SUBJECT: INTRODUCTION OF THE S-4300SE SEM WITH FEATURES AND SOME APPLICATIONS

INSTRUMENT: THE S-4300SE SCHOTTKY-EMISSION SCANNING ELECTRON MICROSCOPE

CONTENTS

1. INTRODUCTION

New analytical techniques such as cathodoluminescence (CL) and electron backscattered diffraction pattern (EBSP) have been brought into scanning electron microscopy (SEM) in addition to high resolution observations. The S-4300SE has been developed in view of these new analytical applications. Fig. 1 shows a general view of the S-4300SE. It allows simple addition of CL, EBSP and EDX detectors for a variety of applications. We report on features of the S-4300SE and some applications using the CL as well as EBSP systems.

2. FEATURES OF THE S-4300SE

(1) Large probe current available

The S-4300SE allows a probe current of 25 nA (maximum) at 20 kV. It is useful for analytical and other applications that require a large probe current.

Fig. 1 A general view of the S-4300SE

(2) Stable probe current supply

The S-4300SE allows not only a large probe current but also allows a stable probe current supply (fluctuation less than 2% for 8 hours) which is useful for EBSP, automated EDX work and other applications that require extra long hours of operation.

(3) High resolution imaging at a large probe current operation

A fine probe is available at a large probe current operation. It is useful for analysis of sub-micron areas. Fig. 2 is a graph showing a relation between the probe current and the probe diameter for typical electron sources including Schottky Emission (SE), Cold Field Emission (FE), heated tungsten source (W), and heated LaB_6 source. It tells us that SE is useful for applications that require a probe current of 1 nA ~ 20 nA and a probe diameter of about 10 nm. Fig. 3 shows a typical high resolution secondary electron image recorded at 15 kV and a probe current of 5 nA.

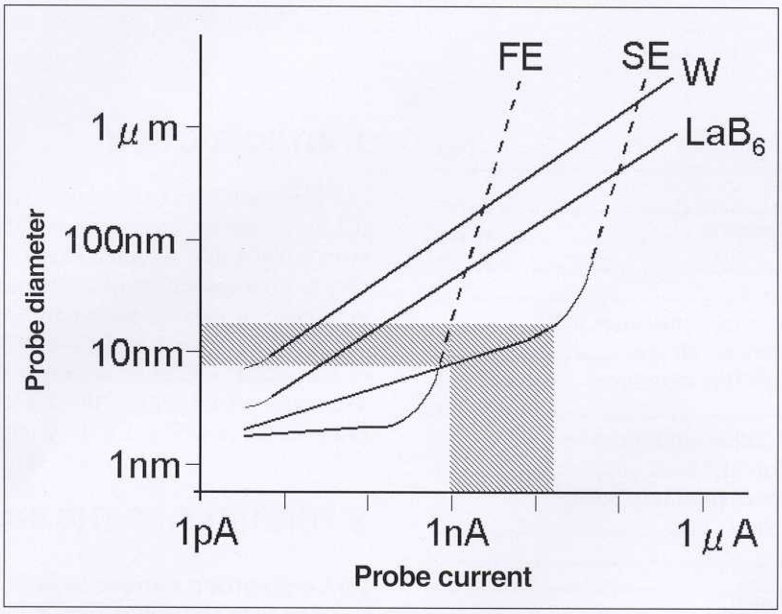


Fig. 2 A relation between probe current and probe diameter for various electron sosurces

