum value of the strength was obtained after removing the distorted layer with a thickness equal to 18 μm from both sides of wafers. According to the work [6], the strength of the silicon wafer after cutting process is equal to 0.05-0.15GPa, and it is increased to 2.0-4.0 GPa, and in certain samples to 6GPa, after removing a layer with a thickness of 100 micrometers from both sides of the wafer by chemical polishing. In the work [4], it is shown that by removing a layer with a thickness of 150-200 μm from both sides of the silicon wafer by chemical etching, the value of the mechanical strength may be increased to 6-8 GPa.

Thus, analysis of the above mentioned works shows that surface conditions of silicon wafers strongly influence the value of mechanical strength σ. Probably the observed scattered data for mechanical strength are connected with the quantity and characteristics of residual defects which exist within the distorted layer created on the surface of silicon wafers. Moreover, the existing data concerning the removed depth from the distorted layer in order to get large values for mechanical strength are contradictory. Therefore, from a brief analysis of literature data, it follows that the problem of increasing the surface quality and mechanical strength of silicon wafers requires further investigation.

The present work is initiated to investigate the influence of cutting and grinding on the mechanical strength σ, microhardness H and fracture toughness K of silicon wafers, and also to study the structure of the distorted layer created on the surface of these wafers after mechanical machining processes using the plasma-chemical etching method.

2. EXPERIMENTAL RESULTS AND DISCUSSIONS

The investigations have been carried out on wafers of silicon single crystal with p-Si(B) of 0.03 Ω.cm. and n-Si(Sb) of 0.01Ω .cm. resistivites, respectively. The investigated samples were 100 mm in diameter, having the following orientations: (111) with 4° disorientation, (100) and (110) without disorientations.

Mechanical strength σ values were determined using a semi- automatic apparatus designed and constructed on the base of the bending methods of hard plates having symmetrical axis [5].

Mechanical strength σ values were determined by the relation

$$\sigma = 3P\,[(1+\nu)\mathrm{Ln}\,(a/r)+(1-\nu)(a^2-r^2)/(2b^2)]/[2\pi h^2] \qquad (1)$$

where a is the radius of fulcrum, b is the radius of a round wafer, which is related to the sides of a square wafer with the relation $b = b'.\,(1+\sqrt{2})/2$, r, p, h and ν are the radius of puanson, the applied load, the thickness of wafer and coefficient of Poisson, respectively.

The investigated silicon wafers having a 100 mm diameter were cut by a cutting machine from ingot.

To remove different crystal defects such as scratches, cracks and etc. from the surface of wafers after mechanical cutting, a number of 30 wafers were subjected to mechanical grinding. Grinding processes were carried out by consecutive changes of free rotating diamond particles having sizes in the range (25-5)μm. Measured mean values of mechanical strength σ are given in Table 1. It is seen from Table 1 that:

Table 1. Measured mean values of mechanical strength σ

Process	Cutting			One side ground wafers			Two side ground wafers		
Orientation	111	110	100	111	110	100	111	110	100
σ (MPa)	188	165	130	130	124	116	110	101	105

a) After cutting and grinding processes, the mean value of mechanical strength σ, which is apparently independent of the types of conductivity, is significantly lower than its theoretical value.

b) After grinding one side of the wafers, the value of mechanical strength (σ) is about 1.5-2 times smaller than its value after cutting processes.

The lower values of σ obtained for one side ground samples can be due to the creation occurrence of non-symmetric stresses by which they can be bent. Furthermore, the free abrasive particles used in the grinding process could be regarded as tiny diamond pyramids used in the Vikkers and Knoop methods. During the grinding process, free diamond particles produce micro shocks to the surface of the wafer, creating scratches, cracks and so on. The main part of these defects is localized within the surface with several microns in thickness.

Significant parts of stresses are distributed in this area. Consequently, structure infringement takes place and a distorted layer is created on the surface of the wafer. Physical and chemical properties of this layer are significantly different from that of the base single crystal. The depth of the distorted layer is proportional to the size of the granule of diamond powder. The structure of this layer is complicated and conventionally divided into the following three sub-layers [6]:

1) Relief sub-layer (with thickness 1-5μm), consisting of chaotically situated ledges and cavities.

2) Layer with cracks (with a thickness of 10-50μm) consisting of blocks of micro-cracks and localized dislocations.

3) Layer with stress, in which the cracks are absent and only consist of bulk elastic stresses and separated localized dislocations.

