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Paper

Industrial standardization and quantification of the carrier concentration in semiconductor devices using electric SPM

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Scanning capacitance microscopy (SCM) is one of techniques imaging two-dimensional carrier distribution in semiconductor devices. We have demonstrated that quantitative analysis of carrier concentration can be performed using calibration curves, which are statistically prepared for respective materials like silicon, silicon carbide, etc. The procedure was circulated to research organizations in domestic round robin test, and this method was found to be available from the viewpoint of comparability among users for the industrial standardization. It was also applied to other microscopes, and their profiles of carrier concentration coincide with each other, suggesting compatible to next-generation microscopes which would come up in the future.

1. Historical overview of SPM development and standardization

Scanning probe microscopy (SPM) is a collective term and consists of a family of microscopes and related techniques using a probe with a sharp tip that can image a three dimensional surface topography and analyze a surface property of specimens. Historically, it originated in the innovation of scanning tunneling microscopy (STM) in 1981 by Gerd Binnig and Heinrich Rohrer at IBM Zurich Research Laboratory [1], enabling us to research surface physics and chemistry with atomically resolution. Succeeding STM, the next most important developments was atomic force microscopy (AFM) applicable to either insulator or conductor [2]. Our company Toray Research Center Inc. has started a business of research and analysis service using AFM (ultra-high vacuum AFM system made by Electrotechnical laboratory, AIST) since 1989.

In the 1990's, a variety of the SPM techniques was developed and the first commercially launched from

Digital Instruments Inc. (emerged with Bruker AXS, then followed by Veeco instruments). Using this commercial AFM, we reported the first study of an evaluation of oxygen-ion scattered craters with ripping structure affecting the yields of secondary ion on a Gallium Arsenide (GaAs) surface [3]. Around the 2000 year, the SPM market was grown and increasing. The operation modes were designed for direct observation and manipulation of biological molecules, mechanical evaluation of polymers, characterization of magnetic materials in data storage, electric testing and failure analysis of semiconductor devices, etc. Over the thirty years, we have learned the applications had changed from fundamental science in vacuum to practical analysis in air or fluid. Nowadays, SPM has been used as not only general purpose tools in laboratories but also inspection tools equipped with automation system in which robots load or unload the probes and samples in industrial fabrication.

Metrology universally demands quantification and

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standardization in the top-down approach, without an exception of SPM, as it progressively enters the mature phase. According to technical roadmap of SPM standardization, the global pre-standardization research was initiated by committee TWA29 of Versailles Project on Advanced Materials and Standards (VAMAS) in 2002. As for international organization for standardization (ISO), TC201 (surface chemical analysis) established the sub committee SC9 for SPM in 2004. In Japan, our stan-(JSCA, dardization activities concerning SPM SPM-WG) have contributed to a publication of International Standard ISO 28600 on standard data transfer format for SPM in 2011 [4], drafts of IS (DIS), new work item proposals (NWIP) and technical reports (TR) for related and further advanced probe technologies.

2. Scope of carrier concentration profiling in semiconductor devices using electric SPM

In semiconductor application, one of the most important matters is evaluation of carrier concentration distribution in real devices like a metal-oxide-semiconductor field-effect transistor (MOSFET), where both high detection sensitivity and spatial resolution are needed. Scanning capacitance microscopy (SCM) and scanning spreading resistance microscopy (SSRM) were based on contact mode AFM techniques imaging electric properties in semiconductor devices, such as the free carrier distribution [5]. These two microscopes had been expected as the above-mentioned analytical solution in the earlier 2000's, but no more they could be applicable to the latest scaling down MOSFET in large scale integration (LSI). During these ten years, in our great deal of practical experiences of analysis service, we concluded that SCM has been suitable for the carrier concentration estimation and SSRM for high resolution imaging, in a mutually complementary manner. Actually, SCM is widely used at users' sites and has enough reproducibility of the signal corresponding to the carrier concentration.

3. Procedure of statistical estimation of carrier concentration from SCM signal

A UHF capacitance sensor from RCA technology of the 1970's, which is equipped with SCM, can detect a small movement of elementary charges of less than 10^{-19} F, though the sensor cannot count the number of the

charges. Therefore, the characteristic of sensor can be easily changed by the condition of the surface and probe, making it difficult to control output signals. In principle, SCM using this sensor can feel amplitude modulation (AM) or frequency modulation (FM) shift of a resonance oscillation of the transmitted Giga Hertz-band wave, mixed with a small ac voltage of dV (about 100 kHz) applied to the semiconductor sample. When a metal coated conductive probe touched on the surface, a local MOS capacitor is formed beneath the thin oxide, as shown in figure 1. In this area, an accumulation and depletion of the volume change induced by the dV can be detected as a signal corresponding to the capacitance change of dC through lock-in-amplifier. The interpretation of the SCM's resonance shift signal, which has been called as 'dC/dV', qualitatively follows a conventional method measuring high frequency capacitance verses voltage (C-V) relationship, though the sensor does not directly detect the capacitance but the change of the frequency, that is a reason of the lack of repeatability.