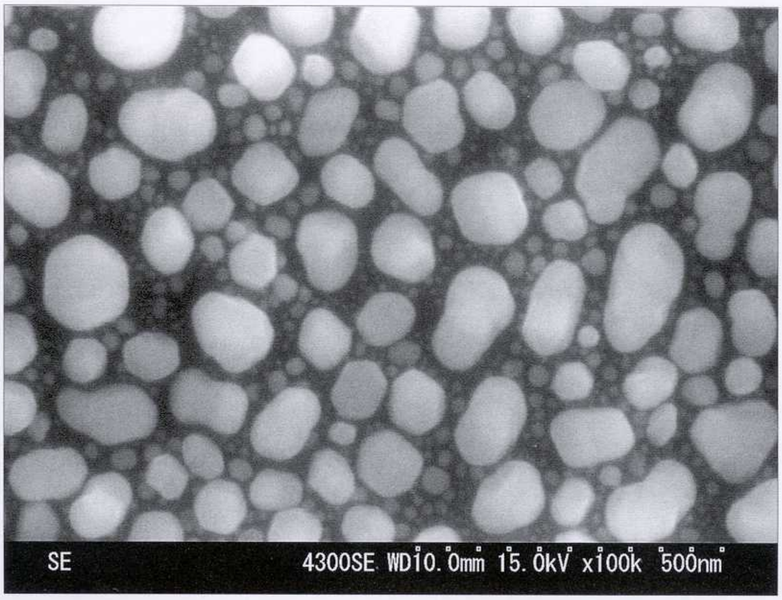


Fig. 3 A high resolution image recorded at a probe current of 5 nA
Accelerating voltage: 15 kV
Probe current: 5 nA

3. APPLICATIONS

3.1 Cathodoluminescence (CL)

When a primary beam of electrons strikes the specimen, a photon emission, which is called cathodoluminescence, takes place. These photons are collected and analyzed for studying photo devices, impurities in semiconductors, and structural defects of materials. The S-4300SE and Hitachi's CL attachment allow high resolution CL imaging for analysis of fine details even at low operating voltages.

3.1.1 CL imaging of InGaN multi-quantum well structure at low operating voltages

For CL imaging of fine details, low operating voltages are required for suppressing diffusions of carrier generation areas. High resolution imaging capabilities and sufficient probe current supplies for a good S/N ratio are also required. Fig. 4 is a schematic diagram showing a multi-quantum well structure of InGaN in GaN laser diode which has been used as a laser light source for an optical recorder. Figs. 5.1 and 5.2 show CL images recorded at 4 kV and 20 kV at a probe current of 0.1 nA and at a wavelength of 425 nm. The carrier generation area looks significantly smaller at 4 kV than 20 kV.

3.1.2 GaN fine structures

Fig. 6.1 shows an SEM image of GaN grown on ZnO/Si substrate. Fig. 6.2 shows an SEM image of a cross-section of the same. Fig. 6.3 is a CL image recorded at a wavelength of 365 nm. The CL image was recorded at 4 kV and a probe current of 0.2 nA. Despite the small probe current, GaN's pillar structures of 50 ~ 100 nm are clearly visible.

