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Characterization of porous silicon by positron annihilation

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Abstract

Characterization of porous silicon was performed by means of positron annihilation. Three components of the positron lifetime were observed, one of which was extremely long. The Doppler-broadened spectrum was sharp compared with that of crystal silicon, and became even narrower in an applied magnetic field. These results show that the porous structure is the cause of positronium formation in porous silicon.

Introduction

Porous silicon is obtained by the anodization of a silicon crystal in hydrofluoric acid and consists of a dense network of fine pores. The physical and chemical properties of porous silicon have attracted strong attention as result of its photoluminescence phenomena/1/. The mechanism of the photoluminescence has been actively studied¹, while its utilization has been discussed as an optical device and a gas and humidity sensor. Positron annihilation spectroscopy is a powerful tool for the study of the physical and chemical properties of porous silicon. The porous silicon surface is covered by a Si-H layer/2/ and the study of this layer is interesting from a view point of positronium chemistry and surface science. Observation by high resolution transmission microscopy has shown that small single crystal silicon spheres are surrounded by oxide in the porous layer/3/. In addition, scanning tunneling microscopy and atomic force microscopy have revealed that the porous structure is partially covered by an amorphous surface layer/4/. The exact microstructure of porous silicon is however still not fully understood. We therefore expect that positronium annihilation, taking particular notice of the positronium yield, should reveal useful information regarding the atomic scale microstructure of porous silicon.

Experimental

The silicon substrates used were boron-doped wafers of 1~2 ohm-cm and 17~23 ohm-cm with (100) surface. The layer formed by anodization could be classified into three groups, according to the anodization conditions, as shown in Fig. 1. The porous layer obtained in region 1 had good quality and was used in our study. In region 3, electrolyte polishing was realized and region 2 forms a transition region between the formation of the porous layer and electrolyte polishing. The substrates were anodized in hydrofluoric acids of 20 wt.% - 55 wt.% at a constant current density ranging from 10 mA/cm² to 100 mA/cm² as indicated by the arrows in Fig. 1. The thickness of the porous layers ranged from 30 μ m to 100 μ m. The porosity, which is defined as the ratio of the reduced mass of the anodized layer to its mass before the anodization, varied from 0.5 to 0.7. Measurements of the positron lifetime and Doppler broadening were carried out on the anodized samples sandwiching a positron source of ²²NaCl or ⁴⁸V produced by the reaction of ⁴⁸Ti(p,n)⁴⁸V. The positron lifetime was measured with a fast-fast coincidence system whose time resolution was 210 ps. The Doppler spectrum was obtained with the aid of a pure Ge detector with an energy resolution of 1.07 keV. The lifetime spectra were analyzed with the computer program POSITRONFIT/5/.

Results and Discussion

The typical characteristics of two porous silicon sample are shown in Table 1.

Table 1. Comparison of lifetime and intensity in two kinds of porous silicon

Sample No. (Resistivity)	HF (%)	Current (mA/cm ²)	Thickness (μ m)	Porosity	$\tau_1(I_1)$ ps(%)	$\tau_2(I_2)$ ps(%)	$\tau_3(I_3)$ ps(%)
A (~2 Ω cm)	55	107	35	0.53	213(84)	510(8)	17300(8)
B (~23 Ω cm)	46	102	33	0.55	233(88)	1700(1)	22800(8)

Three components of the positron lifetime were observed in the porous silicon. τ_1 originated from the lifetime in the free state in the substrate crystal. τ_2 may be characteristic of positronium physisorbed on a small void surface or a positron trapped on its surface. Although it is hard to determine from the lifetime measurement whether τ_2 is attributed to physisorbed positronium or to the surface positron, this lifetime reveals that voids are formed in the porous layer. τ_3 is extremely long and reveals that ortho-positronium is formed in the porous silicon. The Doppler spectrum of the porous silicon is sharp compared with that for the crystalline silicon and became even narrower in an applied magnetic field,

as shown in Fig.2. This suggests para-positronium formation in the porous silicon and we conclude from these results that the porous structure is the cause of the positronium formation. It has recently been reported that positronium formation occurs in hydrogenated amorphous silicon but that no formation occurs in unhydrogenated amorphous silicon^{6/}. The present results suggest that some areas of the porous silicon, probably the void surfaces, have the same atomic structure as hydrogenated amorphous silicon. It is known that large amounts of absorbed hydrogen terminate the surface dangling bonds of porous silicon during anodization in HF solution. In conclusion it has been shown that positron annihilation spectroscopy reveals much information on the microstructure of porous silicon made by different anodization conditions, such as HF concentration and current density.

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Fig. 1: Three groups of anodization condition.

Fig.2: Doppler broadened Spectra of
(a) bulk silicon, (b) porous silicon (B=0)
and (c) porous silicon (B=0.3T).