Materials Science Forum Vols. 740-742 (2013) pp 649-652
Online available since 2013/Jan/25 at www.scientific.net
© (2013) Trans Tech Publications, Switzerland
doi:10.4028/www.scientific.net/MSF.740-742.649

Origin Analyses of Obtuse Triangular Defects in 4deg.-off 4H-SiC Epitaxial Wafers by Electron Microscopy and by Synchrotron X-ray Topography

T. Yamashita[1,2,a]*, H. Matsuhata[1,3], Y. Miyasaka[1,2], H. Ohshima[1], M. Sekine[3], K. Momose[1,2], T. Sato[1,2,b], and M. Kitabatake[1]

[1]R&D Partnership for Future Power Electronics Technology (FUPET) , 2-9-5 Toranomon, Minato-ku,Tokyo 105-0001, Japan

[2]SHOWA DENKO K.K., 1-13-9, Shibadaimon, Minato-ku,Tokyo 105-8518, Japan

[3]Advanced Power Electronics Research Center, National Institute of Advanced Industrial Science and Technology, 1-1-1, Umezono, Tsukuba, Ibaragi 305-8568, Japan

[a]t-yamashita@fupet.or.jp,[b]Takayuki_Sato@sdk.co.jp

Keywords: Silicon carbide (SiC); Triangular-Defects; Synchrotron X-ray Topography; Electron Microscopy

Abstract. Triangular shaped defects with obtuse-angles at the vertex and long base are often observed in surfaces of epitaxial films. We have investigated the origins of them, and it became clear that these defects without well defined origins were formed by contaminations of tantalum carbide particles. Formations of micron-order pipes at the points of origin these defects were also observed. These micron-order pipes were not accompanied by strain and dislocations around them, though their appearances were very similar to the so-called micro-pipes.

Introduction

Current SiC wafers contain several types of defects on epitaxial film surfaces. Among them, triangular shaped defects with obtuse-angles at vertices and long bases are one of the very common defects particularly for the surfaces with off-cut by 4 degrees [1]. It was reported that this type of surface defects gave rise to negative and serious impact on the yield ratio and the reliability of MOS performance [2]. It is already known, that some of these defects are originated from the 3C particles so-called “down-falls”, carrot-like defects, surface scratches, and so on [1], [3]. However, the defects with such apparent origins were in the minority among the defects in our optical microscopy observations. No clear origins were observed for the majority of these obtuse-triangular defects. In order to improve the quality of epitaxial films, and the yield ratios as well as the reliability of MOS performances, we have investigated the origins for the majority of the defects with unclear origin.

Experimentals

Commercially available 4H-SiC wafers with epitaxial films and with off-cut surfaces by 4 degrees from the Si[0001] toward the [11$\bar{2}$0] direction were used. Thicknesses of epitaxial films were measured by Fourier-transform-infrared-spectroscopy. Confocal optical microscope was used to observe the defects on surfaces. Figure 1 shows the typical obtuse triangular defect observed by a confocal optical microscope. It had a long base toward the [$\bar{1}$100] direction of “mm” order in

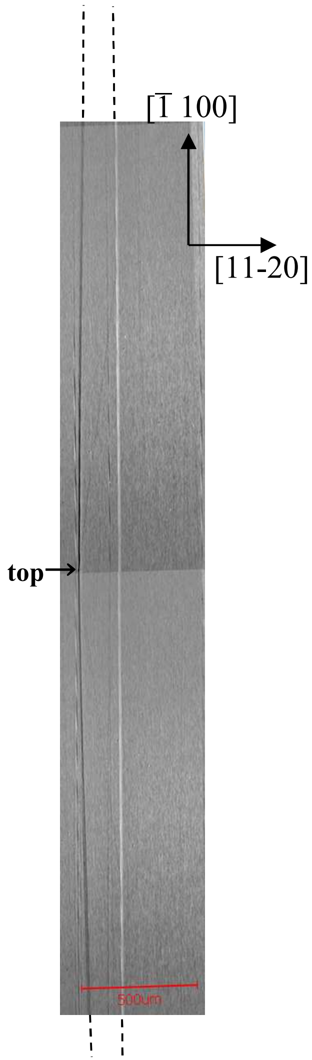

Fig.1 Confocal optical microscope image of an obtuse triangular defect.

