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The effect of annealing on residual stress and dislocation propagation in silicon slices with damaged layer induced by scribing

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The effect of annealing on residual stress and dislocation propagation in silicon slices with a damaged layer induced by diamond scribing, laser scribing and diamond blade cutting was studied by infra-red photoelastic measurements and dislocation pit observations. Residual stress and dislocation propagation both showed clear annealing temperature dependence at temperatures above 500° C, although the residual stress was greatly reduced by a small degree of dislocation propagation. The experimental results can be explained using the stress recovery theory by the model of the damaged layer with a mosaic crystal layer and a single crystal layer with micro-cracks and dislocations.

1. Introduction

There are many mechanical processes involved in the fabrication of semiconductor devices. Elastic and plastic deformations induced by these processes affect the yield as a result of pellet cracking or device leakage problems. Scribing is one such process. Several groups have published work on defect observations for scratches in silicon [1–3]; in these works the silicon specimens are characterized by X-ray topography and the dislocation pit method after being annealing at temperatures above 900° C. Mirsa and Finnie have observed three types of cracks induced by scribing and have discussed the scribing direction [4]. Residual stress induced in the process of semiconductor device fabrication was first quantitatively observed by Kotake and Takasu using the infra-red photoelastic technique [5]. According to that study, the maximum residual stress was 60 kg cm^{-2} for diamond scribing (4-point scriber, load: 150 g, scribing speed: 50 mm sec^{-1}), 36 kg cm^{-2} for laser scribing (laser power: 7 W at 25 kHz, groove depth: 160 μm, scribing speed: 5 mm sec^{-1}), and 4 kg cm^{-2} for diamond blade cutting (blade width: 40 μm, blade radius: 25.3 mm, working at 30 000 rpm, cutting speed: 70 mm sec^{-1}, groove depth: 270 μm). The direction of the stress gradient for the laser scribing was opposite to that used for the other scribing methods. Dislocation pits were also observed after annealing at a temperature of 1000° C for 30 min and Wright etching. However, the effect of annealing on elastic and plastic deformation was not discussed in that work [5].

The purpose of this paper is to report the relation between the annealing temperature and the elastic and plastic deformation, through infra-red photoelastic measurements of residual stress and dislocation pit observations. Although mechanical damage was caused by scribing in this experiment, the results obtained can be applied to all mechanical processes.

2. Experimental procedure

2.1. Photoelastic measurement

Silicon is transparent to radiation of wavelength longer than 1.1 μm. Photoelastic observations were made using a PbO–PbS Vidicon. It is considered that the observations were made with nearly monochromatic radiation of 1.1 μm wavelength for the combination of silicon filter and PbO–PbS Vidicon. The infra-red photoelastic apparatus is shown in Fig. 1 and the quantitative measurement procedure is shown in Fig. 2 [6].