It should be mentioned that between these sub-layers there is no common border, they are somehow inter-penetrated. From the above analysis, it follows that the distorted layer and corresponding internal stresses may account for the lower value obtained for σ after processing. From this view point, it is of great interest to study the structure of the distorted layer, both quantitatively and qualitatively after the grinding process by etching. A plasma-chemical etching (PCE) process was accomplished in the PCE-100T apparatus in the interval (0-45) min with an etching rate of ~ 15 nm/s. To proceed PCE processes, different compositions of the CCl_4, CF_4, O_2 gases were used.

Mechanical strength (σ), microhardness (**H**), fracture toughness (**K**), critical length of micro cracks of the distorted layer (**L**) and length of cracks around the pyramid imprint (**C**) were taken as characteristic parameters for this investigation. The average height of cavity depth (**h**) and linear density of micro-hills (**Z**), microhardness (**H**) and fracture toughness (**K**) were determined by the identification of Vikkers pyramid imprints using the following relations:

$$H=1854 \times 10^{7} P/d^2 \quad (2)$$

$$K= 28 \times 10^{-6} a\sqrt{EH/C^3} \quad (3)$$

$$L_{crit}= 2\gamma E/(\pi\sigma^{2}) \quad (4)$$

Where, **p, d, E, H,** and C are the applied load (g), the imprint diagonal (μm), Young's modulus (Pa), **H-** is the microhardness (Pa), the half of the imprint diameter (μm), the average length of cracks around the imprint (μm), respectively [7]. Parameters H and K are both determined by the same pyramid imprints on the wafer and for determination of their average values, 20 imprints have been used. Results of PCE experiments are presented in figures (1-5). Peculiarities of the investigated parameters are as follows:

1) Microhardness **H** is increased by increasing the time of etching and reaches a maximum at (t=4 min). It is decreased by further increase of etching time and after (t≥20 min.), remains nearly constant.
2) Fracture toughness (**K**) is also increased by increasing the time of etching and at (t≥10 min), reaches a maximum and then is monotonically decreased by the time of etching.
3) Critical length of micro cracks **L** within the distorted layer is sharply decreased by increasing the etching time and at t ≥(8-10) min., it remains constant.
4) Length of crack **C** around the imprint is decreased by increasing the etching time and at t ≥(8-10) min. it becomes stabilized and then remains constant .

Dependence of mechanical strength on the time etching has a relatively complex character. As is seen from Fig. 1:

I) In the time interval t = (0-4) min. mechanical strength is sharply increased.

II) In the time interval t = (4-6) min. It monotonically increased.

III) In the time interval t = (6-10) min. it increases, reaching a maximum, and then by increasing the etching time it reaches a constant value.

Now we analyze the obtained PCE data on the base of the structure of a previously discussed distorted layer created on the surface of wafers after mechanical processing.

Drastic increase of σ, H and K values in the interval t=(0-4)min. (see Figs. 1-3) and sharp decrease of C (Fig. 4) and L(Fig. 5) values at the same time interval indicate that the significant parts of micro and macro damages have been intensively removed from the surface of the wafer by the etching process. As seen from Fig. 6, by increasing the time of etching, the (Z) value is decreased and (h) value increased, which is further evidence for the above mentioned removal of micro-and macro damages created on the surface of wafers after mechanical machining. In other words, in the above time interval, about ~4μm from the relief sub-layer of the distorted layer is removed by etching.

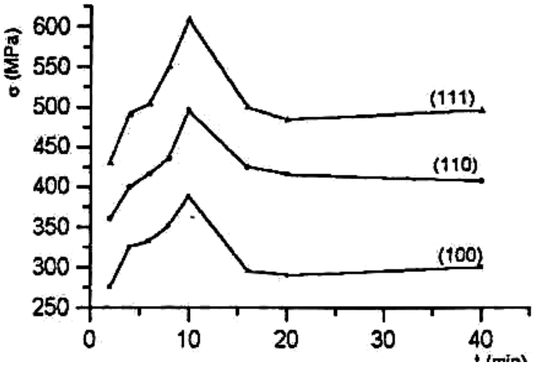

Fig. 1. Dependence of mechanical strength σ on the chemical etching time (t)

