MECHANICAL CHARACTERIZATION OF THIN FILMS WITH APPLICATION TO FERROELECTRICS

Thesis by
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In Partial Fulfillment of the Requirements
for the Degree of
Doctor of Philosophy

1891

CALIFORNIA INSTITUTE OF TECHNOLOGY
Pasadena, California

2006
(Defended June 1, 2005)

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Acknowledgements

I would like to express my sincere acknowledgement of the many individuals who helped me throughout this long journey. The very first person I would like to fully acknowledge is my research advisor and mentor, Professor Gurswami Ravichandran, for his continuous trust, constant support, and strong influence throughout all my research development. It has been a great privilege for me to work with Professor Ravichandran and learn from his experience. I would like to thank my coadvisor Professor Kaushik Bhattacharya for the guidance and influential discussions. His theory work is the motivation for this project and his brilliant ideas provided me with the inspirations for the experimental work. I would also like to acknowledge him and Professors Sosina Hale, David Goodwin, and Alan Molinari for kindly participating in my thesis committee. I would also like to thank Professor Ares Rosakis for discussions about my work and his influential teaching.

The research presented in this dissertation was supported through grant from the Army Research Office through grant no. DAAD19-01-1-0517. The support of the sponsor is gratefully acknowledged.

I would like to acknowledge a number of people who have been very supportive all those years. These are my friends at Caltech who taught me a lot and were by my side when I needed their help. I would like to specifically thank my lab mate and dear friend Dr. Shiming Zhuang, who helped me get started in the lab and passed me his experience in designing, Dr. Doron Shilo for the fruitful discussions and valuable suggestions and Dr. Eric Burcsu for helping me start with ferroelectrics.

I would like to thank the numerous Caltech friends Dr. Vijaya B. Chalivendra, Dr. Murat Vural, Dr. Jiangyu Li, Dr. Kaiwen Xia, Dr. Yabei Gu, Dr. Wei Zhang, Min Tao, Fu-Ling Yang, Hsin-Ying Chiu, Yizhen Zhang, Teresa Kidd, Jean-Thibault De Besombes, Christian Frank, and many others, for their friendship and warm hearted help.

I would like to thank secretaries of Professor Ravichandran, first Ms. Denise Thobe and then Ms. Donna Mojahedi and now Ms. Sydney Garstang. They are very professional and very helpful. I would also like to thank the option secretary Ms. Cynthia Garza for her prompt and warm-hearted help. My life at Caltech would be much harder without their help.

Finally, I would like to thank my whole family for the encouragement, especially for my husband, Junhua. His unconditional support gave me inspiration throughout the research development.

Abstract

One important part of the motivation for this research work comes from the microelectromechanical systems (MEMS) technology. Its basic concept of high volume production and low unit cost can only be achieved when the devices made by microelectronics technique are reliable. The success in this area largely depends on the understanding of materials. However, the mechanical characterization is lagged behind the theoretical work and designing software development. The standard characterization method is still not established. For MEMS actuators, especially for active materials, the desired characterization system for obtaining mechanical properties requires load control feature and the capability of doing dynamic tests. However, there is no such method among the currently available tools for mechanical characterization.

The other part of the motivation comes from the comprehensive research work of Caltech ferroelectric group. This group, which consists of nine faculty members, is aiming to develop new devices, especially new actuators, by the aid of multi-scale theory tools and selected experimental methods. The work presented in this dissertation is an important and key step of this ambitious project: the electromechanical characterization of devices. This will provide validation for the multi-scale materials modeling framework and help to increase the reliability of the actuators and devices.

In this work, two techniques were developed for mechanical characterization, which satisfy the challenging requirements for thin film structures and devices: being able to do dynamic study on fragile ceramic thin film samples with load control feature. The first technique is a new method to characterize mechanical properties of released thin films

under concentrated load. This technique can be used to apply load in the μN–mN range with displacement measured with high accuracy of 0.1 μm. The successful characterization of Si_3N_4 free-standing membranes demonstrated the capability and reliability of this new technique. The elastic modulus and residual stress of Si_3N_4 free-standing thin film were measured to be around 250 GPa and 450 MPa, respectively. These values were in close agreement with values obtained using a different technique as well as those found in the literature. This technique has the potential application on elastic-plastic characterization and characterization of other functional thin film materials such as shape memory alloys.

Pressure bulge test technique, which is another type of load control method suitable for dynamic test, was also developed. The apparatus was designed to be compact to fit into the x-ray diffractometer for in-situ XRD study and had additional compatibility for polarized light microscopy study. Characterization of free standing thin film of single layer amorphous silicon nitride (Si_3N_4) and multi-layered PBT/Si_3N_4, and thick film of single crystal barium titanate ($BaTiO_3$) showed the capability and reliability of this technique. Excellent agreement of the Si_3N_4 Young's modulus between these two developed methods gave the confidence for using these techniques to understand new materials.

In situ x-ray diffraction study was carried out on the single crystal thick films which were loaded with distributed mechanical loading by pressure bulge setup. Direct evidence of 90^o domain switching was obtained from the *in situ* XRD results with the intensity changing in both (002) and (200) orientations. Obvious changes in domain patterns were observed by using the polarized light microscope. The Young's modulus of this barium titanate single crystal thick film with thickness of 100 μm was characterized before the

XRD exam. Using this information, in-plane stress can be analyzed, and the relation between the driving force (the stress) and the microstructural change (volume fraction change in *a*-domain or *c*-domain) can be determined.

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