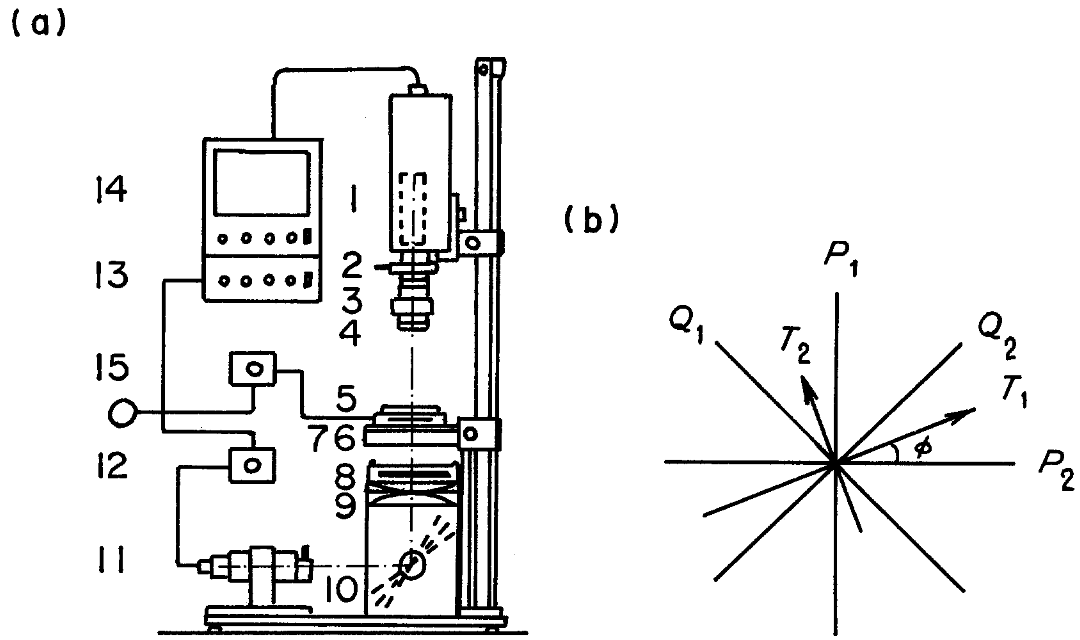


Figure 1 Infrared photoelastic apparatus. (a) Macroscope: 1 infrared Vidicon camera; 2 infrared analyser (P_2); 3 lens; 4 quarter wave plate (Q_2); 5 heating stage; 6 specimen; 7 quarter wave plate (Q_1); 8 infrared polarizer (P_1); 9 condenser; 10 mirror; 11 light source controller; 13 camera controller; 14 monitor TV; 15 heating stage controller. (b) Direction of principal stresses in the specimen (T_1 and T_2); ϕ is the angle between T_1 and P_2.

Principal stress lines were obtained by observation with a plane polariscope; stress magnitude was determined by observation with a circular polariscope. Stress values were obtained by using

$$T_1 - T_k = \frac{2}{n_0^3 A} \frac{h\lambda}{d}, \qquad (1)$$

where T_1–T_k is the difference in magnitude between the two principal stresses, T_1 and T_k, n_0 is the refractive index in the silicon crystal free from stress ($n_0 = 3.4$), λ is the wave length ($\lambda = 1.1\ \mu$m), h is the retardation measured in the wavelength unit λ, d is the light path length in the crystal and the constant A is expressed in the formula of piezo-optical coefficients. The values of $T_1 - T_k$ for $d = 1$ cm and $h = 1$ are shown in Table I, and from Equation 1 it is possible to get the stress value from the value of the retardation experimentally obtained.

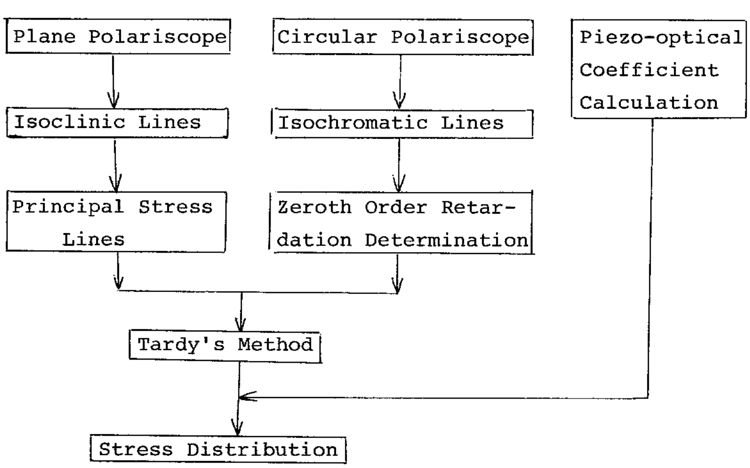


Figure 2 Quantitative stress measurement procedure of the stress in the crystal by photo-elasticity.

TABLE I Principal stress difference which causes a retardation of $1 \times \lambda (\lambda = 1.1\ \mu m)$ in 1 cm light path.

Observation direction	Principal stress directions	Principal stress difference for retardation of $1 \times \lambda$ (kg cm^{-2})
[0 0 1]	[1 0 0] [0 1 0]	36
[0 0 1]	[1 1 0] [$\bar{1}$ 1 0]	52
[$\bar{1}$ 1 0]	[1 1 0] [0 0 1]	43
[$\bar{1}$ $\bar{1}$ 1]	[1 1 2] [$\bar{1}$ 1 0]	45
[$\bar{1}$ 1 0]	[1 1 2] [$\bar{1}$ $\bar{1}$ 1]	50
[1 1 2]	[$\bar{1}$ 1 0] [$\bar{1}$ $\bar{1}$ 1]	50