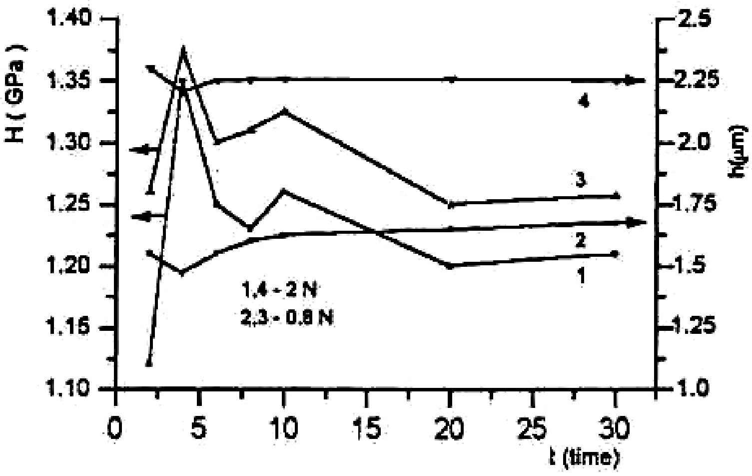

Fig. 2. Dependence of microhardness, H and h on the chemical etching time (t)

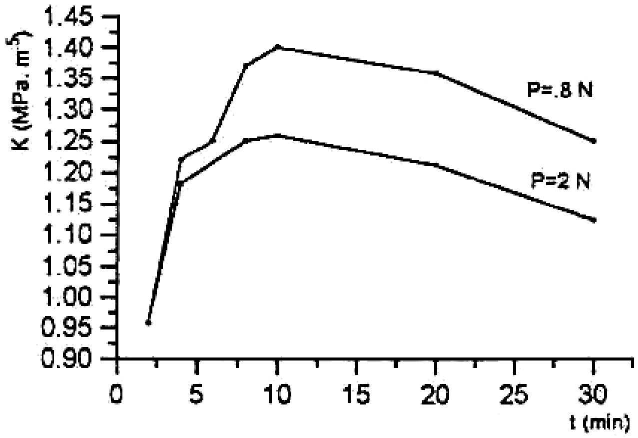

Fig. 3. Dependence of fracture toughness, K on the chemical etching time (t)

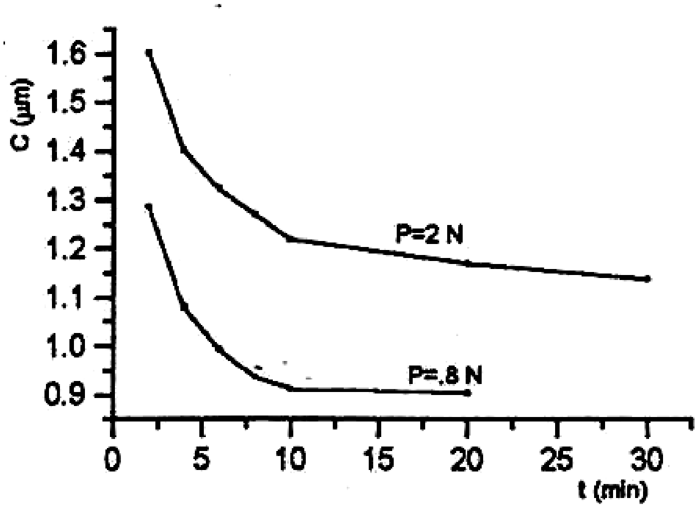

Fig. 4. Dependence of C on the chemical etching time (t)

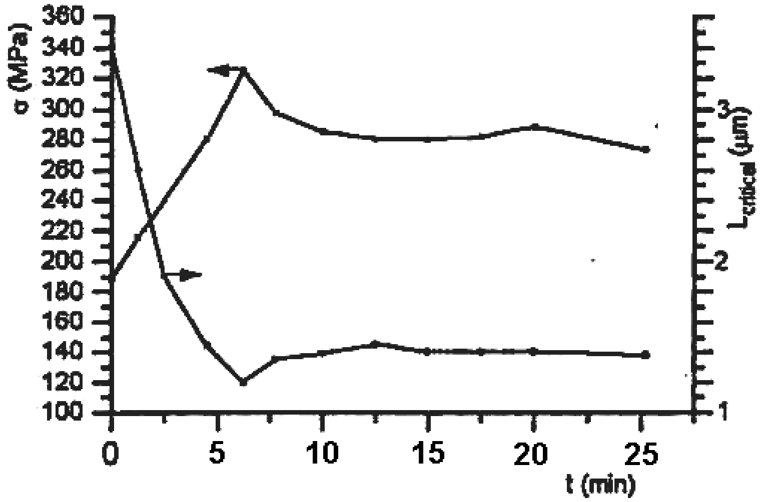

Fig. 5. Dependence of mechanical strength, σ and L_{crit} on the annealing time (t)