All rights reserved. No part of contents of this paper may be reproduced or transmitted in any form or by any means without the written permission of TTP, www.ttp.net. (ID: 129.101.79.200, University of Idaho, Moscow, USA-13/02/15,00:05:41)

length, without characteristic origins at the obtuse-angled vertex, and no other complex defects inside of the defect. This type of defects were commonly observed on certain epitaxial film surfaces. The density of the defects was higher than that of other common defects such as acute triangular defects [4] by about 10 times, or more. We have chosen the defects with the same appearance in Fig. 1 for our work.

Experiments of grazing-incidence synchrotron reflection X-ray topography were carried out at the beam line 15 in Kyushu Synchrotron Light Research Center. The reflection used for the analysis were $\mathbf{g}=\bar{1}\,\bar{1}\,28$ with 0.15 nm for wavelength. Also, photo luminescence (PL) imaging instruments was used to investigate the presence of lattice defects. Scanning electron microscope(SEM) observations were carried out to investigate the surface morphologies at the top parts of obtuse triangular defects. Using a focused ion beam equipment (FIB), specimens for cross-sectional observations were prepared at around the obtuse-angled vertex. Microstructures in cross-sectional specimens were observed using SEM and transmission electron microscope (TEM).

Results and Discussions

No contrasts of stacking-faults and dislocations at corresponding positions of those defects were observed in the PL and X-ray topographic images. These results indicate that the defects of triangular shape with obtuse angles themselves did not consist of lattice defects such as stacking-faults and dislocations, but consisted of surface steps and surface morphologies. Fig. 2 shows SEM images of the vertices, where the triangular surface defects are generated with obtuse angles. Formations of pits with diameter approximately ~5μm at top parts were often observed. Bending, wavy and step-like vertical fine lines in Fig. 2's were observed at surfaces. Pits had three types of shapes;

Type-I: Pits with holes, which continue to deeper part of the epitaxial films, diameter of ~1μm.
Type-II: Pits with hexagonal-shaped and shallow closed bottoms.
Type-III: Pits without clear outlines, or dimples.

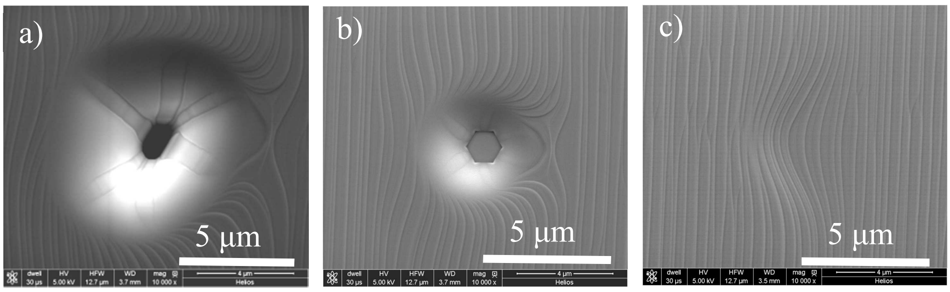

Fig.2 SEM Images of the top of obtuse triangular defects.
a): type-I, b): type-II, c): type-III.
*vertical lines in each figure correspond to bunching of surface steps.

In cross-sectional SEM observations, foreign materials that showed different contrast in SEM images were observed in all three types as shown in Fig.3. The sizes of the foreign materials were of the order of several microns. Their sizes had the following tendency: type-I > type-II >type-III. Depths of these foreign materials are approximately 11~12.4 microns, which are the same or shallower than the epitaxial film/substrate interface position; 12.4 microns in this case. These foreign materials of TYPE-I~TYPE-III were thought to drop from a CVD furnace emvironment at the initial stage of the epitaxial film growth process and thought to be the origins of the surface defects.