2.2. Specimen preparation

Crystallographic orientations and the dimensions of specimens used for the measurement of residual stress induced by scribing are shown in Fig. 3. The specimens were cut from a crystal grown by the Czochralski method. All the surfaces were mirror polished. It was ascertained from the photoelastic observation that they were free from stress. The groove direction and the direction of observation are shown in the same figure. Scribing was made by a diamond 4-point scriber with a 400 g load and a 50 mm sec^{-1} scribing speed. All the specimens were checked by photoelastic observations to ensure that the pellets tested were ones with the same residual stress. Annealing was accomplished in a nitrogen gas atmosphere at temperatures of 300, 400, 500, 600, 700 and 900° C. The annealing times varied from 30 min to 40 h.

For the observations of dislocation pits induced by scribing, three kinds of specimens were prepared. They were (1 1 1) wafers scribed by a diamond scriber (4-point scriber, load: 150 g, scribing speed: 50 mm sec^{-1}), a laser scriber (laser power: 7 W at 25 kHz, scribing speed: 5 mm sec^{-1}, groove depth: 160 μm) and cut by a diamond blade (blade width: 40 μm, blade radius: 25.3 mm at 30 000 rpm, cutting speed: 70 mm sec^{-1}, groove depth: 270 μm). The direction of the scribing groove was ⟨1 1 0⟩. Specimens were annealed in a nitrogen gas atmosphere at 500, 600, 700 and 900° C for 30 min and etched for 2 min in the Wright etchant [7].

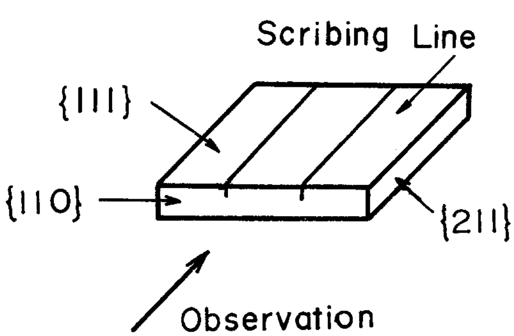


Figure 3 Specimens for the infra-red photoelastic measurement of the residual stress induced by scribing. Specimen dimensions are 5 mm × 5 mm × 1 mm.

