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Scanning Probe Microscope/Atomic Force Microscope

Application News

Visualizing Local Resistance Distributions in an n-Type Si Sample and a Liquid Lithium-Ion Battery Positive Electrode Using SSRM

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User Benefits

- Current distribution (1 pA to 10 mA) and resistance distribution (0.1 Ω to 10 TΩ) in electronic materials such as semiconductors and batteries can be detected over a wide range.
- Carrier concentration distributions in semiconductor devices can be estimated from resistance distributions.
- Distributions of active material and conductive additives, as well as electron conduction paths, in lithium-ion battery electrodes can be estimated from resistance distributions.

■ Introduction

In semiconductor devices and liquid lithium-ion batteries (LIBs), the microscopic distribution of components affects device and battery characteristics, so understanding the resistance distribution inside the device at the nanoscale is important. Scanning spreading resistance microscopy (SSRM), a scanning probe microscopy (SPM) technique performed using an atomic force microscope (AFM), maps the resistance distribution in microscopic areas of a sample, making it effective for estimating the dopant concentration in semiconductor devices and visualizing electron conduction paths inside LIBs. This Application News introduces examples of evaluating an n-type Si sample and a liquid LIB positive electrode using SSRM.

■ Measurement Instrument

An SPM-9700HT™ Plus was used for the measurements (Fig. 1). An SPM scans the sample surface with a fine probe to measure the three-dimensional topography and local physical properties of the sample at high magnification. In SSRM, a bias voltage is applied to the sample while a conductive probe scans its surface, as shown in Fig. 2. By measuring the current flowing between the probe and the sample with a logarithmic amplifier, the current and resistance distributions can be visualized over a wider range than is possible in conventional current mode.



Fig. 1 SPM-9700HT Plus

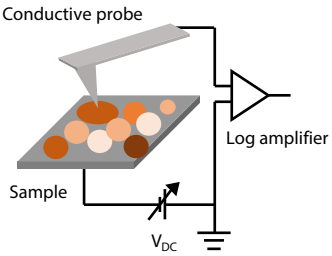


Fig. 2 Schematic Diagram of SSRM

■ Samples and Measurement Conditions

SSRM Measurement of n-Type Si Sample

The n-type Si sample has a stepwise doping concentration profile extending from the surface into the bulk. As pretreatment, the sample was cross-sectioned as shown in Fig. 3, and conduction between the sample holder and the sample was ensured with conductive paint. Table 1 shows the measurement conditions.

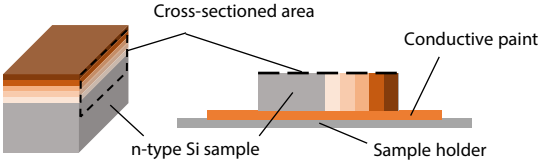


Fig. 3 Schematic Diagram of n-Type Si Sample

Table 1 Measurement Conditions

Instrument:	Scanning Probe Microscope SPM-9700HT Plus
Scanner:	Wide Range scanner (XY: 125 µm, Z: 7 µm)
Mode:	Spreading-Resistance (SSRM) mode
Field of View:	30 µm × 10 µm
Applied Voltage:	+6.0 V

SSRM Measurement of Liquid LIB Positive Electrode

The liquid LIB positive electrode consists of a positive electrode active material (ternary NCM) and a conductive additive. As pretreatment, the sample was embedded in resin (Fig. 4), and the measurement surface was mechanically polished. Electrical contact between the sample holder and the observation surface was established using carbon tape. Table 2 shows the measurement conditions.

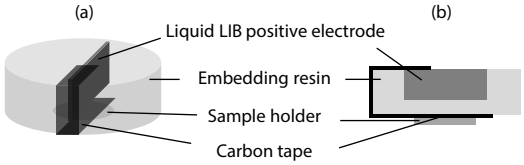


Fig. 4 Schematic Diagram of Liquid LIB Positive Electrode
(a) Overall View, (b) Side View

Table 2 Measurement Conditions

Instrument:	Scanning Probe Microscope SPM-9700HT Plus
Scanner:	Wide Range scanner (XY: 125 µm, Z: 7 µm)
Mode:	Spreading-Resistance (SSRM) mode
Field of View:	30 µm × 30 µm
Applied Voltage:	+10.0 V

■ SSRM Measurement of n-Type Si Sample

Fig. 5 shows the SSRM resistance image of the n-type Si sample, and Table 3 shows the in-plane median resistance and carrier concentration in regions L1 to L5. The resistance display range is 10^3 to 10^{12} Ω. Resistance increases stepwise from right to left across the field of view, corresponding to changes in carrier concentration.

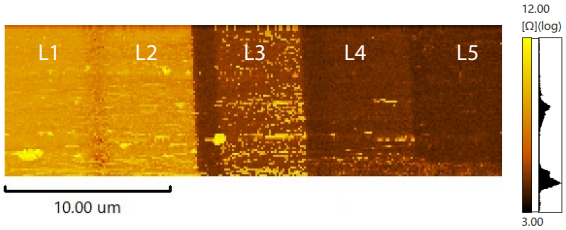


Fig. 5 SSRM Measurement Result of n-Type Si Sample: Resistance Image

Table 3 Resistance and Carrier Concentration in Each Region

	L1	L2	L3	L4	L5
In-plane median resistance [Ω]	2.95 ×10^8	1.66 ×10^8	1.26 ×10^5	4.26 ×10^4	2.63 ×10^4
Carrier concentration [atoms/cm^3] 1)	5.0 ×10^{14}	5.0 ×10^{15}	1.0 ×10^{17}	1.0 ×10^{18}	2.0 ×10^{19}

