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V. Pasetchny, V. Makara, V. Sizontov, O. Rudenko, G. Vesna. Laser-caused modification of structure and mechanical properties of silicon. Journal de Physique IV Proceedings, 1994, 04 (C4), pp.C4-167-C4-169. 10.1051/jp4:1994437 . jpa-00252700

HAL Id: jpa-00252700

<https://hal.science/jpa-00252700>

Submitted on 1 Jan 1994

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Laser-caused modification of structure and mechanical properties of silicon

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ABSTRACT

There were investigated laser annealing conditions which cause relaxation of microtensions near in proximity to dislocation sources. It was determined that density and efficiency of such sources may be varied through oxidation of Si crystallites. Utilization of the found conditions of thermal and laser annealing enabled the authors to set microhardness of porous Si and flexure of Si-SiO₂-Si (polycrystalline) structures.

INTRODUCTION

It is known that oxidation defects in silicon are efficient dislocation sources [1,2]. Thermal cycling as well as annealing of defects through laser radiation impulses allows to cause changes of dislocation sources state in proximity to which microtensions reached critical values. Intergrain borders also are dislocation sources in polycrystalline silicon. In the present work the authors manage to prove that through annealing in the atmosphere containing oxygen at temperature 1100-1200 as well as through laser annealing of the investigated object in the air it is possible to control plastification of monocrystalline and polycrystalline silicon.

EXPERIMENTAL PROCEDURE

The oxidation defects (OD) which are the complexes of SiO₂ inclusions, stacking faults and the ensembles of full and partial dislocations are widely known. Selecting the oxidation regimes that promote the growth of crystallized SiO₂ inclusions we have obtained the anomaly large OD (with linear size of 10 - 20 micrometer and even more) regular geometrical shape. Oxidation defects were chosen as dislocation sources.

Porous layers on monocrystal silicon surface were obtained by means of anodic etching in electrolytes containing HF in ratio 2:1 and 1:1 at currents $j=(9,0 \div 50,0) \mu\text{A}/\text{cm}^2$. The laser treatment of crystals was done by series of impulses with the length of 4 ms, $\lambda=1,06 \text{ mm}$ and energy from $1 \cdot 10^5 \text{ J}/\text{m}^2$ to $5 \cdot 10^5 \text{ J}/\text{m}^2$.

RESULTS AND DISCUSSION

Initiating of dislocation generation by OD may be achieved by irradiation of Si crystal surface by pulsed laser radiation with 1,06 micrometer wave length. The threshold energy which is necessary for dislocation generation by OD (at pulse duration of 4 ms) varies from $1 \cdot 10^5 \text{ J}/\text{m}^2$ to $5 \cdot 10^5 \text{ J}/\text{m}^2$ at sample temperature range 293 ... 573 K (Fig.1). This energy decreases with temperature increasing (Fig.2).

Fig.1. The temperature dependence of the threshold energy for various dislocation sources:
 $\circ$ - seratches, $\triangle$ - the oxidation defects.

Fig.2. Variation of dislocation ensembles paths with surface energy density of laser radiation at various temperatures of sample.

Unlike the thermal annealing the dislocation ensembles paths from sources is almost independent from internal stresses values, but is defined by falling pulses energy and the number of pulses. Long term irradiation of Si surface by pulsed laser with energy pulses about threshold energy is the most effective. It is possible to control both the dislocation ensembles paths and the dislocation density in the local areas of crystalsurface by the accumulation of irradiation dose.

Using of plastified annealing in oxygen ambience made it possible to control stress relaxation process and to handle bending of three-layer $\text{Si}_{\text{mono}} - \text{SiO}_2 - \text{Si}_{\text{poly}}$ structures. Oxidation of these structures (ratio of layer thickness was 50:1:20) in the taken regimes enables to shift the maximum of plates distribution bending by 40 ...60 micrometer (Fig.3a, 3b).

Fig.3a. The distribution of structure bending for three layers $\text{Si}_{\text{mono}} - \text{SiO}_2 - \text{Si}_{\text{poly}}$ silicon structures $h_{\text{Si}} = 500 \mu\text{m}$,
 $h_{\text{SiO}_2} = 1 \mu\text{m}$, $h_{\text{Si}_{\text{poly}}} = 20 \mu\text{m}$.

Fig.3b. The distribution shifting of structure bending after oxidized annealing.

We started to study the influence of long-time irradiation by low power impulses on micromechanical properties of porous silicon. It was found that at doses of 150 ...250 impulses with the energy of $1,25 \cdot 10^5 \text{ J/m}^2$ (with pulse duration of 4 msec) the decrease of microhardness take place. The microhardness of porous silicon samples after irradiation was 20 ...50% less (depending on the material density) than in initiation stage (Fig.4, 5).

Fig. 4. The microhardness dependence of the porous silicon on the laser irradiation dose. The loading on indenter is: 1(□) - 10 g, 2(Δ) - 25 g, 3(•) - 50 g.

Fig. 5. The microhardness dependence of the porous silicon on the laser irradiation dose $\rho_1 = 1.77 \text{ g/cm}^2$, $\rho_2 = 0.99 \text{ g/cm}^2$. The load on indenter is $p = 10 \text{ g}$.

(•) - $E_{\text{imp}} = 12.5 \text{ J/cm}^2$;
(o) - $E_{\text{imp}} = 16.0 \text{ J/cm}^2$;
(Δ) - $E_{\text{imp}} = 21.5 \text{ J/cm}^2$.

This confirms the prospects of laser annealing application for porous silicon layer plastification.

CONCLUSIONS

1. Introduction of dislocation sources and their controlled initiating by thermomechanical or laser effect makes it possible to handle micro- and macroplastic properties of silicon monocrystals or layered structures on its bases.
2. Effective dislocation sources in surface layers of Si samples can be obtained by silicon oxidation in temperature range $1050 \dots 1200^\circ\text{C}$. Density and power of sources can be controlled by the choice of oxidation and cooling regimes.
3. Using of plastified annealing in oxygen ambience made it possible to control stress relaxation process and to handle bending of three-layer $\text{Si}_{\text{mono}} - \text{SiO}_2 - \text{Si}_{\text{poly}}$ structures.
4. Stress relaxation and plastification of silicon surface under the effect of series laser impulses makes it possible to plastify porous silicon layers resulting in decrease of sample microhardness by $20 \dots 50\%$.

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