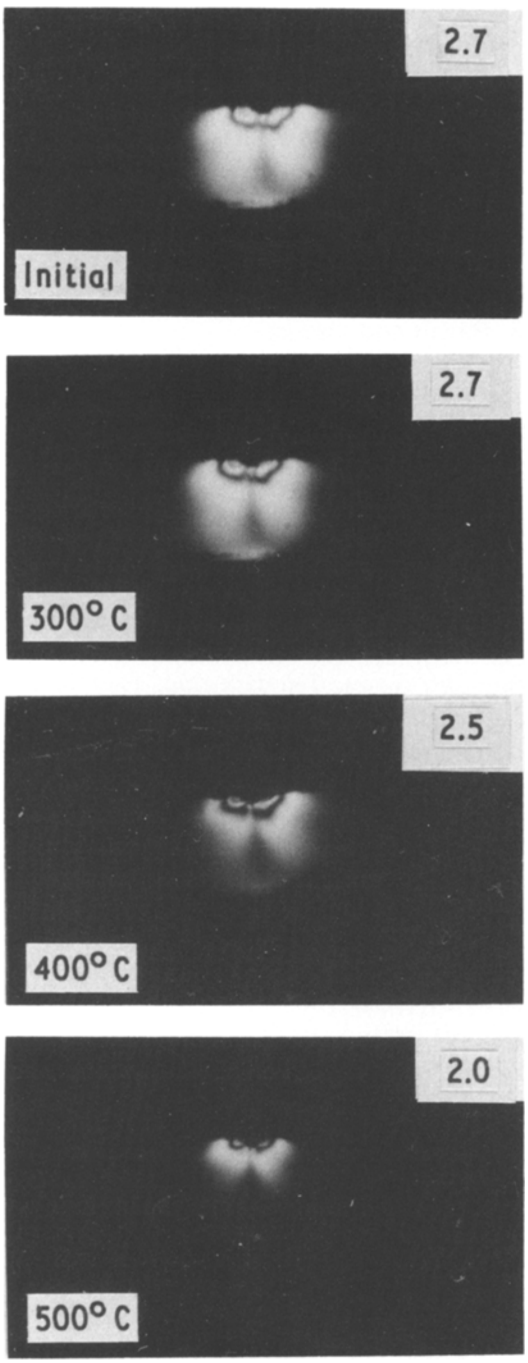
3. Result and discussion

3.1. Residual stress

For the measurement of the elastic deformation, specimens with a large residual stress were used so that the change after annealing could be clearly seen. The maximum stress in the specimen before annealing was determined to be 270 kg cm^{-2} by photoelastic measurement using Equation 1 and the values, 50 kg cm^{-2} in Table I and $d = 5$ mm and $h = 2.7$ from Fig. 4, where the changes of residual stress after annealing at various temperatures are shown. The dark field, apart from the scribing groove, means zero stress and other dark lines near the groove, which are called isochromatic lines, correspond to values of $h = 1, 2, \ldots$. The stress distribution did not change after annealing at 300° C for 30 min. Annealing at 400° C for 30 min reduced the stress only a little. A clear change in the residual stress was observed after annealing at temperatures above 500° C. The annealing effect was found to be the same for specimens annealed for varying times greater than 30 min; this was ascertained at various temperatures by comparing specimens annealed for 3 h with those annealed for 30 min. The result was also the same for the specimen annealed for 40 h at 500° C, the critical temperature for the stress reduction. It is clear that the reduction of the residual stress is accomplished by annealing for 30 min. The reduction of the maximum residual stress according to the annealing temperature is summarized in Fig. 5.

3.2. Dislocation propagation

The plastic deformation was measured by observing the dislocation propagation after annealing and Wright etching. Observed results are shown in Fig. 6 for various annealing temperatures and scribing methods. For the specimens scribed by the diamond scriber and the laser scriber, the distribution range of dislocation pits became smaller as the annealing temperature became lower. In case of the annealing at 500° C, dislo-



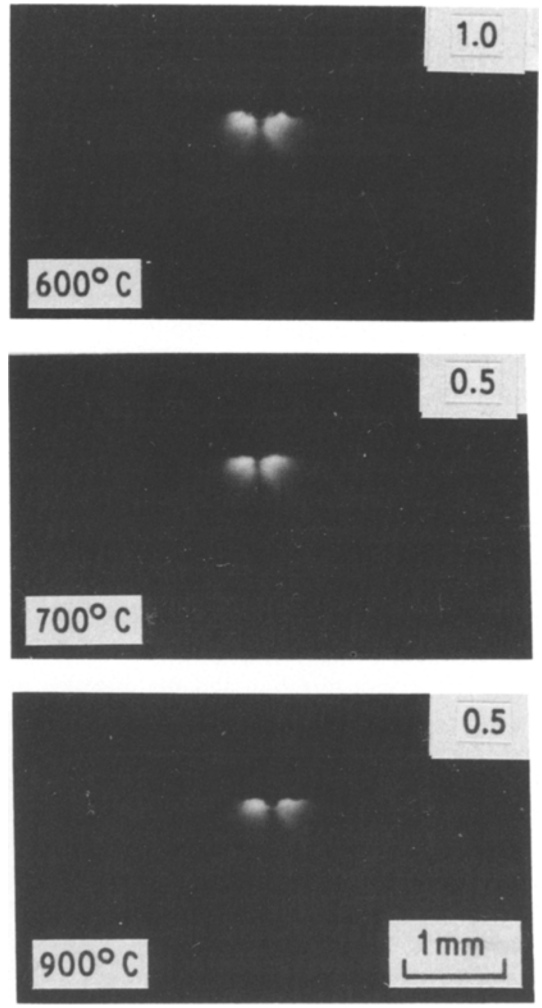


Figure 4 Dependence of the residual stress induced by scribing on the annealing temperature. Specimens were observed using the circular polariscope. Annealing was accomplished for 30 min in nitrogen gas atmosphere. Annealing temperature and observed maximum retardation h in the wavelength unit are shown for each photograph.

cation pits were observed only in the vicinity of the scribing groove. However, there were no dislocation pits observed in the specimen cut by the diamond blade. It has already been shown that the residual stress induced by diamond blade cutting is more than ten times smaller than the stress induced by the other scribing methods [5]. The temperature dependence of the dislocation propagation, which is shown in Fig. 7, was obtained by measuring the distribution distance of dislocation pits from the scribing groove edge. Measurements were performed on specimens scribed by the diamond scriber. It has become clear that the critical annealing temperature for dislocation propagation is about 500° C.