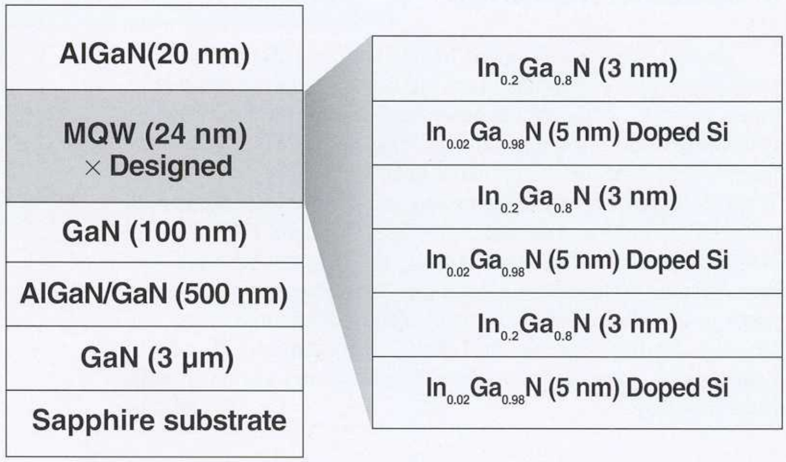


Fig. 4 A schematic diagram of InGaN multi-quantum well structure

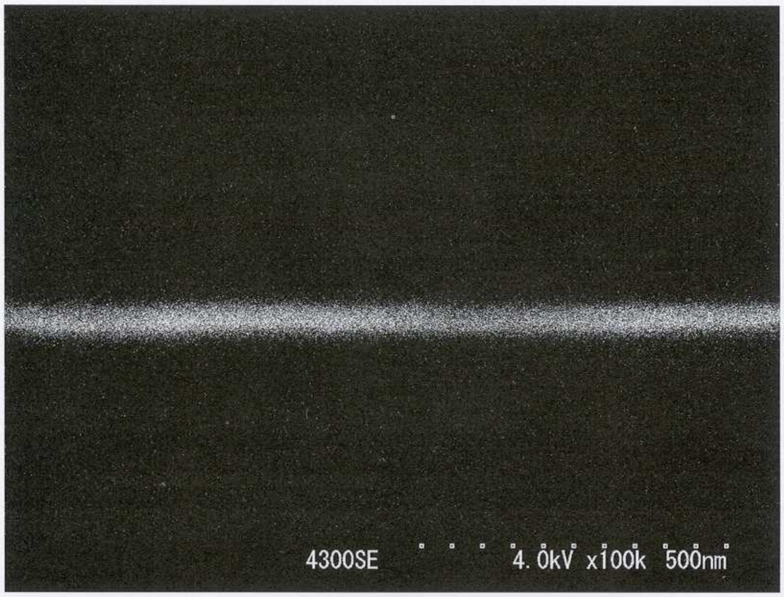


Fig. 5.1 CL image (425 nm) of InGaN multi-quantum well structure
Accelerating voltage: 4 kV, Probe current: 0.1 nA

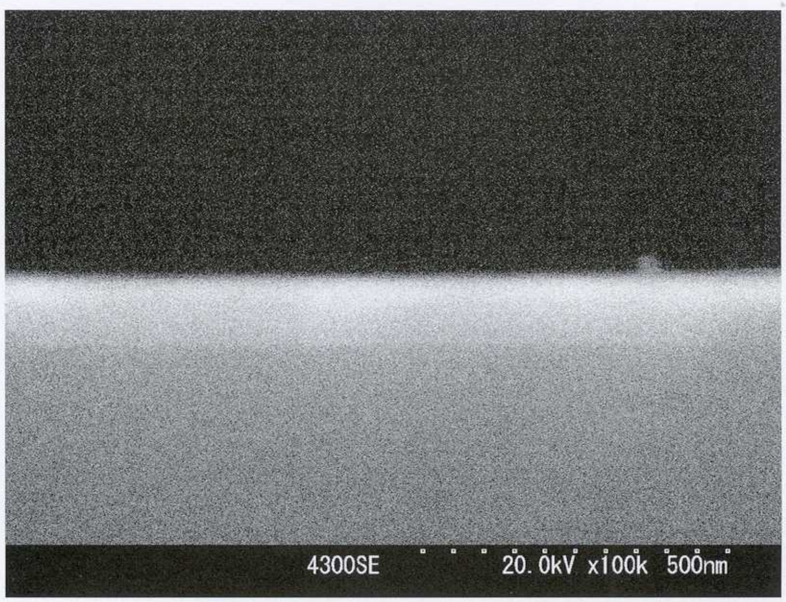


Fig. 5.2 CL image (425 nm) of InGaN multi-quantum well structure
Accelerating voltage: 20 kV, Probe current: 0.1 nA

3.2 EBSP applications

When the primary electron beam strikes a crystalline specimen at angles of 60°~ 80° with respect to the beam, electrons are scattered by diffraction due to crystal orientations. When these electrons are displayed on a fluorescent screen, an electron diffraction pattern which is characteristic of crystal structures is available. The pattern is immediately indexed by computer processing. This technique allows examination of orientations of crystalline materials and helps in studying grain boundaries. The S-4300SE allows fine probes of a few nm with a probe current of 5 nA so that it is useful for studying fine crystal particles and grains.

3.2.1 EBSP imaging of rolled aluminum using various electron sources

EBSP is available by illuminating crystal grains of interest with a fine probe of electrons. The smaller the grains, the finer the probe and the larger the probe current required. It also requires a stable probe current since the imaging may take a long time depending on areas of interest. Fig. 7 shows typical EBSPs recorded by using a LaB_6 SEM and the S-4300SE. The S-4300SE image clearly shows fine crystal grains much better than the LaB_6 SEM's. It is simply because the S-4300SE allows better imaging resolution at a large probe current operation. The EBSP images also show a fairly good stability of the probe current for 8 hour operation.

