doi:10.1017/S1431927614010009
Microsc. Microanal. 20 (Suppl 3), 2014

TEM In-Situ Electrical Testing of a FIB-prepared $BaTiO_3$ Ceramic Base Metal Electrode Capacitor

Zachary Lingley[1], Talin Ayvazian[1], Jesse Theiss[1], Miles Brodie[1], and Brendan Foran[1]

[1]The Aerospace Corporation, 2310 E. El Segundo Blvd., El Segundo, CA 90245-4691

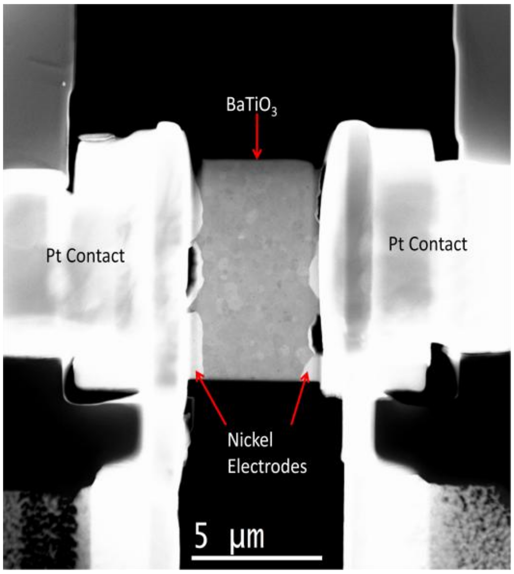
High reliability capacitors require a dielectric layer with both high electrical resistivity and high static dielectric constant. The most commonly used material platform for low cost, base metal electrode capacitors (BME) consists of nickel electrodes and barium titanate-based dielectric layers. Barium titanate ($BaTiO_3$) is ferroelectric material with dielectric constant that depends on factors including grain size and density of ferroelectric domain walls. Failure in BME's typically occurs by slow degradation in electrical resistance followed by a rapid thermal event leading to a short. The initial degradation depends on the structure and composition of the $BaTiO_3$-based dielectric and in particular oxygen vacancies that result from sintering in reducing environments. Oxygen vacancies in the $BaTiO_3$ act as n-type dopants and reduce resistivity [1]. Considerable efforts have been made to counteract the effect of oxygen vacancies and restore the high resistivity by the addition of trace elements that replace the Ti^{+4} ions and act as p-type dopants. However, these elements can affect ferroelectric ordering. Furthermore, the distribution of trace elements is typically concentrated near the grain boundaries resulting in grains with non-uniform characteristics. Thus to create reliable capacitors the relationship between the local crystal structure, ferroelectric domain structure, electrical resistivity, and chemical composition is important to understand.

A single layer of nickel – $BaTiO_3$ capacitor is prepared by focused ion beam lift-out and FIB deposited Pt to make electrical contacts to commercially available TEM grids with electrical leads (designed previously by our group [2]) enabling external electrical biasing and in-situ stressing and measurements of leakage current in the TEM. This allows us to create unique specimens with $BaTiO_3$ dielectric layers harvested from commercial BME capacitors to study electrical degradation while simultaneously characterizing structure, composition, and ferroelectric ordering [3]. An image of a nickel – $BaTiO_3$ sample with Pt connections to an external voltage supply is shown in Figure 1(a), and a higher magnification image of a set of grains with one showing the characteristic domain wall stripe pattern (with grain marked by a black arrow) is shown in Figure 1(b).

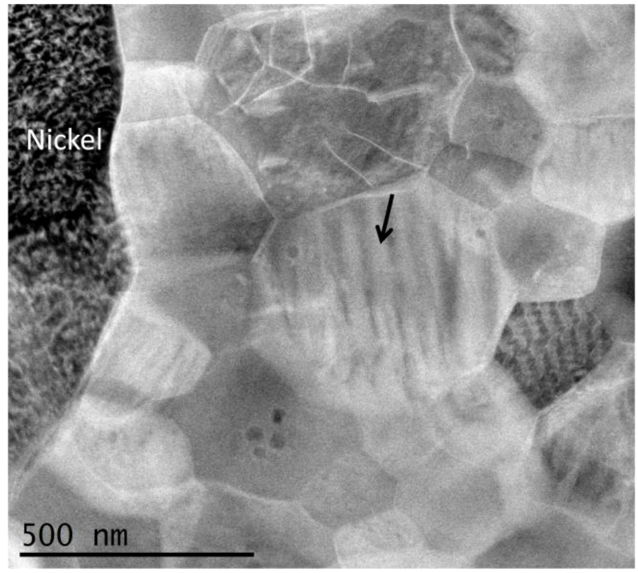
From in-situ electrical measurements of the as-prepared sample, we estimate that resistivity of the specimen is 4-5 orders of magnitude lower than expected for doped $BaTiO_3$ [4]. This suggests that current leakage is due to surface contamination and therefore pointing to the need for an improved surface passivation approach to study the capacitor in the TEM.

References:

[1] GY Yang *et al,* Journal of Applied Physics **96** (2004), p. 7492.
[2] M Mecklenburg *et al,* Microscopy and Microanalysis **19** (2013) p. 458.
[3] A Krishnan *et al,* Materials Research Society Symposium Proceedings **541** (1998), p. 475.
[4] H. Kishi *et al,* Japanese Journal of Applied Physics **42** (2003), p. 1.
[5] This work was supported in part through The Aerospace Corporation's Internal Research and Development Program.



(a)



(b)

Figure 1. (a) A scanning transmission image of a single, electron transparent layer of nickel-BaTiO3 capacitor wired with Pt contacts for in-situ TEM analysis. (b) higher magnification image of the part of the same sample showing the $BaTiO_3$ grain structure and ferroelectric domains in the grain marked with the black arrow.

https://doi.org/10.1017/S1431927614010009 Published online by Cambridge University Press