PREPARATION OF ION-IMPLANTED SILICON FOR TRANSMISSION ELECTRON MICROSCOPY

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This paper deals with the preparation technique of thin foils (below 5000 Å) for the transmission electron microscopy from silicon implanted by N⁺ ions. Mechanical and chemical thinning and anodic oxidation techniques are discussed and selective etching of the surface of silicon for the purposes of the replica technique is noted. Compositions of etching solutions and other experimental parameters are mentioned. The results are demonstrated on electron micrographs showing the radiation damage due to ion implantation in silicon.

1. INTRODUCTION

In the last years ion implantation became a new and significant method of doping impurities into semiconductors. Unfortunately, the ion bombardment of a solid is associated with the radiation damage, which essentially influences electrical properties of the implanted layer. From the point of view of semiconductor technology these properties become worse by the radiation damage. Therefore, the effect of radiation damage on the electrical properties of semiconductors and their improvement after implantation are intensively studied. One of the main tools of radiation damage investigation in crystal lattices is the transmission electron microscopy (TEM), which in the case of implanted materials requires a special thin foil preparation technique.

2. PREPARATION OF THIN FOILS FOR TEM

The initial specimens were circular discs about 30 mm in dia and 100 μm in thickness of a silicon single crystal with a very low density of dislocations and with a specific resistance higher than 100 Ω cm. The discs were mechanically polished and etched in hydrochloric acid gas.

The silicon slabs were implanted at room temperature by N⁺ ions with an energy in a range from 30 to 50 keV and a dose of $1 \times 10^{13} - 2 \times 10^{16}$ ions/cm². Direction of incidence of ions was approximately perpendicular to the surfaces of specimens, which were parallel with crystallographic planes (111), (110) and (100).

2.1. Cutting of specimens

The implanted silicon slabs should be thinned from the side of unimplanted surface to the thickness lower then 5000 Å and small discs not exceeding 3 mm in dia suitable for the specimen holder in the electron microscope should be cut. Because of a very

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high brittleness of silicon single crystals it is not possible to cut the small discs after thinning. For this reason they were cut from the initial slabs before thinning by means of a crown drill and the whetting paste of SiC (grain diameter 50 μm). The question of generation of structure defects by cutting of small discs was answered by the X-ray diffraction topography: the Berg-Barret method in transmission indicated no structure defects except in a very narrow region (about 0.1 mm width) adjacent to the cut\(^1\). Later this conclusion was further supported by the TEM.

2.2. Chemical etching and anodic oxidation

In the case of implanted materials the necessity to conserve the implanted layer strongly limits the choice of preparation techniques of thin foils for TEM. The total path length of nitrogen ions with the initial energy of 30 keV in silicon is about 1300 Å, the projected range is about 40% shorter [1], i.e. the specimens can be thinned to thickness of about 2000 Å (from the unimplanted side) without a danger of disturbing the implanted layer.

The best way of thinning silicon seems to be chemical etching. With the aid of the experimental arrangement shown in Fig. 1 the thinning of silicon is simple, quick and effective. The etchant consists of nitric (60%) and hydrofluoric (48%) acids in the ratio of volumes HNO\(_3\) : HF = 9 : 2 or 9 : 1 [2]. The etchant flows from a reservoir through a tube provided by a regulator of flow into a jet (1 mm in dia) and falls onto the lower (unimplanted) side of the horizontally fixed specimen. Then it flows down along the outer side of the jet and is collected in a vessel. The vessel, the reservoir, the tube and the jet are made of polyethylene which resists the etchant. The etchant can

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