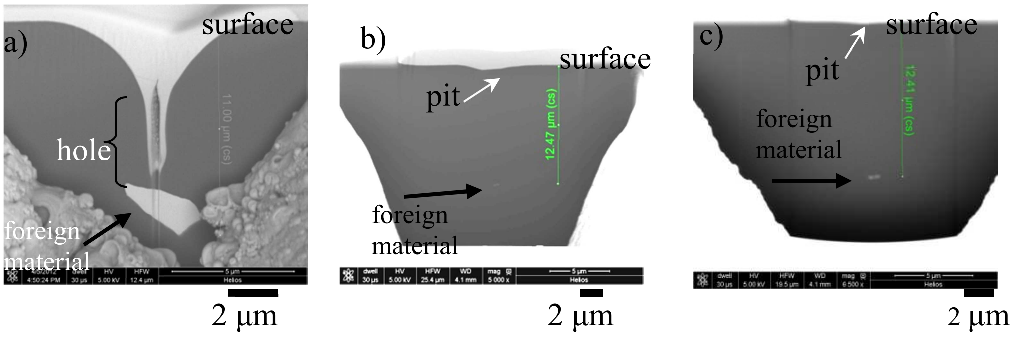

Fig.3 Cross-sectional SEM Images of the vertice of obtuse triangular defects.
a): type-I, b): type-II, c): type-III.

By energy dispersive X-ray spectrometry (EDX) analyses, tantalum was detected as a foreign material of TYPE-I~TYPE-III. These results indicated that the foreign materials are from some components in CVD furnace.

In case of Type-I, the holes penetrate from deep parts to the surfaces of the epitaxial films. The holes were originated from foreign materials as seen in cross-sectional SEM image.

As indicated in Fig.4, X-ray topographic image at the vertex of obtuse triangular defects of Type-I showed a dark and small spot. The contrast is very different from that of the so-called micro-pipes that show large bright spots caused by strong long-range strain around them [5], though the appearances of the micro-pipes in SEM images are very similar to those in Fig. 2's.

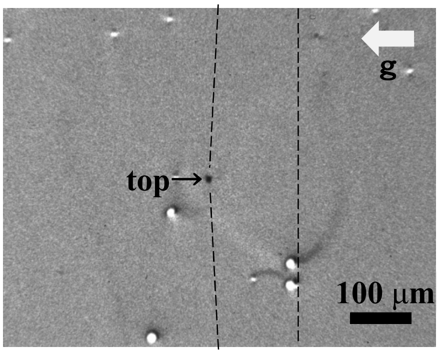

Fig.4 X-ray topography of the vertex of the obtuse triangular defect with a hole. Direction of the reciprocal vector **g**=$\bar{1}\,\bar{1}\,28$ was indicated. Dot lines correspond to outlines of this obtuse triangle defect.

Cross-sectional TEM images of a type-I pit showed the holes originated from the foreign material as seen in Fig. 5's. Neither dislocations, nor stacking-faults could be seen in 4H-SiC epitaxial film around the hole and around the foreign material under the two different diffraction conditions. These results were different from the case of defects formed by 3C particles, which get attached to wafer surfaces in CVD furnaces during the epitaxial film growth. In case of 3C particles, basal-plane dislocations, threading screw dislocations, 8H stacking-faults, 3C-layers were formed [3]. Free energies for interfaces between 3C-SiC particles and 4H-SiC epitaxial films will be small, and so that close contacts between two different crystals take places. The formations of dislocations and stacking-faults will occur at the interfaces between them even with random orientations. On the other hand, TaC is known to be a very stable material, and free energy at the interface between 4H-SiC and TaC will be high, so that 4H-SiC epitaxial film may grow without forming close contact interface. This may reduce strain at the interface, and reduce the formations of lattice defects in 4H-SiC around the interfaces.

TaC's particles in epitaxial films were observed as fragments with several micron meters in size. This may indicate that the fragments are formed by detachments induced by thermal stress at surfaces of TaC materials in CVD furnaces, but not by sublimations.

