

# Domain structure and polarization reversal in ferroelectrics studied by atomic force microscopy

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The ferroelectric domain structure and its dynamics under applied electric field have been studied with nanoscale resolution by atomic force microscopy (AFM). Two mechanisms responsible for the contrast between opposite domains are proposed: large built-in domains are delineated in friction mode due to the tip-sample electrostatic interaction, and small domains created by an external field are imaged in topography mode due to piezoelectric deformation of the crystal. The ability of effective control of ferroelectric domains by applying a voltage between the AFM tip and the bottom electrode is demonstrated. It is experimentally confirmed that the sidewise growth of domain proceeds through the nucleation process on the domain wall. © 1995 American Vacuum Society.

## I. INTRODUCTION

Ferroelectric materials are a special group of polar dielectrics which possess the ability to switch their spontaneous polarization with an external electric field. The opposite ferroelectric domains have piezoelectric, pyroelectric, electro-optic and nonlinear optic constants which are opposite in sign, and this character of the antipolar domains is the basis for many applications. Over the last few years interest in ferroelectrics has essentially increased as a result of recent achievements in the processing of ferroelectric thin films and their wide application in various electronic and optoelectronic devices. Because the arrangements of domain structures have a direct influence on the macroscopic properties of ferroelectric samples, it is of great importance to be able to examine the ferroelectric domain structure on the smallest possible scale. To visualize the domain structures of ferroelectric materials, several methods have been applied, such as polarization microscopy, surface etching, powder decoration, and electron microscopy.<sup>1-4</sup> Recently the possibility of revealing ferroelectric domains using atomic force microscopy (AFM) in the micron range has been reported.<sup>5</sup> AFM promises to be a powerful tool for the study of ferroelectric domains providing the nanoscale resolution of domain structure.<sup>6,7</sup> Due to the very small curvature of the AFM tip, an extremely high electric field can be generated by applica-

tion of a comparatively low potential. This makes AFM a very promising technique for the modification of domain structure by local polarization reversal and for the creation of a highly ordered domain structure with an average resolvable spacing close to the physical limit. Another attractive prospect for AFM, not yet realized, would be direct nanoscale imaging of domains during their switching. This would allow one to directly study the nature of ferroelectric behavior.

In the present article, an analysis of the basics of ferroelectric domain contrast during AFM imaging is given. Using a conductive cantilever under variable electric potential, a controllable local switching of polarization in the ferroelectric crystal has been realized and the dynamics of domain nucleation, growth and interaction, have been studied with nanoscale resolution.

## II. EXPERIMENT

To investigate the domain contrast in AFM we used a guanidinium aluminum sulfate hexahydrate (GASH) crystal, chemical formula  $C(NH_2)_3Al(SO_4)_2 \cdot 6H_2O$ . GASH is an uniaxial ferroelectric, belonging to the trigonal crystal system (space group  $P31m$ ).<sup>8</sup> The spontaneous polarization  $P_s$  is about  $0.35 \mu C/cm^2$ , the dielectric constant,  $\epsilon_c$  along the polar axis is 15, and the coercive field is about  $1.5 \times 10^5$  V/m. GASH has a perfect cleavage plane normal to the polar axis  $c$ , which is suitable for studying the domain structure. The thickness of crystal plates, which were cleaved in air, varied from 50 to 200  $\mu m$ . The samples were investigated in an ambient environment by means of a commercially available microscope (SPA 300, Seiko Instruments) operating both in topography and friction modes. Static and dynamic domain

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