No dislocation pits were observed in the specimen for which the damaged layer was removed by etching in an etching solution (conc. HF (49%): conc. HNO_3 (70%): conc. CH_3COOH in a 1:3:2

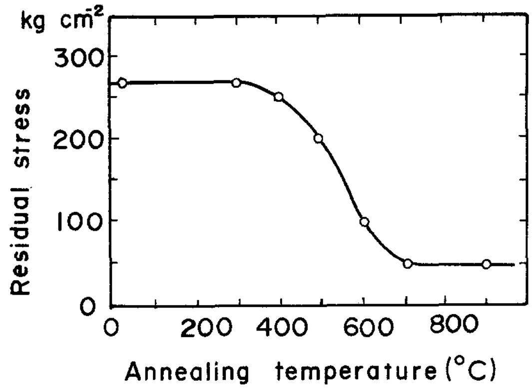


Figure 5 Change of the maximum residual stress induced by scribing after annealing for 30 min at various temperatures.

volume ratio) at 25° C for 1 min. This indicates that the dislocation propagation after annealing is produced by the damaged layer and is related to the residual stress, since the stress is also reduced by removing the damaged layer [5].

3.3. Discussion

The critical annealing temperatures was determined, from the results shown in Figs 5 and 7, to be about 500° C for both residual stress relaxation and dislocation propagation, although the residual stress was to a large extent reduced by a small plastic deformation. The reduction of residual stress after annealing corresponds to the dislocation propagation. The experimental results can be explained by considering the structure of the damaged layer. It is natural to propose the model that the mechanically damaged layer, caused for example by scribing, is formed of two parts: a mosaic crystal layer and a single crystal layer with micro-cracks and dislocations, although the interface is not always clear. The model is schematically shown in Fig. 8a. Annealing at high temperatures near the melting point leads to the recrystallization of the mosaic crystal layer, but annealing at low temperatures mainly produces the dislocation movement. Some dislocations move into the inner part of the crystal; the dislocation density becomes small and the layer with dislocations becomes thicker, as shown in Fig. 8b. The distribution of these dislocations can be observed by the etching method. However, it is hard to observe dislocations in the specimen annealed at low temperatures because the single crystal layer with high dislocation density is confined to the vicinity of the mosaic crystal layer. The residual stress measured by the photoelastic technique results mainly from the single crystal layer with micro-cracks and dislocations. The thin layer, with high dislocation density in the specimen before annealing, causes the large residual stress, and the thick layer with low dislocation density in the specimen annealed at low temperatures produces the small stress.

In the recovery theory in metals, there is a relation [8]

$$\frac{d\sigma}{dt} = -C \exp\{-(U-\beta\sigma)/kT\}, \qquad (2)$$

where σ is the stress, U is the activation energy, T is the temperature, k is the Boltzmann constant and C and β are constants. The equation shows that the rate of stress relaxation depends on both the temperature and the stress. A large stress produces a large reduction of the stress and a corresponding large shift of the dislocation distribution. This is why dislocation pits were not observed in specimens cut by the diamond blade, whose residual stress was much smaller than the stress in the specimens scribed by other methods [5]. After annealing at high temperatures, the residual stress becomes small and the rate of the stress relaxation also becomes small. The dislocation mobility is also small at this stage. The experimental results shown in Figs 5 and 7 can be explained by the model of the damped layer using Equation 2 in this way.

For dislocations and plastic flow in the diamond structure, many theoretical and experimental data were summarized by Alexander and Haasen [16]. The relation between dislocation movement and the stress and temperature was also explained in detail in their work. There are two theories for the stationary creep rate, $\dot{\epsilon}_w$, which is proportional to the dislocation velocity:

$$\dot{\epsilon}_w \propto \tau^{m+2} \exp(-U_c/kT), \qquad (3)$$

and

$$\dot{\epsilon}_w \propto \exp[-(U_c - \omega\tau)/kT], \qquad (4)$$

where τ is the stress, k is the Boltzmann constant, T is the temperature and U_c and ω are constants. It is said that experimental data fit a power law for the stress, Equation 3, rather than the exponential relation, Equation 4 [9]. Considering this fact, the formula of stress relaxation, Equation 2, might also be changed to a power law for the stress. Nevertheless, even in that case, the qualitative discussion of the recovery of the scribing

