Application of ERBS analysis on O diffusion in TiO₂ films

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Electron Rutherford Backscattering (ERBS) is a technique that depends on the recoil energy transferred from the scattering electron to a nucleus in a large-angle deflection. This energy transfer depends on the mass of the scattering atom. Analysing the energy of the scattered electrons reveals thus which atoms are present in the near surface layer [1, 2].

In simple cases where there are 2-3 separate peaks due to different elements that do not overlap one can simply fit the spectra with the corresponding number of Gaussians. In more complicated cases the peaks overlap and a unique fitting of the spectrum based on a larger number of Gaussians can not be obtained. Besides the composition of each layer it is then of interest to measure their thickness. The added complexity usually require the measurement of the sample under different geometries and/or incoming energies and the simultaneous analysis of all spectra. As this work shows, fitting of the data allows for very precise thickness and compositional determination. Among other examples [3], the analysis of a Si₃N₄ layer on TiO₂ is particularly interesting. In this case the peaks overlap strongly, and show that highly consistent estimates of the thickness of the Si₃N₄ film are obtained for different measurement geometries.

This procedure was then applied to the study of oxygen auto-diffusion through the use of isotopic marking, in an attempt to obtain the depth profile distribution attributed to the diffusions. High-resolution measurement of the energy of electrons backscattered from oxygen atoms makes it possible to distinguish between ¹⁸O and ¹⁶O isotopes as the energy of elastically scattered electrons depends on the mass of the scattering atom.

Oxygen diffusion in TiO₂ is of particular interest, because it is the material that has received most work in memristors and is a well studied system within ReRAMs (resistive random access memories) [4]. Conventional ion-beam techniques are capable of such resolution but require particular care to ensure that radiation damage does not contribute to the measurement. Here we address these limitations by employing an electron-scattering technique to measure interdiffusion in $Ti^{16}O_2 / Ti^{18}O_2$ and $Ti^{18}O_2 / Ti^{16}O_2$ bilayers.

Thermal annealings for 5 minutes at $500 - 800^{\circ}$ C in Ar atmosphere were conducted on our samples. Under these conditions, we were able to determine the activation energy for O self-diffusion in TiO₂ at about 0.9 eV. By thermally treating samples for different times (5 – 100 min.) at a fixed temperature (650°C), the diffusion regularity was also studied. The Arrhenius plot of diffusion length versus time exhibits two regions, suggesting that different diffusion mechanisms are involved.

References.

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