Chemical analysis of lead zirconium titanium oxide films

Laurent Sagalowicz, Thomas Maeder, Paul Muralt and Nava Setter.

Laboratoire de Céramique, Département de Matériaux,
Ecole Polytechnique Fédérale de Lausanne, Lausanne, CH-1015, Switzerland.

Original version: “Trinoculaire" dc Microscopies Electroniques. Lausanne, juin 1995, p. 5.

Lead zirconium titanium oxide (PZT) thin films are used for their ferroelectric and piezoelectric properties. The composition of the PZT is very important for these properties, and may also be influenced by inter-diffusion with the bottom electrode, on which the PZT film has been grown at rather elevated temperature (550°C-600°C). The bottom electrode is essentially made of RuO_2 deposited on a chromium adhesion layer which also helps to block oxygen diffusion. A part of the multi-layer device is shown in Figure 1. Since now no comprehensive study concerning rnicroanalysis of the device have been made, the composition of the films as well as the other layers are not precisely known. It is therefore not possible to relate the composition to the functional properties of the device and to optimize the parameters of deposition. It was the aim of this work to study the composition of the $Pb(Zr,Ti)O_3$ layer, and the bottom electrode by mean of energy dispersive X-ray analysis (EDX) and energy electron loss spectroscopy (EELS). The experiments were performed with the Hitachi HF 2000 equipped with a field emission gun located at the EPFL.

The EDX analysis reveals that the layers appear in their sequence of deposition, and no significant diffusion was observed. EELS was used to improve the spatial resolution. For example chromium was found in the PZT by EDX but was not found by EELS. It is therefore possible that a part of the chromium detected comes from the chromium adhesion layer.

A precise determination of the composition of the PZT is in process and will be presented The analysis of a standard sample reveals that the ratio between titanium and zirconium does not vary significantly with the thickness, but that there is very large loss of lead in thin regions due to the ion milling method used for preparation. The parameterless correction method developed by Vancappellen (1) will be used to take into account the effect of sample preparation as well as absorption.

Reference

Van Cappellen E., (1990), Microsc. Microanal. Microstruct. 1, 1-22.

Figure 1 : Dark field electron micrograph showing a part of the multi-layer device. The Cr, RuO_2, and $Pb(Zr,Ti)O_3$ (PZT) layers are indicated.