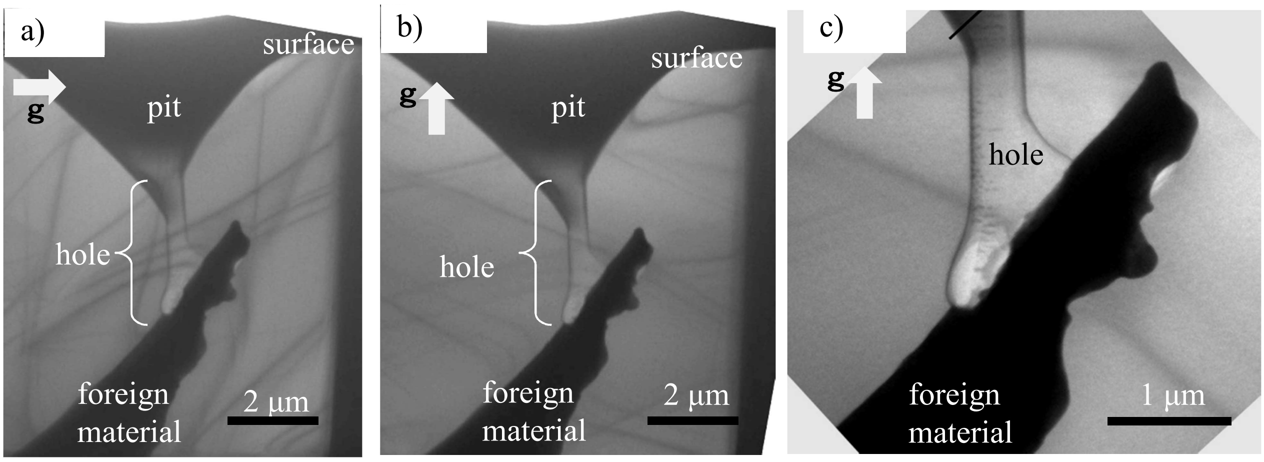

Fig.5 Cross-sectional TEM images of the top part of an obtuse triangular defect with a hole around the 4H-SiC [$\bar{1}$ 100] zone axis observations. Directions of **g** are inserted in the images.
a): Image taken under **g**=11$\bar{2}$ 0 condition. b): Image taken under **g**=0004 condition.
c): Enlarged image of fig. b).

Summaries

Triangular shaped defect with the obtuse-angle at the vertex is one of the most common defects observed in wafers obtained commercially. The origin for the majority of this type defect was unkown. By the present experiment, it became clear that majority of this type of defects were originated from foreign materials, tantalum carbide in this case. The results were different from the one originated from 3C particles [3]. In the case of 3C particles, many lattice defects were introduced in the epitaxial films. On the other hand, in TaC particles, the formations of lattice defects are restrained. The observed micron-order pipes were not accompanied by strain and dislocations, thus these were different type of defects from the so-called micro-pipes. The present observed results indicated many micron-order pipes were formed during epitaxial film growth by foreign materials contamination. Protection from the TaC particles will improve quality of epitaxial films, and as well as the yield ratios, and the reliability of MOS.

Acknowledgments

High Voltage Electron Microscope Laboratory, Nagoya University Ecotopia Institute is acknowledged. This work was performed with the approval by the Kyushu Synchrotron Light Research Center (Proposal No. 1105051AS) . This work is supported by Novel Semiconductor Power Electronics Project Realizing Low Carbon Emission Society by METI and NEDO in Japan.

References

[1]D. Muto, T. Yamashita, K. Yamatake, Y. Miyasaka, K. Momose, and T. Sato, Proceedings of the 19th SiC and Related Wide Bandgap Semiconductors Seminar (2012)89-90.

[2] J. Sameshima, O. Ishiyama, A. Shimozato, K. Tamura, H. Ohshima, T. Yamashita, T. Tanaka, N. Sugiyama, J. Senzaki, H. Matsuhata, and M. Kitabatake, Abstract of the European Conference on Silicon Carbide and Related Materials 2012.

[3]B. Chen, H. Matsuhata, T. Sekiguchi, K. Ichinoseki, and H. Okumura, Acta Mater. 60 (2012) 51-58.

[4]T. Yamashita, K. Momose, D. Muto, Y. Shimodaira, K. Yamatake, Y. Miyasaka, T. Sato, H. Matsuhata, and M. Kitabatake, Mater. Sci. Forum Vols. 717-720(2012)363-366.

[5] X. R. Huang, M. Dudley, W.M. Vetter, W. Huang, S. Wang and C.H. Carter, Appl. Phys. Lett 74, 353(1999).

Silicon Carbide and Related Materials 2012
10.4028/www.scientific.net/MSF.740-742

Origin Analyses of Obtuse Triangular Defects in 4deg.-Off 4H-SiC Epitaxial Wafers by Electron Microscopy and by Synchrotron X-Ray Topography
10.4028/www.scientific.net/MSF.740-742.649