3.2.2 Crystal orientation analysis of iron/steel transformation structure

Fine iron/steel transformation structures increase toughness. The EBSP allows fine phase distributions and their orientations of these materials. Fig. 8 shows colored orientations and phase separations of austenite (fcc) and ferrite (bcc). Clear phase separation and orientation images are available at 25 kV and a probe current of 2 nA and submicron order crystal grains of austenite are also visible as shown by arrows.

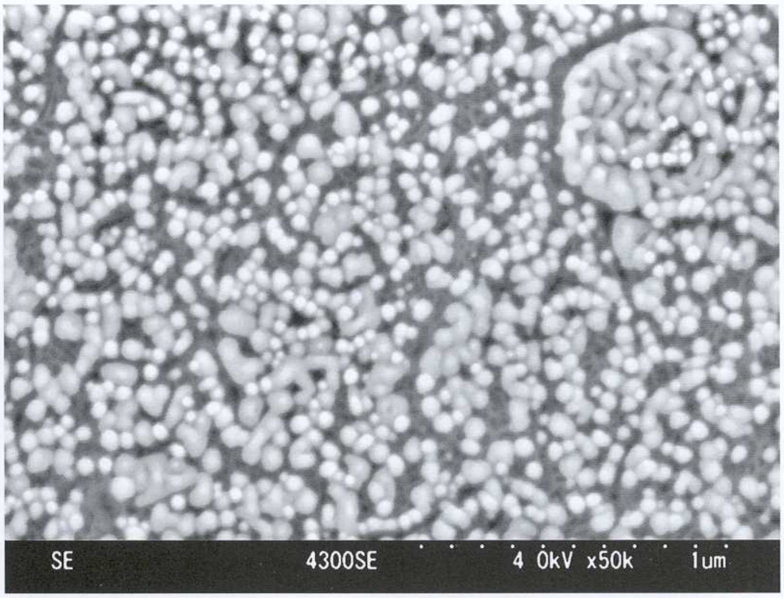


Fig. 6.1 SEM image of GaN on ZnO/Si substrate

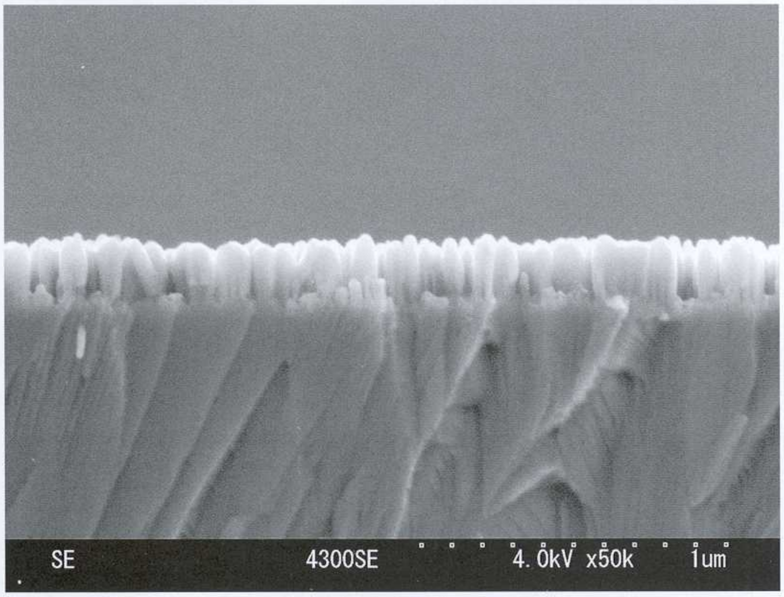


Fig. 6.2 SEM image (cross-sectional view) of GaN on ZnO/Si

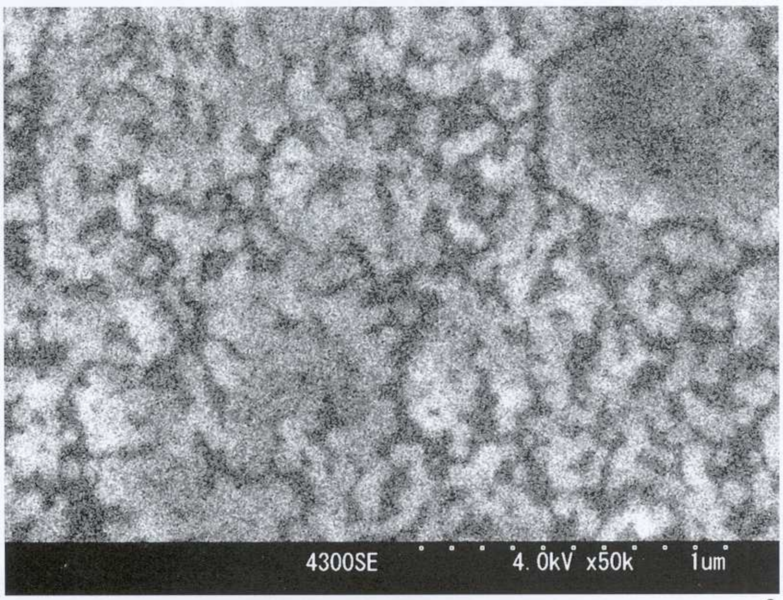


Fig. 6.3 CL image (365 nm) of GaN on ZnO/Si
Accelerating voltage: 4 kV
Probe current: 0.2 nA
Specimen, courtesy of Prof. Yasushi Nanishi and Dr. Tsutomu Araki, Department of Photonics, Faculty of Science and Engineering, Ritsumeikan University, Japan