Fig. 1 Schematic of SCM. AC bias voltage oscillates a local MOS capacitor beneath the probe



Fig. 2 Correlation between carrier concentration and normalized dC/dV signals.

In such a background, we tried to examine and found a kind of correlations between concentration and dC/dV

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signals as shown in figure 2, since there were no traceable ways except for using amount of well-known samples like dopant implanted wafers, as well as real devices. At last, the tendency of the sensor's characteristic has been resulted from thousands of data which contribute to decreasing errors. In our method, not absolute values of the signal but a sequence of functions is necessary for the estimation, in which logarithm values of carrier concentration N in the horizontal axis were calculated from dC/dV signals in the vertical axis. Figure 3 shows an initiation procedure of plotting data and drawing the calibration curve in the case of the reference sample of silicon carbide (4H-SiC) epitaxial wafers. The vertical axis of the dC/dV signal was normalized the maximum to 1. By calculating inversely, in-plane distribution of the concentration with less than 20 % was also revealed in the profile of figure 4.

Figure 5 shows one of the results of practical analysis of real devices. Each implanted diffusion layers were observed in the dC/dV image on the cross-section of a power MOSFET [6]. In depth profiles of figure 5 (b), it is possible to quantitatively compare with the peak values. Similarly, this method has been applied to other materials like indium phosphate (InP) laser diodes for optical communication, copper indium gallium selenide (CIGS) thin layers for photovoltaic cell and *etc*.



Fig. 3 Calibration curve for 4H-SiC sample plotted by using carrier concentration of epitaxial wafers.



Fig. 4 Distribution of the carrier concentration in the 4H-SiC epitaxial wafer. (a) dC/dV image of the surface and (b) a lateral profile of the carrier concentration along the white line in Fig. 4(a).



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Fig. 5 SCM observation of a power MOSFET. (a) dC/dV image of the cross-section and (b) depth profiles of the statistically estimated carrier concentration.

4. Round robin test of estimation method for carrier concentration using SCM, SNDM, *etc*

The increasing popularity of SPM has given some kinds of effects for driving needs in a daily use and proposing industrial standardization of available techniques, that is the bottom-up approach in contrast of metrology standardization. In this case, round robin test is a preferred way to circulate the samples or recipes of the measurements among laboratories in the world wide for a short period, since it is possible to collect data for the proofs of standardization instead of traceable way.

User-to-user compatibility with SCM data was examined in domestic prior to global circulating. We used IMEC's epitaxial wafers with stair-structure profile which were widely used for the reference sample of dopant and carrier concentration for conventional spreading resistance measurement techniques [7]. They made pile-up structures in the boundaries as buffer layers for avoiding dopant diffusion. We also prepared AIST's dopant implanted wafers with continuous dynamic range for the carrier concentration evaluation, of which depth structure is similar to practical devices for both dopant and carrier concentration evaluation. Figure 6 shows results of the IMEC's sample. Apparent difference in images was observed, but the carrier profiles calculated independently using the users' calibration curves were almost coincident each other except for a couple of missed structures. Since participants of the round robin test do not have enough times for statistical estimations, the discrepancy of the carrier concentration is not ignorable as now but the accuracy would be improved by revising their calibration curve steadily.

Electric SPM techniques would progress year by year, while the computing technology was updated together. It may be time to think we discuss the substitution of next generation microscopies for SCM. In our previous study, it has been demonstrated that both profiles with the concentration estimated from different signals such as the dC/dV of SCM and spreading resistance R of SSRM were coincident under the same condition of the sample [8]. Finally, we introduce a state-of-the-art scanning nonlinear dielectric microscopy providing with possibility of atomic resolution [9], which is one of the most likely substituting technologies. Figure 7 shows a depth profile of the IMEC's sample obtained by a commercial SNDM launched by SII Nanotechnology Inc. The carrier concentration was calculated by the same way as SCM, and good fitting with each other was also confirmed.



Fig. 6 Round robin test for user-to-user compatibility with SCM data of IMEC's epitaxial wafer. (a) dC/dV images and (b) depth profiles at users' sites

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Fig. 7 Depth profile of the statistically estimated carrier concentration from SNDM data of IMEC's epitaxial wafer.

5. Conclusions and future plans

Statistical method using calibration curve is found to be effective for quantitative analysis of carrier concentration in semiconductor devices. It is also suggested that estimated concentration are comparable among users. This method does not depend on measurement mode, suggesting compatible to SCM, SSRM, SNDM and other next generation electric SPM.

Needs of the quantification of carrier concentration using electric SPMs are still strong in the semiconductor industry, and this kind of industrial standardization would be important and hurried as long as it is available, even if there are no proofs for traceable guarantees of the quantification. Because we know users need analytical solutions in the very front of them. This is also because they cannot wait the time until calibration certificated. In this way, relative values such as carrier concentration obtained by electric SPMs should be standardized under each country agreement. Then, we prepare a document for the analytical protocol of the carrier concentration conversion procedure in our working group of ISO/TC201/SC9. We also plan to circulate it in the overseas research organization in VAMAS's round robin test program for the timely industrial standardization.

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