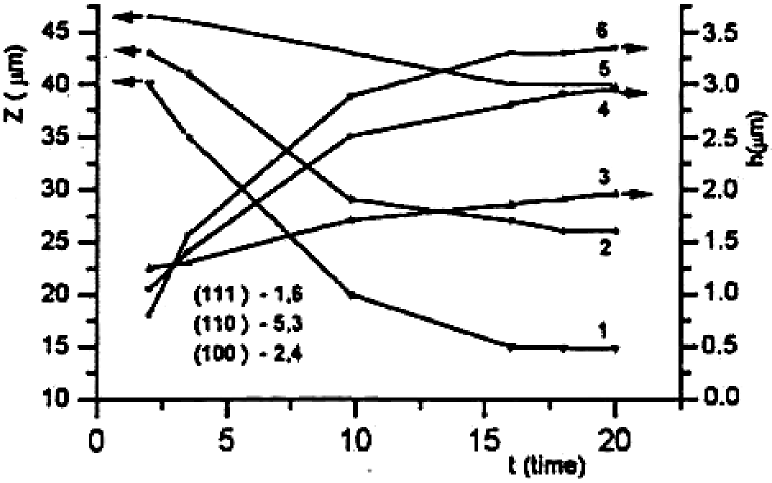

Fig. 6. Dependence of Z and h on the chemical etching time (t), Z : linear density of micro aspirates h: max. height of cavity depth

In the time interval t≥(6-8) min., h and Z reach stable values, σ and **K** are monotonically increased (see Figs. 1, 3), and H, L, and C decreased (see Figs. 2, 4, 5).

Removing the relief sub-layer and some parts of the crack zone causes the relaxation of the distorted layer mechanical stresses to occur. From one side the surface quality of wafers is increased, and from the other side, depth and also width of dimples due to etching process are increased. It is probable that interaction of these factors leads to a monotonous rise of σ and K, and a decrease of H values. Decreased values of H, C and L are proof of the improvement of the surface quality of wafers by increasing the etching time.

Increasing the etching time further in the time interval $6 \leq t \leq 10$ min. leads to an increase in the number of etch dimples. Basically, the etching process occurs in the crack zone, where the thickness of the etching layer is ≅ 9-15μm. In this time interval the number of micro-hills is strongly decreased, while their heights are increased. Lengths of both micro-cracks C and L are decreased, reaching constant values. A combination of these factors causes the σ and K to increase and reach maximum values, while by increasing the etching time they tend to decrease.

One reason for the decreased σ and K values by increasing the etching time t≥10 min., is that the height of micro-hills (h) is increased and their linear density (Z) is decreased by increasing the etching time. In this case, the depth of etch dimples is not increased, but their horizontal size is increased. In agreement with the work [8], this causes the concentration of stresses to increase, which in turn makes the σ and K values to decrease.

Thus, analysis of the obtained results indicate that there is a close correlation between the characteristic parameters of σ, H, K, etc., and the micro-structure of the distorted layer created on the surface of wafers after mechanical machining processes.

It is concluded that at initial parts of etching , t=0-10min., values of mechanical strength and other parameters are determined by the thickness and also micro-structure of the distorted layer, while by increasing the etching time t ≥10 min ,they are determined by changing the parameters of the micro-relief surface of the wafers. Moreover, the obtained correlation between parameters σ, H, K and the micro-structure of the distorted layer allows us to suggest the method of plasma chemical etching as a suitable method to investigate the micro structure of the distorted layer after mechanical machining processes.

3. CONCLUSION

The highlights of the experimental results obtained from this investigation can be summarized as:

1) The experimental value of mechanical strength σ, which is apparently independent of the types of conductivity, is significantly lower than its theoretical value after the wafers have undergone cutting and grinding processes.

2) Lower values obtained for mechanical strength after machining processes are described on the base of the availability of the distorted layer and corresponding internal stresses created on the surface of wafers.

3) Plasma-chemical etching leads to an increase in the value of mechanical strength.

4) Dependency of mechanical strength on the etching time is qualitatively described by the microstructure of the distorted layer and parameters of micro relief surface of the wafers.

5) Correlation between parameters σ, H ,K and the microstructure of the distorted layer allows us to suggest the method of plasma chemical etching as a suitable method of investigating the microstructure of the distorted layer after the mechanical machining processes.

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