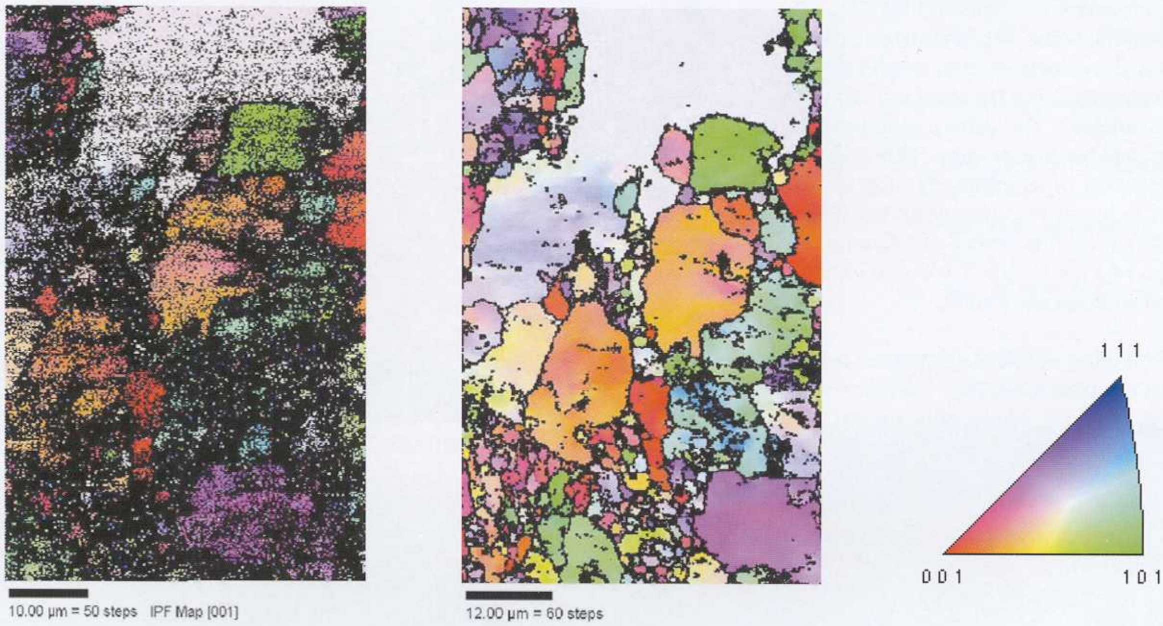
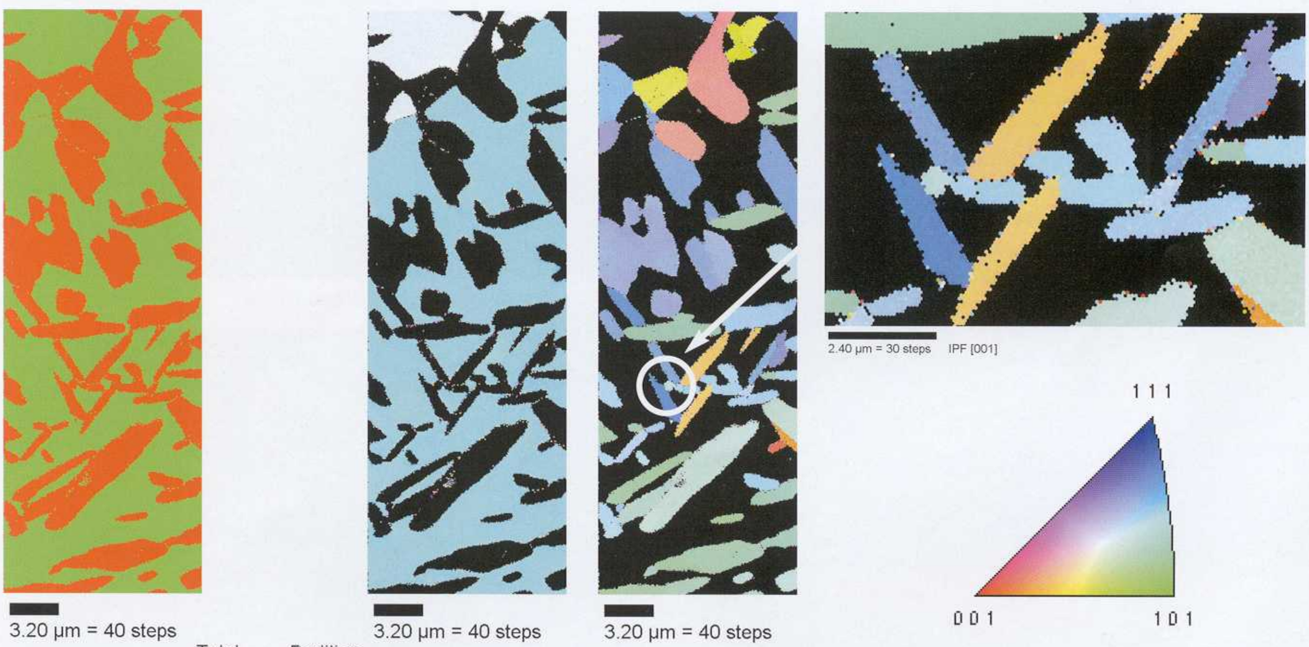


Fig. 7 EBSP analysis of rolled aluminum

Accelerating voltage: 15 kV, Probe current: 1.5 nA
Analytical area: 45 µm × 65 µm, Acquisition time: 8 hours



Phase	Total Fraction	Partition Fraction
fcc_generic	0.417	0.417
bcc_generic	0.583	0.583

Phase separation image

Orientation image of bcc

Orientation image of fcc

Fig. 8 Phase separation and orientation images of austenite (fcc) and ferrite (bcc)

Accelerating voltage: 25 kV, Probe current: 2 nA
Analytical area: 15 µm × 40 µm, Acquisition time: 1 hour

4. CLOSING REMARKS

Heated tungsten filament SEMs and/or LaB_6 SEMs have been primarily used for analytical applications that require a large probe current. These SEMs have been limited in their imaging resolutions and analytical areas. The S-4300SE allows sub-micron area analyses which have been difficult up to this time. We trust that it will be useful for various fields of research as well as quality control in industries. We wish to thank Prof. Yasushi Nanishi and Dr. Tsutomu Araki, Faculty of Science and Engineering, Ritsumeikan University, Japan for providing specimens and technical assistance on nitride semiconductors, and Messrs. Seiichi Suzuki and Shinichiro Adachi, TexSEM Laboratories, Japan for providing specimens and technical assistance on EBSP.

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