Characterization of the Surface Potential of PZT-films for an Electrostatic Printing Process

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Abstract - The materials investigated in this paper are PZT-thick films on polycrystalline Al_2O_3-substrates metallized with a Pt-electrode. The uniformly polarized films were locally polarized in the opposite direction with a metallic electrode as well as with an electron beam in the SEM. The surface potential resulting from the latent charge image at the film surface was measured after polarization and after contrast enhancement by corona charging. Furthermore, the potential contrast could be visualized in the SEM at small accelerating voltages and minimal sample currents. The depth profile of the polarization perpendicular to the film surface was measured by the pyroelectric laser intensity modulation method (LIMM). The influence of film inhomogeneities on the surface potential is discussed.

INTRODUCTION

The switchable polarization is a property of ferroelectric materials which is used in several applications in the field of information storage. An electrostatic printing technique is a special application, where ferroelectric films are used as permanent masters for multicopy printing [1]. This process is similar to the xerographic process, where the information is stored in a photoconductor. The advantage of ferroelectric films consists in the permanence of storage.

Imaging is done by poling, where the stored information is erasable either electrically (by switching of the polarization) or thermally (by heating above T_C). The latent image of polarization is developed by a liquid toner consisting of charged particles, which are attracted by the electric field at the surface of the film at the polarized image points.

EXPERIMENTAL PROCEDURE

The investigated ferroelectric thick films (d≈100μm) consist of a low sintering $Pb[Zr_{1-x}Ti_x]O_3$-$PbMg_{1/3}Nb_{2/3}O_3$-material (PZT-PMN), deposited on a Pt-metallized Al_2O_3-substrate (50x50 mm^2) and sintered at T=950°C [2, 3]. The thick films are characterized by a dielectric constant ε of 1500 at 1 kHz, a remanent polarization P_R of 10...12 μCcm^{-2} and a coercive field E_C of 0,8...1 $kVmm^{-1}$ (50 Hz sinus).

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The electric surface potential was measured with an electrostatic voltmeter (ESVM) [4]. The sample was scanned with a motor driven x-y-table with the sensor of the ESVM (Monroe 170-1/1017EH) being fixed above the sample surface in a distance of about 100 μm.

The SEM investigations were carried out with a Zeiss electron microscope DSM 962. For the visualization of the potential contrast it is necessary to work at low accelerating voltages (0,5 to 3 kV) and minimal sample currents (Ip≈1 pA) to avoid additional charging of the surface of the insulating films. The equilibrium between the current of incident primary beam and the sum of the currents of the secondary electrons, the back scattered electrons and the minimal sample current has to be kept constant during the observation [5,6]. The poling experiments in the SEM were performed at an accelerating voltage of 15 kV at polished layers, where the penetration depth of the beam is ≈ 0.7 μm. During this process so many charges are locally bombarded to the surface until the resulting electric field exceeded the coercive field and the corresponding region was polarized [7]. For the generation of defined test structures during the e^--beam polarization the lithographical program ELPHY [8] was used.

For the LIMM-measurements [9] Al-electrodes (thickness 50 nm, ∅ 2 mm) were evaporated on the film surface. The electrode was exposed to a laser beam (laser diode with λ=830 nm) which was intensity modulated sinusoidally in the frequency range from 10 to $2 \cdot 10^6$ Hz. The absorbed thermal energy results in a temperature wave in the film. The attenuation and therefore the penetration depth of this wave is decreased with increasing frequency. Due to the pyroelectric effect the local temperature change in the film causes a pyroelectric current between ground and top electrode which was measured phase sensitive by a lock-in amplifier. From the frequency dependent current I(ω) the thickness dependence of the polarization P(z) can be calculated [10].

RESULTS AND DISCUSSION

Electrostatic Voltmeter

At first the films were polarized using a metallic electrode with a field strength of 2 $kVmm^{-1}$ (background polarization).