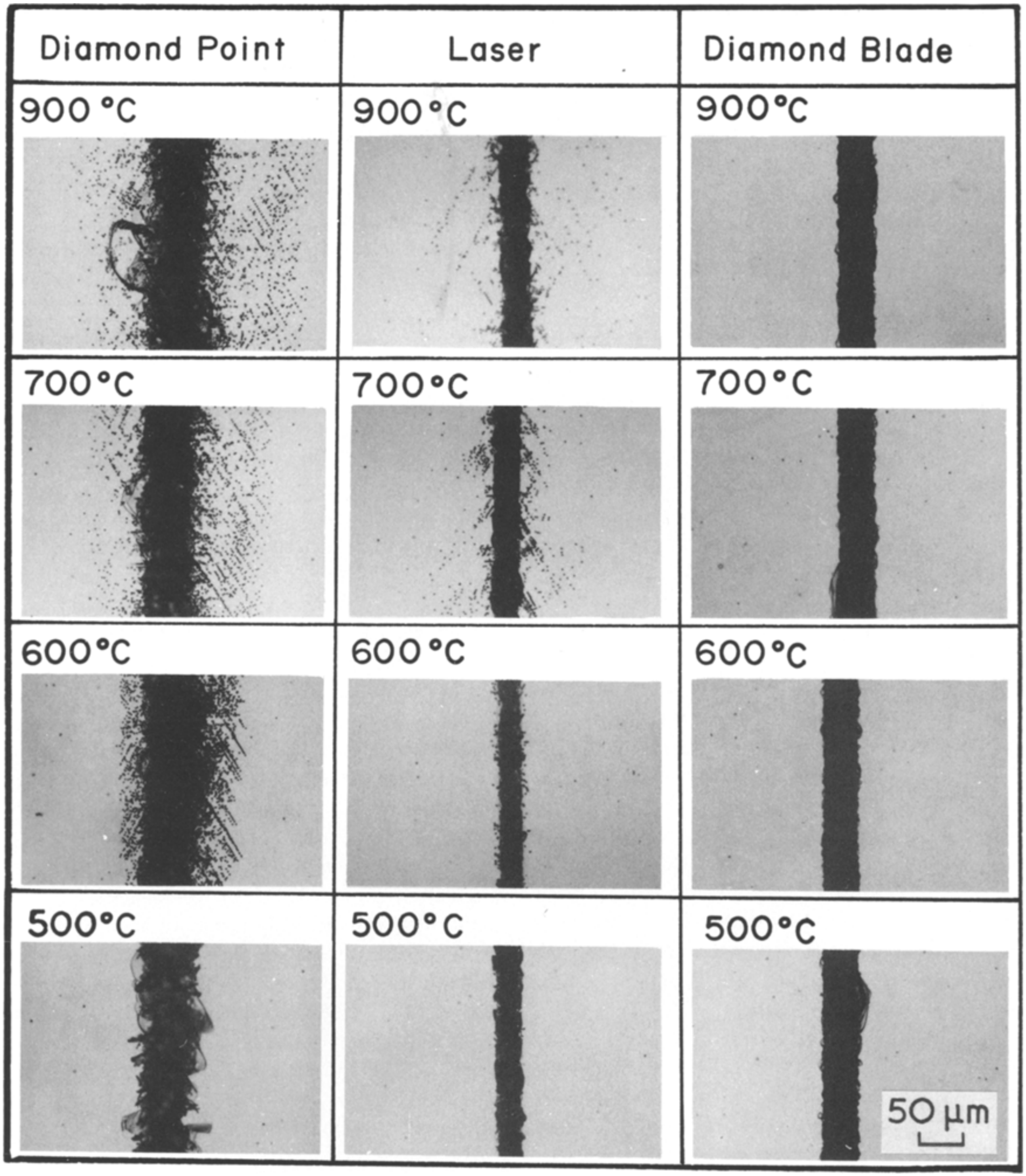


Figure 6 Dislocation pit distribution induced by scribing. Specimens were annealed for 30 min at various temperatures and etched by the Wright etchant.

induced stress by annealing remains the same as that related before.