■ SSRM Measurement of Liquid LIB Positive Electrode

Fig. 6 shows the SSRM measurement results of the liquid LIB positive electrode. The display ranges are 10^{-4} to 10^{-10} A for current and 10^{5} to 10^{11} Ω for resistance. In the topographic image (a), the fine particle shapes of the active material and its surroundings are observed. In the current image (b) and resistance image (c), the current and resistance values differ among active material particles. Focusing on active material particles (1) to (3) outlined in white, the median resistance values are (1) 4.57×10^{8} Ω, (2) 3.89×10^{9} Ω, and (3) 1.26×10^{10} Ω. The high-resistance particle (3) may be electrically isolated by cracks inside the particle or by gaps between the particle and its surroundings. Uneven resistance is also observed within the same particle. Furthermore, in the resistance image, the black regions of particularly low resistance are thought to be the conductive additive, while the bright yellow regions of particularly high resistance are thought to be the embedding resin. In the current image (d), displayed over a range equivalent to the conventional current-mode detection range, the current saturates in both the conductive additive and the active material, making it impossible to distinguish conductivity differences. SSRM effectively detects small current changes within large current variations, as shown by this sample.

■ Conclusion

SSRM measurements enabled the visualization of local resistance distributions in an n-type Si sample and a liquid LIB positive electrode. Because SSRM provides current and resistance distribution information over a wide range, something not achievable with conventional current measurements, the method is expected to be further used for improving the performance of semiconductors and batteries.

Acknowledgement

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References

1) N. Duhayon et al., “Assessing the performance of two-dimensional dopant profiling techniques”, JVSTB 22, 385 (2004).

Related Applications

1. Analysis of Positive Electrode (NCM) for Lithium Ion Battery by EPMA and SPM, Application News No. P117

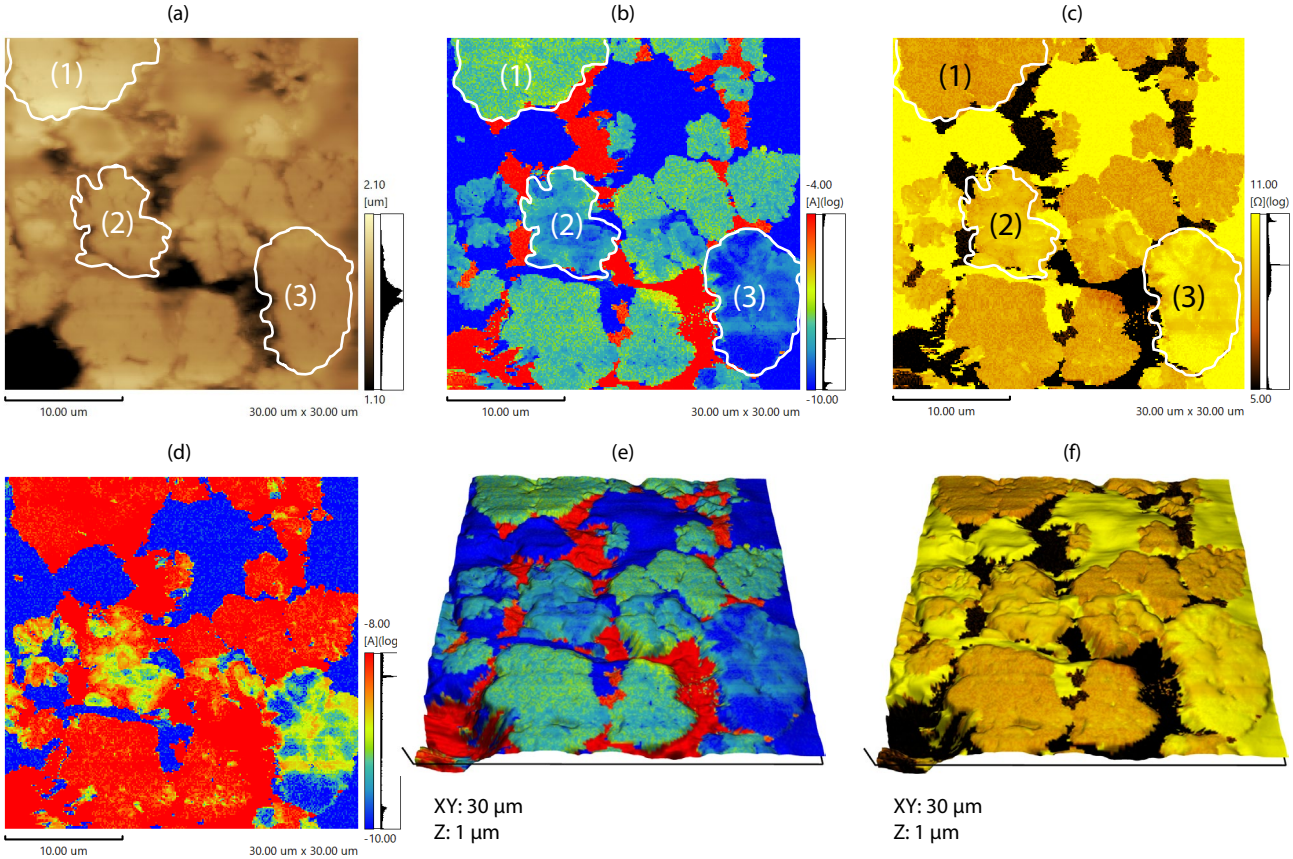


Fig. 6 SSRM Measurement Results of Liquid LIB Positive Electrode
(a) Topographic image, (b) Current image, (c) Resistance image,
(d) Current image displayed in a range equivalent to the current detection range of the conventional current mode
(e) 3D overlay of topographic and current images, (f) 3D overlay of topographic and resistance images

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