Scratching the surface: Real-time monitoring of surface changes at the atomic level

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A team of researchers at Aix Marseille Université in Marseille, France led by Dr. Frédéric Leroy developed a technique that allows them to follow physical processes occurring at surfaces of materials at the atomic level in situ and in real time. This new process allowed the research team to study the kinetics of decomposition of a thin layer of silicon dioxide deposited onto silicon during a thermal treatment, a critical component in micro-electronics. The approach is based on the principles of electron microscopy.

Silicon dioxide is one of the most important building blocks of micro-electronics and its thermal stability is critical to device performance. The decomposition of a thin layer of silicon dioxide onto silicon has been the focus of great scientific interest for four decades. Previous studies show that the decomposition occurs non-homogeneously at the surface via the local formation of holes in the oxide layer that extend laterally. Understanding the elementary atomic processes responsible for the opening velocity of these holes is necessary to improve the silicon oxide performance.

For the research team to achieve a better understanding of nanomaterials properties, advanced characterization tools were needed.

"We needed to be able to characterize the structural (crystallography, size, shape) and the chemical properties at the same time and to be able to follow in situ and in real time the changes during a given process for a rapid feedback on the experimental parameters," Leroy explained. "Our approach based on low energy electron microscopy is the corner stone of our achievements."

However, even with the new instrument, the team encountered challenges. Obtaining real time measurements of the thermal decomposition of the silicon dioxide was particularly difficult since the complete process occurs in just a few minutes in a narrow temperature window.

"It was impossible to adjust all control parameters of the electron microscope before the decomposition process started since silicon dioxide is amorphous, so we had to adjust finely the settings within a few seconds as soon as the oxide decomposes in order to characterize the whole process," Leroy explained.

However, the meticulous measurement yielded some surprising results. Leroy and his research team found experimental evidence that the decomposition process was not initially in a steady state regime as previous studies had argued.

"Our results imply that the conventional view of a steady state regime for the silicon dioxide decomposition related to a simplified reaction $Si+SiO_2 \rightarrow 2SiO(g)$ occurring at the hole edge is not generally true," Leroy said. Instead, the team's results imply that silicon dioxide decomposition occurs via hole nucleation and opening with a circular shape. The velocity of holes opening is intimately related to the decomposition rate of silicon dioxide at the periphery of the holes. Initially, large holes open fast thanks to a chemical reaction catalyzed by species such as Si hydroxyls present inside the hole. Researchers suspect these species agglomerate during long thermal annealing and are released inside the holes during the silicon dioxide decomposition.

The main applications of this work are in micro-electronics, particularly all steps of thermal treatments.

"We have shown that the silicon dioxide formed by a wet chemical treatment is highly defective after a long thermal annealing," Leroy said. "The next step in our research is to study the interplay between chemical reactions and the enhancement of the mobility of nanostructures."

More information: "Catalytically enhanced thermal decomposition of chemically grown silicon 2 oxide layers on Si(001)," by F. Leroy, T. Passanante, F. Cheynis, S. Curiotto, E. B. Bussmann and P. Muller, *Applied Physics Letters* March 15, 2016. DOI: 10.1063/1.4941799

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