For the dislocation movement, George *et al.* measured the velocity of screw and 60°-dislocations in silicon for various temperatures and stresses [10]. The velocities measured ranged from 5×10^{-6} cm sec^{-1} to 1.8×10^{-4} cm sec^{-1} at 700° C, for example. The distance of dislocation movement, after annealing for 30 min at this temperature, is calculated to be from 90 μm to 3.24 mm. Our experimental result for the dislocation movement measured from the spreading range of dislocation

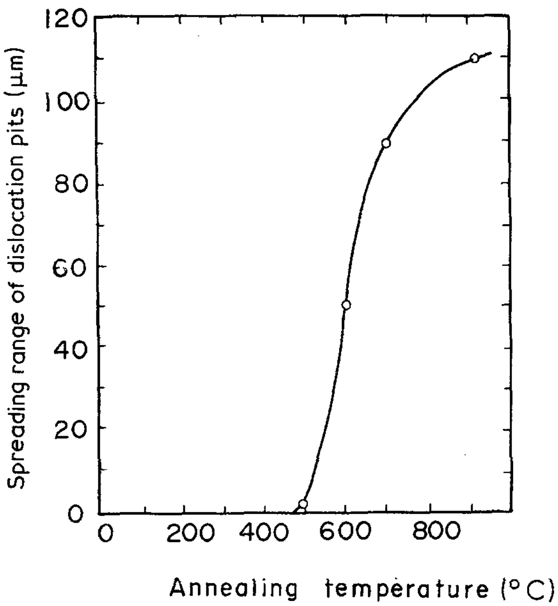


Figure 7 Temperature dependence of the dislocation propagation. The dislocation propagation range was obtained by measuring the distribution of the distance of dislocation pits from the scribing groove edge. Measurements were performed for the specimens scribed by the diamond scriber.

pits after annealing at 700° C for 30 min was 90 μm (Fig. 7). This is smaller than the mean value of dislocation movement calculated from the result obtained by George *et al.* The fact that the stress reduction and the dislocation propagation were accomplished by annealing for 30 minutes at high temperatures can be explained by the large velocity of the dislocation movement measured by George *et al.* [10].

Although the discussion on creep and the elastic/plastic transition was related to the well established mechanical behaviour of silicon by many researchers [9], this paper represents some new empirical results. They are the direct observation of the internal stress by photoelasticity, its dependence on the annealing temperature and the property of the mechanically damaged layer in silicon crystal.

4. Conclusion

The effect of annealing on elastic and plastic deformation in silicon was studied by infra-red photoelastic measurements of residual stress and dislocation pit observations for crystals mechanically damaged by scribing. The critical annealing temperature for both stress relaxation and dislocation propagation was about 500° C. A small degree of dislocation movement reduced the residual stress to a great extent. These phenomena were explained by the model of the damaged layer, which is formed by the mosaic crystal layer and the single crystal layer with microcracks and dislocations.

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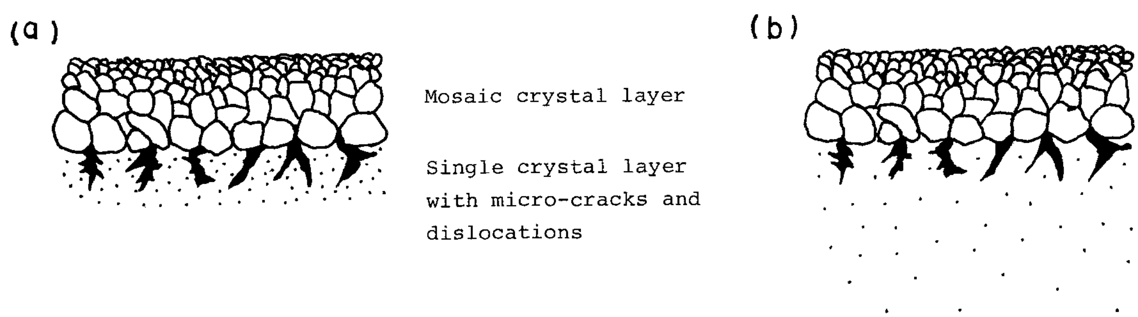

Figure 8 Model of the damaged layer, (a) before annealing; (b) after annealing at high temperatures. Dislocations are shown by dots.

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