

Analysis of high-k materials with Local Electrode Atom Probe

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Abstract

Hafnium-based high-k dielectrics are currently replacing the SiON gate in CMOS transistors due to their high dielectric constant (high-k), large bandgap and high thermodynamic stability with Si. The basic capability of atom probe tomography (APT) to analyze this type of material with good spatial resolution has been presented in the last few years. However, little has been said about the reproducibility and the measurement yield. Major questions like reproducibility and reliability of the measurements and possible measurement artifacts have hardly been discussed when this work was started. The main focus of the work presented here is to analyze high-k and metal gate materials and to describe the procedures necessary to obtain reliable and reproducible results with high spatial resolution and high measurement yield. Using these procedures, APT has a high potential of being widely used in the semiconductor industry on a daily basis.

The developed procedures in the following areas are presented: specimen preparation, cap layer engineering, measurement parameters and data analysis.

Specimen preparation by focused ion beam (FIB) for APT analysis is an important step to increase the reproducibility of APT measurements and the measurement yield. Procedures to obtain operator-independent tips are described in detail.

In order to protect the region of interest during FIB specimen preparation, cap layers are deposited via electron beam deposition. Different material properties like adhesion, number of isotopes, evaporation field and the sputtering rate in the FIB are investigated and their influence on the results is presented. Due to the introduction of the cap layer stack Cr / Si / Ni, high measurement yield can be achieved.

To obtain meaningful results, measurement parameters in the APT like the laser energy or the specimen temperature have to be chosen carefully. The influence of the specimen temperature is very small, whereas the high laser energy causes diffusion of the analyzed material. Using low laser energies leads to reproducible results.

Data treatment is the final important step to obtain correct results. Background subtraction of the random noise is described and the influence on the chemical composition and the depth scale is discussed. By applying the background subtraction and depth scale correction, reproducible results are obtained.

The reconstruction procedure in APT and its limitations are summarized and in measurements of boron delta layers deviations from the currently used reconstruction model are observed and discussed.

After the investigation of these procedures, their application to a number of different high-k and high-k / metal gate samples is presented. The comparison of APT results with different analysis techniques proves the high reproducibility of APT. For the analysis of 2 nm thick HfO₂ / ZrO₂ layers sub-nm depth resolution, correct chemical composition and high measurement yield are achieved. Different high-k samples, deposited with Metal Organic Chemical Vapor Deposition (MOCVD) and Atomic Layer Deposition (ALD), are analyzed and good reproducibility with high measurement yield is obtained. More complex material stacks consisting of HfO₂ and metal gate (TiN), deposited on silicon or silicon / germanium are analyzed and sub-nm depth resolution with correct chemical composition is achieved.

Abstract

In der Basis der CMOS-Transistoren sind Hafnium-basierte Dielektrika mit hoher Dielektrizitätskonstante (high-k) im Begriff, das Siliziumoxinitrid (SiON) aufgrund ihrer hohen dielektrischen Konstanten, großer Bandlücke und hoher thermodynamischer Stabilität zu Silizium zu ersetzen. In den vergangenen Jahren wurde gezeigt, dass man mit Hilfe der 3-dimensionalen Atomsonde (atom probe tomography = APT) diese Materialien mit guter Tiefenauflösung messen kann. Jedoch wurde wenig über die Reproduzierbarkeit und die Messausbeute berichtet. Wichtige Fragen wie die Reproduzierbarkeit und die Verlässlichkeit der Messungen sowie potenzielle Messartefakte waren, als diese Arbeit begonnen wurde, kaum Gegenstand der Diskussion. Das Hauptziel der vorgelegten Arbeit ist die Analyse von Dielektrika und Metallbasis (metal gate) und die Beschreibung der Prozeduren, die nötig sind, um verlässliche und reproduzierbare Ergebnisse mit guter Tiefenauflösung und hoher Messausbeute zu erzielen. Wenn man diese Prozeduren befolgt, dann hat die Atomsonde gute Chancen, sich in der Halbleiterindustrie als eine Standardmessmethode zu etablieren.

Die entwickelten Prozeduren in den folgenden Anwendungsgebieten werden vorgestellt: Probenpräparation, Verwendung der Schutzschichten, Messparameter und die Datenauswertung.

Probenpräparation für die Atomsonde mit Hilfe von fokussiertem Ionenstrahl (focused ion beam = FIB) ist ein wichtiger Schritt, um die Reproduzierbarkeit von Messungen und die Messausbeute in der Atomsonde zu erhöhen. Methoden, um eine benutzerunabhängige Spitzenpräparation zu erzielen, werden im Detail beschrieben.

Um den zu untersuchenden Bereich während der FIB-Probenpräparation zu schützen, werden Schutzschichten mit Hilfe von Elektronenstrahlabscheidung aufgetragen. Unterschiedliche Materialeigenschaften wie Adhäsion, Anzahl der Elementisotope, Verdampfungsfeld und die Sputterrate in der FIB werden untersucht und ihr Einfluss auf die Resultate wird präsentiert. Auf Grund der Einführung des Schichtstapels bestehend aus den Schichten Cr / Si / Ni, konnte die Messausbeute erhöht werden.

Um vernünftige Messresultate zu erhalten, müssen die Laserenergie und die Proben temperatur sorgfältig ausgewählt werden. Der Einfluss der Proben temperatur ist sehr klein, wohingegen die hohe Laserenergie eine Diffusion des analysierten Materials hervorruft. Das Verwenden von niedrigen Laserenergien führt zu reproduzierbaren Resultaten.

Die Datenauswertung ist der finale, wichtige Schritt, um korrekte Resultate zu erreichen. Untergrundabzug des Zufallsrauschens wird beschrieben und der Einfluss auf die chemische Zusammensetzung und die Tiefenskala wird diskutiert. Durch den Untergrundabzug und die Tiefenskalakorrektur werden reproduzierbare Ergebnisse erzielt.

Die Rekonstruktionsprozedur der Atomsonde und deren Grenzen werden zusammengefasst und anhand der Messungen von Bor-delta-Proben werden die Abweichungen von dem aktuellen Rekonstruktionsmodell beschrieben und diskutiert.

Nach der Untersuchung dieser Prozeduren wird deren Anwendung für zahlreiche Proben, bestehend aus reinen Dielektrika oder Dielektrika mit Metallbasis, demonstriert. Der Vergleich der Atomsondenresultate mit zahlreichen Messmethoden bestätigt die hohe Reproduzierbarkeit der Atomsonde. Bei der Analyse der 2-nm dicken HfO_2 / ZrO_2 Schichten wurden eine Tiefenauflösung von unter einem Nanometer, korrekte chemische Komposition und hohe Messausbeute erreicht. Unterschiedliche Proben, mit metallorganischer chemischer Gasphasenabscheidung (MOCVD) oder der Atomlagenabscheidung (ALD) abgeschieden, wurden analysiert und gute Reproduzierbarkeit mit hoher Messausbeute wurde erreicht. Kompliziertere Materialschichten, bestehend aus HfO_2 und TiN auf Si oder SiGe, wurden analysiert und auch hier wurden eine Tiefenauflösung von unter einem Nanometer und korrekte chemische Komposition erreicht.

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- Shariq, Ahmed; Mutas, Sergej et al (2009). Investigations of field-evaporated end forms in voltage- and laser-pulsed atom probe tomography, *Ultramicroscopy* (109), pages: 472-479, <http://dx.doi.org/10.1016/j.ultramic.2008.10.001>
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